

UNITED STATES PATENT OFFICE

2,640,029

PROCESS FOR PREVENTING CORROSION

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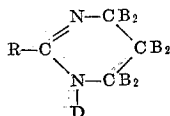
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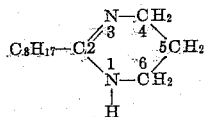
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This invention relates to the inhibition of corrosion of metals, and particularly to a composition for use in preventing corrosion of metals and particularly iron, steel, and ferrous alloys. The corrosion inhibitors contemplated herein find special utility in the prevention of corrosion of pipe or equipment which is in contact with a corrosive oil-containing medium, as, for example, in oil wells producing corrosive oil or oil-brine mixtures, in refineries, and the like. Our inhibitors may, however, be used in other systems or applications. They appear to possess properties which impart to metals resistance to attack by a variety of corrosive agents, such as brines, weak inorganic acids, organic acids, CO_2 , H_2S , O_2 , etc.

In its broadest aspects our invention contemplates a process for preventing corrosion of metals, comprising the step of applying to such metals a substituted tetrahydropyrimidine of the formula type:



where D is a member of the class consisting of $\text{D}'-\text{R}$ and R ; D' represents a divalent organic radical containing less than 25 carbon atoms, composed of elements from the group consisting of C, H, O, and N; R is a member of the class consisting of hydrogen and hydrocarbon radicals, with the proviso that at least one occurrence of R contains from 8 to 32 carbon atoms; B is a member of the class consisting of hydrogen and hydrocarbon radicals containing less than 7 carbon atoms, with the proviso that at least three occurrences of B be hydrogen. A simple example of a compound of this type is shown in the formula below which is that for 2-octyl tetrahydropyrimidine and in which the elements of the ring are numbered to conform to the conventional numbering system.



We have found that compounds of this type and related compounds containing the tetrahydropyrimidine nucleus and which are more fully described below are especially effective inhibitors for the corrosion of metals. In the formula above the relatively high molecular weight hydrocarbon radical appears as a substituent of the 2-carbon

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atom of the tetrahydropyrimidine nucleus. Reagents of this type are particularly simple to prepare and are very effective corrosion inhibitors. We have found, however, that equally effective compounds result when the relatively high molecular weight hydrocarbon group occurs as a substituent of one of the nitrogen atoms, or of a relatively small organic radical attached to one of the nitrogen atoms. Further we have found that the ring carbon atoms of the cyclic nucleus may be substituted by relatively small hydrocarbon groups, i. e., groups containing 6 or fewer carbon atoms, without affecting the apparently inherent corrosion preventing or inhibiting character of the tetrahydropyrimidines, provided the molecule contains at least one hydrocarbon radical having from 8 to 32 carbon atoms.

The preparation of tetrahydropyrimidines substituted in the 2-position by hydrocarbon radicals is well described in the literature and is readily carried out by reaction between a monocarboxylic acid and a diamine, or a polyamine, containing at least one primary amino group, and at least one secondary amino group, or another primary amino group separated from the first primary amino group by three carbon atoms. This reaction is generally carried out by heating the reactants to a temperature of 230°C . or higher, usually within the range of 250° to 300°C ., at which temperatures water is evolved and ring closure is effected. For details of the preparation of such reagents, see German Patent 700,371, dated December 18, 1949, to Edmund Waldmann and August Chwala; German Patent 701,322, dated January 14, 1941 to Karl Miescher, Ernst Urech, and Willi Klarar; and U. S. Patent 2,194,419, dated March 19, 1940, to August Chwala.

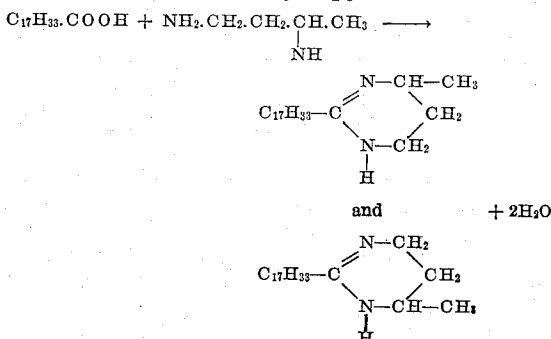
When a monocarboxylic acid containing 9 or more carbon atoms is employed in the above described synthesis, the resulting tetrahydropyrimidine will contain a 2-substituent consisting of a hydrocarbon radical containing 8 or more carbon atoms. Suitable corrosion preventive reagents may, therefore, be made directly by reaction of carboxylic acids such as oleic acid, linoleic acid, linolenic acid, erucic acid, tall oil fatty acid, naphthenic acid, nonoic acids, alpha-naphthylacetic acid, abietic acid, crude pine rosin acids, and the like with suitable amines such as those enumerated below:

Examples of amines suitable for this synthesis include 1,3-propylenediamine, dipropylene triamine, 1,3-diaminobutane, 2,4-diaminopentane, N-ethyl-trimethylene diamine, N-aminoethyl-trimethylene diamine, aminopropyl stearylamine,

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tripropylenetetramine, tetrapropylenepentamine, high boiling polyamines prepared by the condensation of 1,3-propylene dichloride with ammonia, and similar diamines or polyamines in which there occurs at least one primary amino group separated from another primary or secondary amino group by three carbon atoms.

The preparation of tetrahydropyrimidines substituted in the 2-position by a relatively high molecular weight hydrocarbon radical is exemplified by the reaction between oleic acid and 1,3-diaminobutane. If one molecular equivalent of oleic acid is reacted with one mole of 1,3-diaminobutane at a temperature of about 250° C. to 300° C., two moles of water are evolved, the reaction proceeding as shown in the equations below to form a mixture of 2-heptadecenyl, 4-methyl tetrahydropyrimidine and 2-heptadecenyl, 6-methyl tetrahydropyrimidine.



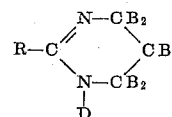
We have discovered that equally suitable corrosion preventive reagents may be obtained by introducing into the tetrahydropyrimidine molecule a hydrocarbon group of proper size as a portion of the substituent attached to the 1-nitrogen atom of the ring or as the substituent of the 1-nitrogen atom of the ring. Where the aliphatic hydrocarbon group occurs in this position it is unnecessary that the 2-carbon atom substituent contain 8 or more carbon atoms. It may be, in fact, only a hydrogen atom or a methyl group, ethyl group, phenyl group, or other relatively small hydrocarbon groups, although it is not restricted to such small groups. The preparation of the tetrahydropyrimidine compounds in which the higher molecular weight hydrocarbon radical occurs as a portion of the nitrogen atom substituent is also readily carried out by methods analogous to those already described. In this case, however, a number of alternative procedures are possible. For example, one may prepare 2-methyl, 1-octadecyl tetrahydropyrimidine by reaction of octadecyl aminopropylamine with acetic acid at a temperature of about 250° C. to 300° C. until two moles of water are evolved for every mole of acetic acid employed. The same reagent may be prepared by the reaction of 2-methyl, tetrahydropyrimidine with octadecyl bromide followed by separation of the resulting alkylation products to isolate the desired compound.

Particularly suitable for the synthesis of tetrahydropyrimidines having a high molecular weight hydrocarbon group attached to the 1-nitrogen of the ring or present as a portion of the 1-nitrogen substituent, are the high molecular weight N-substituted propylenediamines and dipropylenetriamines. Some of the former are commercially available products, exemplified by N-aminopropyl stearylamine, which may be readily prepared by condensation of acrylonitrile,

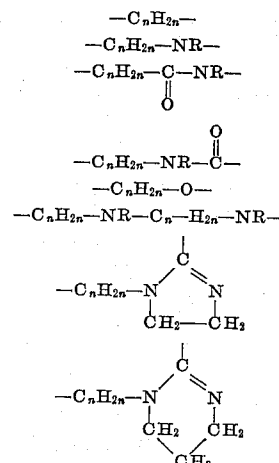
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with a high molecular weight amine, such as stearylamine, followed by hydrogenation and separation of the diamine. Analogous diamines derived from cetylamine, crude tall oil amine, abietylamine, oleylamine, dodecylamine, and similar are suitable for synthesis of the present corrosion inhibitors.

As stated above, our invention contemplates, in its broadest aspects, the use of tetrahydropyrimidines of the formula type

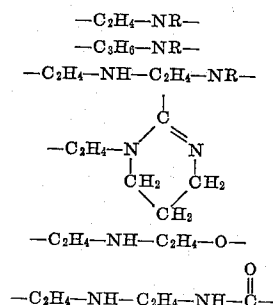


where the symbols have their previously described meaning. In these reagents, generally at least three occurrences of B will be hydrogen atoms. In the more common reagents D' will be a relatively small divalent organic radical composed of elements from the group consisting of C, N, O, and H, and may be a hydrocarbon group, an alkyleneamido group, an alkylene-amino group, a substituted acetyl group, and the like, such as are illustrated in the following examples



where *n* is the numeral 1 to 6, or where $-C_nH_{2n}-$ as a whole represents a divalent hydrocarbon radical containing from 1 to 6 carbon atoms and which may be aliphatic, cycloaliphatic, aromatic, or mixed in character, and where R, as before, is hydrogen or a hydrocarbon radical.

We have found that particularly effective corrosion preventive reagents result when the tetrahydropyrimidine compound contains basic nitrogen groups in addition to those nitrogen atoms inherent to the tetrahydropyrimidine ring. In general, compounds of this type are those in which the basic nitrogen group is contained in the radical D in the above formula. Examples of the radical D', where it contains basic nitrogen groups, are as follows:

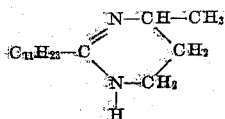


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where the symbols have their previous significance.

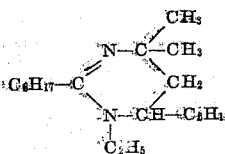
Examples of substituted tetrahydropyrimidines suitable for practice of the present invention and in which the hydrocarbon group containing from 8 to 32 carbon atoms is a 2-position substituent include the following:

1.



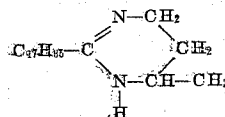
2-undecyl, 4-methyltetrahydropyrimidine

2.



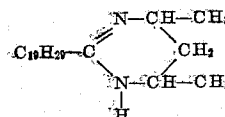
1-ethyl, 2-octyl, 4-dimethyl, 6-cyclohexyltetrahydropyrimidine

3.



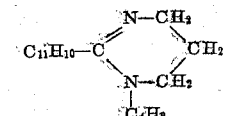
2-heptadecyl, 6-methyltetrahydropyrimidine

4.



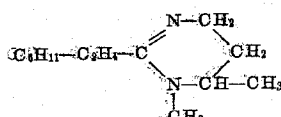
2-abietyl, 4,6-dimethyltetrahydropyrimidine

5.



2-naphthylmethyl, 1-butyltetrahydropyrimidine

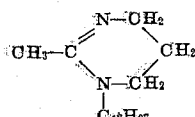
6.



2-cyclohexylethyl, 1,6-dimethyltetrahydropyrimidine

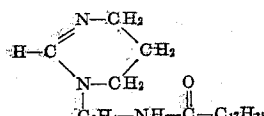
Examples of suitable corrosion preventives in which the hydrocarbon radical containing from 8 to 32 carbon atoms is a part of the 1-position substituent include the following:

7.



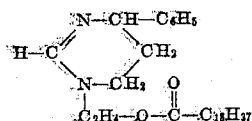
1-octadecyl, 2-methyltetrahydropyrimidine

8.



1-oleylamidoethyltetrahydropyrimidine

9.



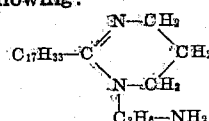
1-steroyloxyethyl, 4-phenyltetrahydropyrimidine

Examples of corrosion preventives useful in the practice of the present invention which contain

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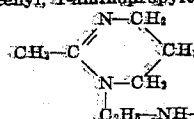
basic nitrogen groups besides those in the ring include the following:

10.



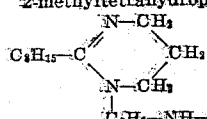
2-heptadecyl, 1-aminopropyltetrahydropyrimidine

11.



Chloroparaffin alkylation product of 1-aminopropyl, 2-methyltetrahydropyrimidine

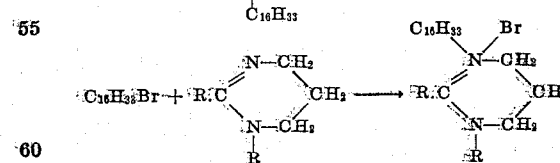
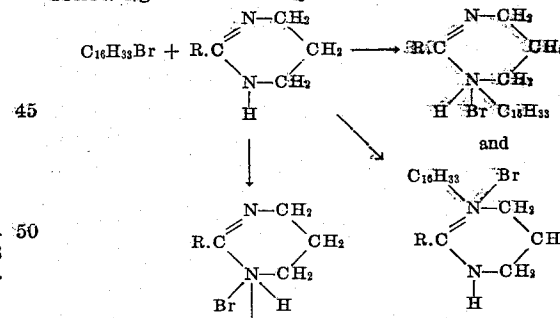
15 12.



1-dipropylenediamino, 2-cyclopentylpropyltetrahydropyrimidine

Although we have shown above the composition of a number of effective inhibitors which are tetrahydropyrimidines containing at least one hydrocarbon radical having from 8 to 32 carbon atoms, we should like to point out that, in general, the most effective reagents and those having the most desirable solubility characteristics are those in which the hydrocarbon group contains from 10 to 20 carbon atoms. Examples of such preferred groups are decyl, oleyl, abietyl, stearyl, and the like.

The corrosion preventive products of the present invention, since they contain a tetrahydropyrimidine ring, may, in general, be alkylated to form either a 1-alkyl-substituted tetrahydropyrimidine, or a quaternary ammonium salt, where the alkyl group is attached to either or both the 1 and 3 nitrogen atoms. For example, using cetyl bromide as a typical alkylating agent, the following reactions may be carried out:

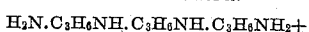


Instead of the cetyl bromide used in the examples above, one may use other alkylating agents such as methyl bromide, benzyl chloride, ethyl sulfate, dichloroethyl ether, chloroparaffin, etc., to obtain equally suitable derivatives of tetrahydropyrimidines which may be employed in the present process.

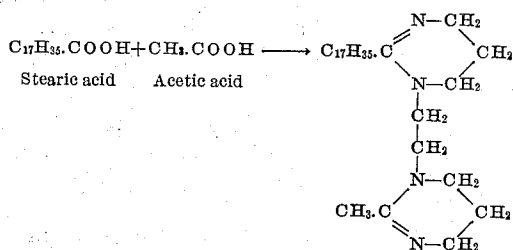
Although we have described the corrosion inhibitors of our process as tetrahydropyrimidines, we may, in many instances, employ these compounds in the form of their salts, either with organic or inorganic acids. Being relatively strong bases, the tetrahydropyrimidines readily form such salts, and where the reagent contains basic groups in addition to the tetrahydropyrimidine ring nitrogen atoms, they may form di- or polysalts. Examples of acids which may be used to form such salts are hydrochloric acid, sulfuric acid, acetic acid, glycolic acid, oxalic acid, maleic acid, oleic acid, abietic acid, phosphoric acid, petroleum sulphonic acid, naphthenic acid, rosin, phenylacetic acid, benzoic acid and the like.

Salts of the tetrahydropyrimidines, such as those above described, appear to be equally as effective as the free bases. Probably, in the dilute solutions in which they are employed as corrosion inhibitors, the salts hydrolyze or otherwise decompose to some extent and reach an equilibrium with the acids and other constituents of the corrosion medium.

While we have described our corrosion inhibitors as tetrahydropyrimidines and have illustrated them above as single ring compounds, it should be pointed out that in some instances reagent compounds containing two or more heterocyclic rings, such as two tetrahydropyrimidine rings may be employed. For example, if one reacts one mole of tripropylenetetramine with a mole of stearic acid to form a substituted heptadecyltetrahydropyrimidine, and then reacts this further with another mole of a carboxylic acid at a suitable high temperature, a ditetrahydropyrimidine is obtained.



Tripropylenetetramine



Such ditetrahydropyrimidines are intended to be included when reference is made to substituted tetrahydropyrimidines herein or in the claims.

Many obvious simple derivatives of the herein described corrosion inhibitors may be prepared which are also effective. For example, we have defined the group R in the structural formulae above as being a member of the class consisting of hydrogen, and hydrocarbon groups. Actually, the use of halogenated hydrocarbon groups appears to yield equally effective reagents, and chlorohydrocarbon groups, particularly, are readily introduced during synthesis. Since the chlorine atoms in these groups are relatively non-reactive and yield products with solubilities similar to the hydrocarbon derivative, they do not differ greatly in behavior from the corresponding hydrocarbon derivative.

The method of carrying out our process is rela-

tively simple in principle. The corrosion preventive reagent is dissolved in the liquid corrosive medium in small amounts and is thus kept in contact with the metal surface to be protected. Alternatively, the corrosion inhibitor may be applied first to the metal surface, either as is, or as a solution in some carrier liquid or paste. Continuous application, as in the corrosive solution, is the preferred method, however.

The present process finds particular utility in the protection of metal equipment of oil and gas wells, especially those containing or producing an acidic constituent such as H_2S , CO_2 , organic acids and the like. For the protection of such wells, the reagent, either undiluted or dissolved in a suitable solvent, is fed down the annulus of the well between the casing and producing tubing where it becomes commingled with the fluid in the well and is pumped or flowed from the well with these fluids, thus contacting the inner wall of the casing, the outer and inner wall of tubing, and the inner surface of all well-head fittings, connections and flow lines handling the corrosive fluid.

Where the inhibitor composition is a liquid, it is conventionally fed into the well annulus by means of a motor driven chemical injector pump, or it may be dumped periodically (e. g., once every day or two) into the annulus by means of a so-called "boll weevil" device or similar arrangement. Where the inhibitor is a solid, it may be dropped into the well as a solid lump or stick, it may be blown in as a powder with gas, or it may be washed in with a small stream of the well fluids or other liquid. Where there is gas pressure on the casing, it is necessary, of course, to employ any of these treating methods through a pressure equalizing chamber equipped to allow introduction of reagent into the chamber, equalization of pressure between chamber and casing, and travel of reagent from chamber to well casing.

Occasionally, oil and gas wells are completed in such a manner that there is no opening between the annulus and the bottom of the tubing or pump. This results, for example, when the tubing is surrounded at some point by a packing held by the casing or earth formation below the casing. In such wells the reagent may be introduced into the tubing through a pressure equalizing vessel, after stopping the flow of fluids. After being so treated, the well should be left closed in for a period of time sufficient to permit the reagent to drop to the bottom of the well.

For injection into the well annulus, the corrosion inhibitor is usually employed as a solution in a suitable solvent, such as mineral oil, methyl-ethyl ketone, xylene, kerosine, or even water. The selection of solvent will depend much upon the exact reagent being used and its solubility characteristics. It is also generally desirable to employ a solvent which will yield a solution of low freezing point, so as to obviate the necessity of heating the solution and injection equipment during winter use.

For treating wells with packed-off tubing, the use of solid "sticks" or plugs of inhibitor is especially convenient. These may be prepared by blending the inhibitor with a mineral wax, asphalt or resin in a proportion sufficient to give a moderately hard and high-melting solid which can be handled and fed into the well conveniently.

The amount of corrosion preventive agent required in our process varies with the corrosiveness of the system, but where a continuous or semi-continuous treating procedure is carried out as

described above, the addition of reagent in the proportion of from one part per 1,000 to one part per 20,000 or more parts of corrosive fluid will generally provide protection.

The effectiveness of the herein described reagents for the prevention of corrosion is illustrated by the data of the following table. These data show the reduction in attack of mild steel by 1% hydrochloric acid by various tetrahydropyrimidines over a range of concentrations. Duplicate weighed mild steel plates were immersed in 1% hydrochloric acid solution of the reagent shown for a period of 24 hours at a temperature of 28° C., after which they were removed, washed and again weighed. The loss in weight is expressed as the percentage of the loss in weight of the plates in 1% hydrochloric acid containing no inhibitor.

Concentration of tetrahydropyrimidine in 1% HCl (1%)	Loss in weight as Percent of Blank Loss		
	Compound of Example 1	Compound of Example 3	Compound of Example 10
0.005	5.1	6.2	4.7
0.01	4.3	4.9	3.6
0.02	4.1	4.3	2.9
0.04	3.7	3.8	3.0
0.06	3.7	4.0	3.0

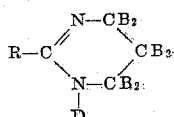
The protective action of the herein described reagents appears to be maintained for an appreciable time after treatment ceases, but eventually is lost unless another application is made.

For the protection of gas wells and gas-condensate wells, the amount of corrosion inhibitor required will usually be within the range of one-half to 3 lbs. per million cubic feet of gas produced, depending upon the amounts and composition of corrosive agents in the gas and the amount of liquid hydrocarbon and water produced. However, in no case does the amount of inhibitor required appear to be stoichiometrically related to the amount of acids produced by a well, since protection is obtained with much less tetrahydropyrimidine than usually would be required for neutralization of the acids produced.

Recapitulating, we have found that the corrosion of metals, and particularly ferrous metals, may be inhibited by the application thereto of a substituted tetrahydropyrimidine in which a substituent at either or both the 1- or 2-position of the ring contains a hydrocarbon group having from 8 to 32 carbon atoms. Of this broad genus of corrosion inhibitors, there are several subclasses which may be employed effectively in our process. Such sub-classes are, (1) those in which the 1-position substituent contains an amino group, (2) those in which the 1-position substituent is free of amino groups, (3) those in which the 4- and/or 5- and/or 6-position ring carbons are substituted, etc.

Having thus described our invention, what we claim as new and desire to secure by Letters Patent, is:

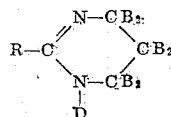
1. A process for preventing corrosion of metals including the step of applying to such metals a substituted tetrahydropyrimidine of the formula type:



where D is a member of the class consisting of D'-R and R; D' represents a divalent organic

radical containing less than 25 carbon atoms, composed of elements from the group consisting of C, H, O and N; R is a member of the class consisting of hydrogen and hydrocarbon radicals, with the proviso that at least one occurrence of R contains from 8 to 32 carbon atoms; B is a member of the class consisting of hydrogen and hydrocarbon radicals containing less than 7 carbon atoms, with the proviso that at least three occurrences of B be hydrogen.

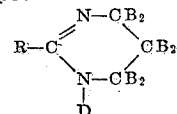
2. A process for preventing corrosion of ferrous metals including the step of applying to such metals a substituted tetrahydropyrimidine of the formula type:



where D is a member of the class consisting of D'-R and R; D' represents a divalent organic radical containing less than 25 carbon atoms, composed of elements from the group consisting of C, H, O and N; R is a member of the class consisting of hydrogen and hydrocarbon radicals,

with the proviso that at least one occurrence of R contains from 8 to 32 carbon atoms; B is a member of the class consisting of hydrogen and hydrocarbon radicals containing less than 7 carbon atoms, with the proviso that at least three occurrences of B be hydrogen.

3. A process for preventing corrosion of ferrous metals including the step of applying to such metals a substituted tetrahydropyrimidine of the formula type:

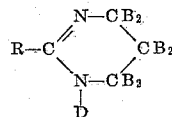


where D is a member of the class consisting of D'-R and R; D' represents a divalent organic radical containing less than 25 carbon atoms, composed of elements from the group consisting of C, H, O and N; R is a member of the class consisting of hydrogen and hydrocarbon radicals, with the proviso that at least one occurrence of R contains from 10 to 20 carbon atoms; B is a member of the class consisting of hydrogen and hydrocarbon radicals containing less than 7 carbon atoms, with the proviso that at least three occurrences of B be hydrogen.

4. A process for preventing corrosion of ferrous metals including the step of applying to such metals 2-heptadecyl, 6-methyltetrahydropyrimidine.

5. A process for preventing corrosion of ferrous metals including the step of applying to such metals 1-oleylamidoethyltetrahydropyrimidine.

6. A process for preventing corrosion of ferrous metals including the step of applying to such metals a substituted tetrahydropyrimidine of the formula type:

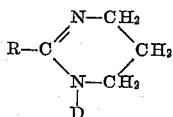


where D is a member of the class consisting of D'-R and R; D' represents a divalent organic radical containing less than 25 carbon atoms, composed of elements from the group consisting of C, H, O and N and containing at least one amino group; R is a member of the class consisting of hydrogen and hydrocarbon radicals, with the proviso that at least one occurrence of R

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contains from 10 to 20 carbon atoms; B is a member of the class consisting of hydrogen and hydrocarbon radicals containing less than 7 carbon atoms, with the proviso that at least three occurrences of B be hydrogen.

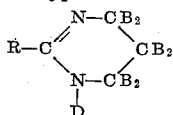
7. A process for preventing corrosion of ferrous metals including the step of applying to such metals a substituted tetrahydropyrimidine of the formula type:



where D is a member of the class consisting of D'-R and R; D' represents a divalent organic radical containing less than 25 carbon atoms, composed of elements from the group consisting of C, H, O and N and containing at least one amino group; R is a member of the class consisting of hydrogen and hydrocarbon radicals, with the proviso that at least one occurrence of R contains from 10 to 20 carbon atoms.

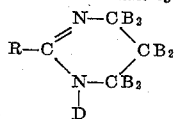
8. A process for preventing corrosion of ferrous metals including the step of applying to such metals 2-heptadecenyl, 1-aminopropyltetrahydropyrimidine.

9. A process for preventing corrosion of oil and gas well equipment, including the step of injecting into the well a substituted tetrahydropyrimidine of the formula type:



where D is a member of the class consisting of D'-R and R; D' represents a divalent organic radical containing less than 25 carbon atoms, composed of elements from the group consisting of C, H, O and N; R is a member of the class consisting of hydrogen and hydrocarbon radicals, with the proviso that at least one occurrence of R contains from 8 to 32 carbon atoms; B is a member of the class consisting of hydrogen and hydrocarbon radicals containing less than 7 carbon atoms, with the proviso that at least three occurrences of B be hydrogen.

10. A process for preventing corrosion of oil and gas well equipment, including the step of injecting into the well a substituted tetrahydropyrimidine of the formula type:



where D is a member of the class consisting of D'-R and R; D' represents a divalent organic radical containing less than 25 carbon atoms, composed of elements from the group consisting of C, H, O and N; R is a member of the class consisting of hydrogen and hydrocarbon radicals, with the proviso that at least one occurrence of R contains from 10 to 20 carbon atoms; B is a member of the class consisting of hydrogen and hydrocarbon radicals containing less than 7 carbon atoms, with the proviso that at least three occurrences of B be hydrogen.

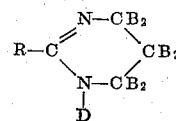
11. A process for preventing corrosion of oil and gas well equipment, including the step of injecting into the well 2-abietyl tetrahydropyrimidine.

12. A process for preventing corrosion of oil and gas well equipment, including the step of

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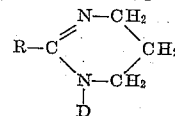
injecting into the well 1-octadecenyl, 2-methyltetrahydropyrimidine.

13. A process for preventing corrosion of oil and gas well equipment including the step of injecting into the well a substituted tetrahydropyrimidine of the formula type:



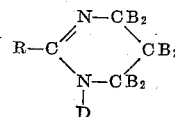
where D is a member of the class consisting of D'-R and R; D' represents a divalent organic radical containing less than 25 carbon atoms, composed of elements from the group consisting of C, H, O and N and containing at least one amino group; R is a member of the class consisting of hydrogen and hydrocarbon radicals, with the proviso that at least one occurrence of R contains from 10 to 20 carbon atoms; B is a member of the class consisting of hydrogen and hydrocarbon radicals containing less than 7 carbon atoms, with the proviso that at least three occurrences of B be hydrogen.

14. A process for preventing corrosion of oil and gas well equipment including the step of injecting into the well a substituted tetrahydropyrimidine of the formula type:



where D is a member of the class consisting of D'-R and R; D' represents a divalent organic radical containing less than 25 carbon atoms, composed of elements from the group consisting of C, H, O and N and containing at least one amino group; R is a member of the class consisting of hydrogen and hydrocarbon radicals, with the proviso that at least one occurrence of R contains from 10 to 20 carbon atoms.

15. A process for preventing corrosion of metals including the step of applying to such metals a carboxylic acid salt of a substituted tetrahydropyrimidine of the formula type:



where D is a member of the class consisting of D'-R and R; D' represents a divalent organic radical containing less than 25 carbon atoms, composed of elements from the group consisting of C, H, O and N; R is a member of the class consisting of hydrogen and hydrocarbon radicals, with proviso that at least one occurrence of R contains from 8 to 32 carbon atoms; B is a member of the class consisting of hydrogen and hydrocarbon radicals containing less than 7 carbon atoms, with the proviso that at least three occurrences of B be hydrogen.

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