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PROTECTIVE FILM FOR FERROUS METALS

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This invention relates to a composition suitable for forming a water-soluble corrosion-protective film on ferrous metal objects, the method of forming said film on said objects, and ferrous metal objects coated with a film obtained from said composition.

The protection of various ferrous metal objects against rusting or corrosion during storage or warehousing presents a serious problem for industry. Several methods of protecting steel plates, casting, dies, stainless steel and ferrous metals used in cold working or drawing tools, against corrosion prior to the final use of such materials, have been employed. Such methods have been costly and quite inconvenient from the standpoint of application and removal of the coatings prior to the use of the metal object. Such prior methods have involved such means as packing in grease, such as Cosmoline, and more recently by plastic films applied by dipping, rolling or spraying; the main disadvantages of these types of protective coatings are in the cost of the materials used or in the cost and difficulty of removing the grease or plastic coatings to render the metal objects suitable for final use.

The principal objective of the present invention is to provide a protective coating which may be easily removed, when desired, by washing, dipping or spraying the coated metal with water. A coating of this type to be most effective should have the following characteristics. It should be impervious to its corrosive environment, cover the metal completely and should not mar the metal finish. It should form a clear uniform film which is nonhygroscopic, nontacky, and noncrystalline. It should be easy to apply, easy to remove, and leave a clean nongreasy surface after removal.

It has been found that the above requisites may be met by an aqueous solution consisting essentially of "Victamide"; monoammonium phosphate; a gum, exemplified by carboxymethyl cellulose and the like, that is capable of controlling the viscosity of the solution and improving its film-forming properties; a compatible wetting agent or surfactant such as "Aerosol IB" that serves to increase the ability of the coating solution to wet ferrous metal surfaces, and water. In order to obtain the maximum protective effect, the above-mentioned components should be combined in somewhat critical proportions.

The "Victamide" component is a condensation or reaction product of anhydrous ammonia and phosphorus pentoxide in which the reaction is carried out in the presence of excess anhydrous ammonia at a temperature of at least 150° C. for a sufficient period of time to form a product having a total nitrogen to phosphorus mole ratio between 1 and 1.5, with an ammonium nitrogen content of 60–80% of the total nitrogen. The product may be made in the manner described in United States Patent No. 2,122,122 (issued June 25, 1938, to W. H. Woodstock) or by vapor phase reaction of the ingredients under anhydrous conditions. The product will normally have a nitrogen to phosphorus ratio of about 1.25, a P₂O₅ content of approximately 76%, a total NH₃ content of about 22.4%, and nuclear or amide nitrogen calculated as NH₃

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of about 7.0%. Such analysis is typical and is not intended to set the exact composition of the reaction product to be employed in the composition of this invention. "Victamide" is a product sold by Victor Chemical Works and may be designated as an ammonium salt of an amidopolyphosphate.

The ratio of Victamide to monoammonium phosphate and their proportion of the total composition are quite important. It has been found that if the amount, by weight, of monoammonium phosphate is greater than the amount of Victamide, the film-forming quality of the composition is quite inferior because crystallization prevents the forming of an impervious coating. On the other hand, if the amount of Victamide is much greater than the monoammonium phosphate, the film becomes too tacky and sticky to permit the proper handling and stacking of the coated metal objects. We have found that the amount of monoammonium phosphate should be from 80–100% of the amount of Victamide present with the combined amounts of these two components representing from 25–28% of the coating composition. Amounts higher than 28% do not permit the formulation of a clear compatible solution, whereas amounts lower than 24% give solutions which form irregular and non-coherent type films. The preferred composition should contain about 14% Victamide and 13% monoammonium phosphate.

Other components of the coating composition are a gum, a wetting agent and water.

The gum, which serves the purpose of controlling the viscosity of the solution and improving the film-forming property, may be varied to some extent. Carboxymethyl cellulose has been found to be the most effective gum for the purpose. Other materials such as gum tragacanth, starch, polyvinyl alcohol, alginates, etc. may have utility in the composition but are not considered to be as effective as carboxymethyl cellulose. The amount of carboxymethyl cellulose may be varied from about 0.8 to 1.5% with satisfactory results, but the optimum effect is obtained when the coating composition contains about 1%. The preferred carboxymethyl cellulose is one in which from 1.2–1.4 of the hydroxy groups of cellulose are substituted with sodium carboxymethyl (—CH₂COONa) groups, such as that known as Hercules CMC—120H having a viscosity of 100–300 centipoises at 25° C. in 1% concentration.

The wetting agent or surfactant is added for the purpose of increasing the ability of the coating solution to wet the surfaces of slightly soiled ferrous metal surfaces. Selection of a wetting agent which is compatible with the other components of the composition in their necessary concentration is quite critical. The commercial product "Aerosol IB," which is a dibutyl sodium sulfosuccinate (produced by American Cyanamid Co.) has been found to be suitable. The concentration of this surfactant or wetting agent in the coating composition is preferably about 0.2%, but may be increased to about 1% without detrimentally affecting the coating solution.

The concentration of the various components of the coating composition is quite important. In other words, the amount of water in the liquid composition is quite critical. A water content of 70–72% is quite satisfactory. Increasing the water to 74% greatly reduces the rust retarding efficiency of the coating, while a water content of 76% renders the solution quite ineffective for the intended purpose.

The preferred formula for the new coating composition shown above may be modified to some extent without too seriously affecting the nature and efficiency of the coatings produced on steel and ferrous metal objects. The limitations discussed above show that for the most

effective results, the new composition should be formulated within the following composition range:

Victamide	Percent
Monoammonium phosphate	13- 14
Carboxymethyl cellulose	12- 14
Surfactant	0.8- 1.5
Water	0.2- 1.0
	74.0-69.5

In the preparation of the new coating composition, care must be exercised in solubilizing and blending the components to obtain a substantially clear homogeneous solution.

A typical composition and its method of preparation is as follows:

Victamide	Percent
Monoammonium phosphate	14
Carboxymethyl cellulose	13
Dibutyl sodium sulfosuccinate (Aerosol IB)	1
Water	0.2
	71.8
	100.0

First, Victamide is dissolved in water at 60° C., with agitation, to form a clear colorless 40% solution of the Victamide. This requires some time as the Victamide first takes up a portion of the water to form a gel which requires about an hour for complete solution.

Second, a 2% solution of the carboxymethyl cellulose is prepared by dusting dry powdered carboxymethyl cellulose (in this example Hercules CMC-120H) in water agitated at a high rate of shear while heating at a temperature of 70-80° C. Actually, the solution formed is a colloidal suspension in the water in the form of a smooth liquid composition.

Third, dibutyl sodium sulfosuccinate (Aerosol IB) is dissolved in water at about 80° C. to form a 20% solution.

In the compounding of the coating composition, 2000 grams of the above 2% solution of carboxymethyl cellulose, 40 grams of the 20% solution of the surfactant and 40 grams of water were mixed in a blending vessel. Then 1400 grams of the above 40% Victamide solution were added with constant agitation. The solution was heated to 60-70° C. and 520 grams of monoammonium phosphate added while maintaining the temperature and agitation until the monoammonium phosphate is dissolved. The hot solution was filtered through a 300 mesh screen to remove any globules of undissolved carboxymethyl cellulose which may be present. This procedure yields about one gallon of the new liquid coating composition. It had an apparent viscosity of about 400 centipoises at 26° C. and a specific gravity of 1.1700 at 26° C.

In an alternate method of preparation, a dry mixture of carboxymethyl cellulose and the surfactant is made up as a component A containing 83.33% carboxymethyl cellulose and 16.67% of surfactant (Aerosol IB). A dry mixture of Victamide (51.8%) and monoammonium phosphate (48.2%) is made up as component B. These components may be stored or shipped, and the final coating solution prepared, when desired, by dissolving, for example, 1.2 parts by weight of component A in 71.8 parts water at about 80-90° C. under strong agitation for a sufficient period of time (about 15 minutes) to form a smooth solution. While maintaining the temperature and agitation 27.0 parts of component B is added and stirring continued until a clear viscous solution of the coating composition is obtained.

The coating solutions prepared as in the above examples may be applied to steel plate or other ferrous metal objects by spraying, brushing or dipping without prior special preparation of the surfaces to be coated. The coating solution has high surface activity which permits it to spread smoothly over areas which have been soiled

by fingerprints and other shop soil. After drying the resulting coating is a non-crystalline, transparent, tenacious film which affords temporary protection of the coated surface against atmospheric corrosion during storage in the warehouse or shop. Tools of various kinds may be protected by such coating while being held in shop supply rooms, hardware stores, and the like. When ready for final or consumer use the coated steel plates, tools, etc. may be washed with hot water to remove the coating and present a clean, grease-free metal surface. This secondary advantage of the coating composition is due to the normal deterative qualities of its phosphate components.

After applying the coating solution to the metal surfaces, it should be dried for a sufficient period to produce a dry, nontacky film at a temperature not exceeding 100° C. For example, the following conditions have been found satisfactory:

- (a) 90 minutes at room temperature
- (b) 12 minutes at 90° C.
- (c) 12 minutes with circulating air at 70° C.

The above drying conditions may be varied as long as the temperature is below 100° C. Higher temperatures cause the protective film to crack and lose its effectiveness.

The effectiveness of the coating in preventing the rusting of steel plates is illustrated by the following accelerated corrosion tests.

A number of mild steel (SAE 1020) panels, 2" x 6" in size where cut from a sheet as received without any prior cleaning except the wiping of the surface with a paper towel. Part of these test panels were dipped in the solution prepared in Example 1 and allowed to air dry for 24 hours to form a dry coating thereon. These test pieces and several which were not treated were suspended in a large closed jar above a 50% aqueous slurry of calcium hypochlorite. The atmosphere above the hypochlorite is highly corrosive and permits the carrying out of accelerated corrosion tests. The test panels were held in this atmosphere for 16 hours at room temperature and then examined for surface rust. The coated panels showed no corrosion, whereas the surfaces of the untreated panels were completely covered with rust.

Some of the coated panels were then dipped in hot water at about 90° C. to remove the coating. These panels were found to be free of any oily or dirt contamination which was present on the original untreated panels.

The term "carboxymethyl cellulose" shall hereinafter refer to carboxymethyl cellulose as well as alkali salts thereof.

We claim:

1. A composition suitable for use in producing a water-soluble, substantially clear, uniform, nonhygroscopic, nontacky, noncrystalline rust prevention coating on ferrous metal objects consisting essentially of: 13-14% by weight of ammonium salt of an amidopolypolyphosphate; 12-14% by weight of monoammonium phosphate; 0.8-1.5% of a member of the group consisting of carboxymethyl cellulose, natural gums, starch, polyvinyl alcohol, and alginates which serve to control the viscosity and film-forming properties of the composition; 74-69.5% by weight water; and dialkyl alkali metal sulfosuccinate in an amount not greater than 1% by weight.

2. A composition suitable for use in producing a water-soluble, substantially clear, uniform, nonhygroscopic, nontacky, noncrystalline rust prevention coating on ferrous metal objects consisting essentially of: 13-14% by weight of ammonium salt of an amidopolypolyphosphate; 12-14% by weight of monoammonium phosphate; 0.8-1.5% of carboxymethyl cellulose which serves to control the viscosity and film-forming properties of the composition; 74-69.5% by weight water; and dialkyl alkali metal sulfosuccinate in an amount not greater than 1% by weight.

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3. The method of forming a protective film on steel and ferrous metal objects which comprises applying a coating of the composition of claim 1 to the surface of the metal object and drying the coating at a temperature of not over 100° C.

4. A steel or ferrous metal object coated with a water-soluble film obtained from the composition set forth in claim 1.

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