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RUST INHIBITORS FOR AQUEOUS SOLUTIONS

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9 Claims. (Cl. 252—77)

This application is a continuation-in-part of our co-pending application Serial No. 106,509, filed May 1, 1961, and now abandoned.

The present invention relates to water-soluble corrosion inhibitors, and is more particularly concerned with aqueous solutions which normally cause rusting of ferrous metal surfaces in contact therewith.

It is well known to those familiar with the art that aqueous solutions in contact with ferrous metal surfaces have a strong tendency to induce rusting. This is particularly noticeable in the case of automotive cooling systems containing aqueous coolants, such as water, water-alcohol mixtures, or water-glycol mixtures. Rust is formed at the expense of the base metal, and severe corrosion can progress to a point wherein the walls of vessels or pipes are penetrated. Accumulations of rust in a system lower its volumetric capacity and operating efficiency, interfering with circulation of liquids and with the proper operation of valves. In extreme cases rust can completely clog the system.

It is therefore an object of the present invention to provide novel rust-inhibiting compositions.

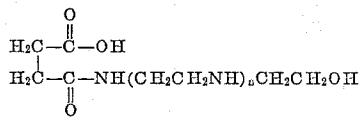
Another object of the invention is to provide water-soluble anti-rust agents for aqueous solutions that come into contact with ferrous metal surfaces.

A further object of the invention is to provide aqueous coolants that are effectively inhibited against rusting of ferrous metal surfaces.

Other objects and advantages inherent in the invention will become apparent to those skilled in the art, from the following detailed description.

It has now been found, in accordance with the present invention, that rusting of ferrous metal surfaces in contact with aqueous coolants can be prevented, simply and economically. In this respect, it has now been found that small amounts of certain water-soluble succinamic acids, when dissolved in aqueous coolants, in small amounts effectively prevent rusting of ferrous metal surfaces in contact therewith.

In accordance with the present invention there is provided an aqueous coolant containing a small amount, sufficient to prevent rusting of ferrous metal surfaces in contact therewith, of a succinamic acid having the formula:



wherein n is an integer varying from 1 to 4. The invention also provides a method for inhibiting rusting in cooling systems which comprises adding to an aqueous coolant, the above described succinamic acid.

The succinamic acid additives of this invention are readily prepared by reacting succinic acid anhydride with a specific type of polyethylene amine reactant. The amine reactant has the formula,

$$H_2N\ (CH_2CH_2NH)_nCH_2CH_2OH$$

wherein n is an integer varying from 1 to 4. Non-limiting examples of the amine reactant are hydroxyethyl-ethylenediamine, hydroxyethyl-diethylenetriamine, hydroxyethyl-triethylenetetramine, and hydroxyethyl-tetraethylenepentamine.

The reaction between the succinic acid anhydride and the amine reactant is an amide-forming reaction, which takes place readily without formation of water of condensation. The reaction is exothermic and is substantially completed without heating. It is desirable, however, to heat the reaction mixture at a temperature from about 75° C. to about 95° C., during a period of from about one-half hour to three hours to insure complete reaction. Preferably the reaction is carried out in a water-solvent.

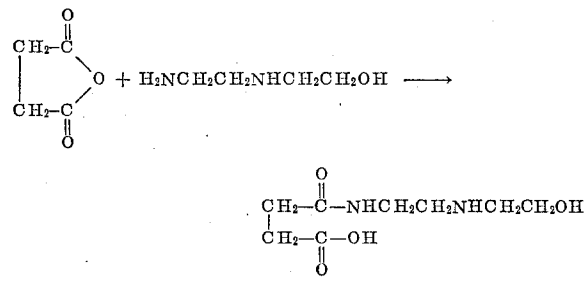
The aqueous coolant contemplated herein can be water. It may also include aqueous anti-freeze mixtures, such as water-isopropanol and water-ethylene glycol mixtures. The coolant may also contain small amounts of other materials for specific purposes, such as antioxidants and lubricants (for water pumps). The amount of the succinamic acid anti-rust agent to be added to the aqueous coolant will vary from about 0.1 percent and about 10 percent, by volume, and preferably from about 0.1 percent and about 5 percent, by volume.

Some degree of care must be exercised in selecting suitable environments in which the rust inhibitors of the present invention are used. In this respect, it has been found that subjection to elevated temperatures above about 80° C. results in impairment of ability to inhibit corrosion for extended periods of time.

The following examples are intended to illustrate the preparation of the novel anti-rust compositions of the present invention, but are not to be considered in any sense as being limited thereto.

Example I

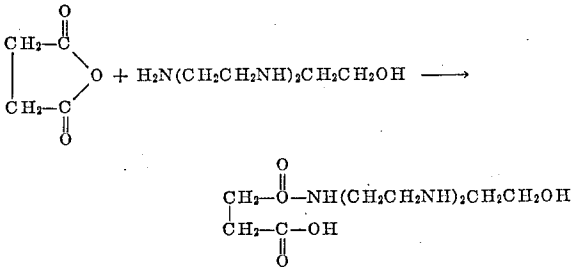
A quantity of 104 grams (1 mole) of hydroxyethyl-ethylenediamine is gradually added at room temperature with stirring to a mixture of 100 grams (1 mole) succinic acid anhydride and 204 grams of distilled water. The reaction is exothermic and the temperature rises rapidly to 85° C. At the completion of the addition, the reaction mixture is stirred at 85° C. for 1 hour. The final product, the succinamic acid of hydroxyethyl-ethylenediamine, containing 50% water, is clear and fluid at room temperature. The following equation illustrates the formation of the reaction product of succinic acid anhydride and hydroxyethyl-ethylenediamine.



Example II

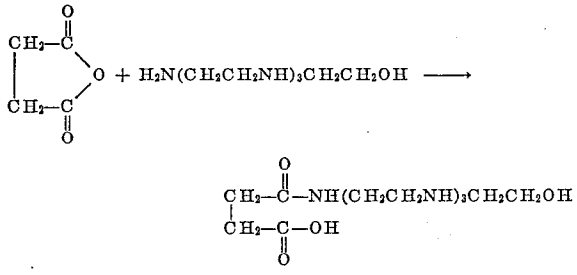
A quantity of 73.5 grams (0.5 mole) of hydroxyethyl-diethylenetriamine is gradually added at room temperature with stirring to a mixture of 50 grams (0.5 mole) succinic acid anhydride and 101.5 grams of distilled water. The reaction is exothermic and the temperature rises rapidly to 85° C. The mixture is then stirred at 95° C. for a period of two hours. The final product contains 50% water and is clear and fluid at room temperature. The following equation illustrates the formation of the

reaction product of succinic acid anhydride and hydroxyethyl-diethylenetriamine.



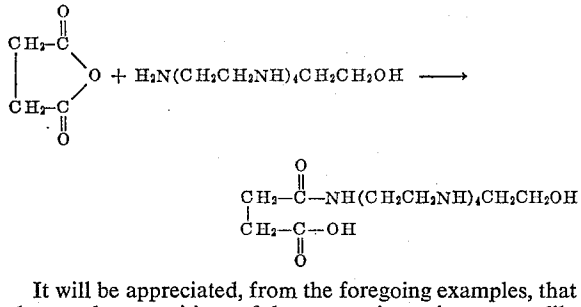
Example III

A quantity of 95 grams (0.5 mole) of hydroxyethyl-triethylenetetramine is gradually added at room temperature with stirring to a mixture of 50 grams (0.5 mole) succinic acid anhydride and 123 grams of distilled water. The reaction is exothermic, and the temperature rises rapidly to 89° C. At the completion of the addition, the mixture is stirred at 95° C., for one hour. The final product contains 50% water, and is clear and fluid at room temperature. The following equation illustrates the formation of the reaction product of succinic acid anhydride and hydroxyethyl-triethylenetetramine.



Example IV

A quantity of 116.5 grams (0.5 mole) of hydroxyethyl-tetraethylenepentamine is gradually added at room temperature with stirring to a mixture of 50 grams (0.5 mole) succinic acid anhydride and 144.5 grams of distilled water, The reaction is exothermic, and the temperature rises rapidly to 86° C. At the end of the addition the mixture is stirred at 95° C. for one hour. The final product, contains 50% water, and is clear and fluid at room temperature. The following equation illustrates the formation of the reaction product of succinic acid anhydride and hydroxyethyl-tetraethylenepentamine.



It will be appreciated, from the foregoing examples, that the novel compositions of the present invention are readily prepared by the addition reaction of succinic acid anhydride and the aforementioned specific type of hydroxyethyl-amine reactants.

Rust tests to determine the suitability of these compounds as corrosion inhibitors are carried out using ordinary steel wool as the corrodible material. Two-gram samples of steel wool are placed in individual clear glass bottles of 8 ounce size and covered with about 200 cc. of aqueous coolant containing from 0.1 to 2.0 percent, by volume, of the corrosion inhibitor. Controlled samples

of steel wool from the same source are tested without including a corrosion inhibitor in the coolant. The bottles are vented to insure air contact with the surface of the water in the bottles. This test is considered to prevent the failure of a corrosion inhibitor at first sign of rust forming on the sample of steel wool.

The following test-results (Table I) are obtained from runs of the corrosion inhibitors in water alone.

TABLE I

Test	Inhibitor	Conc., Vol. Percent	Results
1	None		Rust occurs within 24 hours.
2	Example I	0.5	No rust after 29 months.
3	Example II	0.5	Do.
4	Example III	0.5	Do.
5	Example IV	0.5	Do.

The following tests (Table II) are run using a 50% water-50% isopropanol solution to determine the effectiveness of the corrosion inhibitors of the present invention when used with anti-freeze compounds.

TABLE II

Test	Inhibitor	Conc., Vol. percent	Results
1	None		Rust within 24 hours.
2	Example I	0.5	No rust at end of 1 month.
2A	do	2.0	Do.
3	Example II	0.5	Do.
3A	do	2.0	Do.
4	Example III	0.5	Do.
4A	do	2.0	Do.
5	Example IV	0.5	Do.
5A	do	2.0	Do.

Similar tests are run using a solution of 50% water and 50% ethylene glycol as shown in Table III, below.

TABLE III

Test	Inhibitor	Conc., Vol. percent	Results
1	None		Rust within 24 hours.
2	Example I	0.5	No rust at end of 1 month.
2A	do	2.0	Do.
3	Example II	0.5	Do.
3A	do	2.0	Do.
4	Example III	0.5	Do.
4A	do	2.0	Do.
5	Example IV	0.5	Do.
5A	do	2.0	Do.

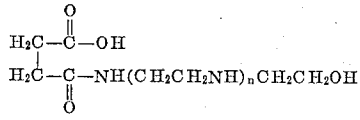
The results obtained by these tests clearly demonstrate the corrosion-inhibiting characteristics of the novel compositions of the present invention. They are effective when present, in minor amounts, in water and in water and anti-freeze solutions. They have been shown to remain effective even after periods of about two and one-half years. On a comparative basis, it is found that when the foregoing experiments are repeated, employing as a corrosion inhibitor a succinamic acid obtained by reacting succinic acid anhydride with ethanolamine, rusting occurs within 24 hours, thus demonstrating the criticality of employing as the amine reactant, an amine of the previously described general formula wherein *n* must have a value of at least 1.

Although the present invention has been described with preferred embodiments it will be understood that various modifications and adaptations thereof may be resorted to, without departing from the spirit and scope of the invention, as those skilled in the art will readily understand.

We claim:

1. A method for inhibiting rusting of ferrous metal surfaces in a cooling system of the type through which an aqueous coolant is circulated, which comprises contacting said metal surfaces with an aqueous coolant consisting

essentially of water and a small amount, sufficient to prevent rusting of said metal surfaces, of a succinamic acid having the formula:



wherein *n* is an integer from 1 to 4.

2. A method as defined in claim **1** wherein said succinamic acid is the hydroxyamine salt reaction product of succinic acid anhydride and hydroxyethyl-ethylenediamine.

3. A method as defined in claim **1** wherein said succinamic acid is the hydroxyamine salt reaction product of succinic acid anhydride and hydroxyethyl-diethylenetriamine.

4. A method as defined in claim **1** wherein said succinamic acid is the hydroxyamine salt reaction product of succinic acid anhydride and hydroxyethyl-triethylenetetramine.

5. A method as defined in claim **1** wherein said succinamic acid is the hydroxyamine salt reaction product of

succinic acid anhydride and hydroxyethyl-tetraethylenepentamine.

6 A method as defined in claim **1** wherein said coolant additionally contains isopropanol.

7. A method as defined in claim **1** wherein said coolant additionally contains ethylene glycol.

8. A method as defined in claim 1 wherein said succinamic acid is present in an amount from about 0.1 to about 10 percent, by volume.

9. A method as defined in claim **1** wherein said succinamic acid is present in an amount from about 0.1 to about 5 percent, by volume.

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