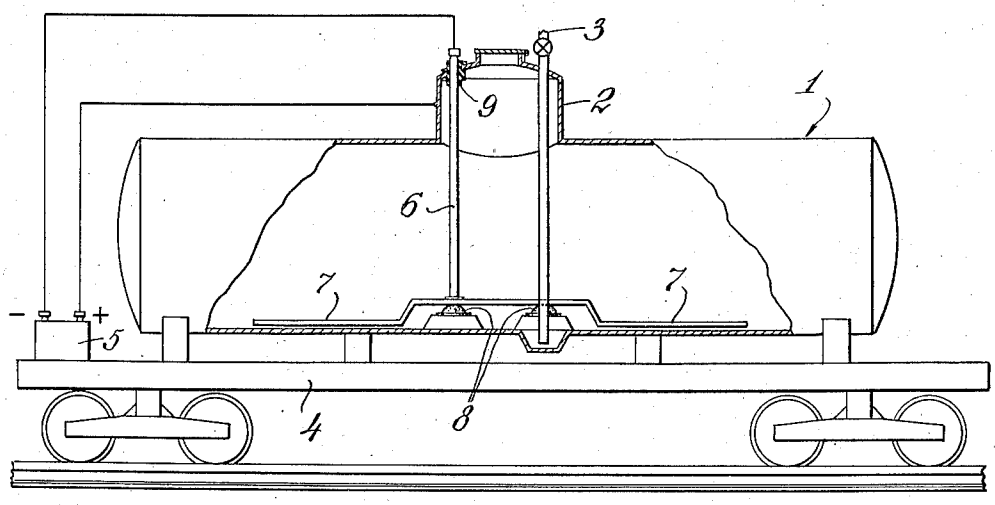
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PREVENTING CORROSION OF FERROUS METALS BY AMMONIACAL SOLUTIONS OF AMMONIUM NITRATE

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PREVENTING CORROSION OF FERROUS METALS BY AMMONIACAL SOLUTIONS OF AMMONIUM NITRATE

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This invention relates to a process for preventing corrosion of a ferrous metal in contact with aqueous solutions of ammonium nitrate and particularly of aqueous ammoniacal solutions of ammonium nitrate.

For many years past aqueous ammoniacal solutions of ammonium nitrate have been on the market particularly as a material for use in the production of fertilizers. The tank cars commonly available for the shipment of these solutions as well as the equipment in which they are handled by the fertilizer manufacturers are made of various ferrous metals such as iron, steel and special alloy steels. These solutions are, however, corrosive towards the ferrous metals. For example, a hot rolled, mild steel in contact with a solution containing 60% ammonium nitrate, 20% ammonia and 20% water was found to be corroded at the rate of 0.55 inch penetration per year. A saturated aqueous solution of ammonium nitrate containing as little as 1% ammonia was found to corrode this steel at the rate of 0.57 inch penetration per year.

In view of the highly corrosive nature of these solutions, extensive research has been carried out to find methods for sufficiently reducing the rate of corrosion of ferrous metals by these solutions so that it would be practicable to store, ship and handle them in iron and steel equipment. One type of method developed involved the addition to the solution of certain inhibitors. For example, as disclosed in U. S. P. 2,215,077, which issued September 17, 1940, to Beekhuis and Macomber, the addition of a small amount of certain sulfur compounds, e. g., ammonium thiocyanate, greatly reduced the rate of attack of iron and steel by the solution. While the addition to the solution of such inhibitors has made it practicable to handle the solutions in apparatus made of ferrous metals, even with these inhibitors present there is generally some residual small rate of corrosion of the metal by the solution. Furthermore, under some conditions of production, shipment or use of these solutions, it would be advantageous if some means were available whereby corrosion of ferrous metals by the solutions could be prevented without having to add a foreign material to the solution, with the attendant expense of providing the foreign material for addition to the solution and contamination of the solution therewith.

It has also been proposed, in U. S. P. 2,135,160, which issued November 1, 1938, to Herman A. Beekhuis, Jr., to passify a ferrous metal by treatment with a strongly oxidizing agent such as chromic acid and then to contact the passified metal with an ammoniacal solution of ammonium nitrate in which a chromate has been dissolved to maintain the passivity of the ferrous metal in contact with the solution. As in the methods described above, a disadvantage of this method is the necessity for dissolving in the ammonium nitrate-ammonia solution a foreign material.

While non-ammoniacal aqueous solutions of ammonium nitrate do not have the high corrosiveness towards ferrous metals that the ammoniacal solutions do, nevertheless such solutions are corrosive towards these metals. For example, a saturated aqueous ammonium nitrate solution was found to corrode a hot rolled, mild steel at the rate of 0.006 inch penetration per year and the presence in such a solution of as little as 0.1% nitric acid increased the rate of corrosion to 0.047 inch penetration per year. Such non-ammoniacal ammonium nitrate solutions are frequently encountered in commercial practice and in some cases it is not possible to employ in such solutions certain inhibitors which can be successfully used in the ammoniacal solutions. For example, in producing ammonium nitrate by reaction of ammonia and nitric acid fed into a saturator vessel containing an aqueous ammonium nitrate solution, it is frequently desirable to maintain a low concentration of nitric acid in the solution. It is impracticable to dissolve in such a solution a thiocyanate as an inhibitor and yet it would be advantageous to have available some means for reducing the rate of attack by the acid-ammonium nitrate solution of the ferrous metal used as a material of construction for the saturator.

It is an object of this invention to provide a method for reducing the rate of corrosion of a ferrous metal by an aqueous ammonium nitrate solution which may be used without the need for contaminating the solution by addition thereto of foreign substances, or may be used in conjunction with the use of chemical agents, such as inhibitors, to further reduce the residual slow rate of corrosion of a ferrous metal by the inhibited solution.

We have discovered that by making the surfaces of a ferrous metal in contact with an aqueous solution of ammonium nitrate the anode in an electric circuit which includes a cathode in contact with the solution and a source of direct electric current which imposes upon the anode an electric potential within a particular range, corrosion of that ferrous surface is completely prevented or at least greatly reduced. The range of voltages within which the potential imposed on

the anode must lie will vary somewhat, depending upon the specific metal of which the anode is constructed, composition of solution with which it is in contact, etc. In all cases the voltage is below the decomposition potential of the solution in contact with the inert electrode used as a cathode and the ferrous metal of the anode and above that at which the flow of current acts to promote corrosion of the ferrous metal surfaces of the anode.

In case the container for the ammoniacal ammonium nitrate solution is of large size, such as a tank car or storage tank of the size customarily used for the shipment and storage of these solutions, in which it is impracticable to employ cathodes having surface areas of the same order of magnitude as the surface areas of the container which are to be protected against corrosion by the solution, the application to the electrodes of voltages below the decomposition potential of the solution will not of itself be sufficient to prevent corrosion of an initially active metal. In such cases, however, we have discovered that by first passivating the metal surfaces and thereafter making the passive surfaces the anode in contact with the ammoniacal ammonium nitrate solution, applied voltages below the decomposition potential of the solution and above that at which the flow of current acts to promote corrosion of the ferrous metal surfaces of the anode will not only maintain the anode surfaces passive against attack by the solution but will even increase the effectiveness of the passive film on the metal surfaces.

Any method for passivating a ferrous metal may be used to effect the initial passivation of the ferrous metal surfaces. Preferably, the metal surfaces may be treated with an oxidizing agent such as a solution of chromic acid, dichromate, potassium permanganate or nitric acid, as disclosed in U. S. P. 2,135,160. The metal surface may be washed with a solution of a strong oxidizing agent, e. g., 10% $KMnO_4$ + 5% chromic acid (CrO_3) solution in water. Even relatively milder oxidation treatments are effective in conjunction with the electrolytic treatment of the containers with the ammoniacal ammonium nitrate solution in contact with the metal surfaces as herein described. Thus, the oxidizing treatment to passivate the metal may be accomplished by steaming the metal surface at temperatures of at least 85° C. and then air-drying the surfaces.

In the case of a tank car in which the interior surface of the container for an aqueous ammoniacal solution of ammonium nitrate has been passified and then made an anode with a cathode dipping into the solution in the central part of the tank, the potential applied to the tank and cathode therein should be at least 1.25 volts and not above 2.2 volts. Somewhat below 1.25 volts the flow of electrical current between the electrodes in the solution acts to promote corrosion of the ferrous metal anode by the solution. Above 2.2 volts the current acts to electrolytically decompose the solution, liberating gaseous products which, accumulating in the free space in the container above the liquid surface, present a hazardous condition. Furthermore, decomposition of the material in the solution represents a loss of valuable material. With a voltage in the range 1.25 to 2.2 volts, practically complete protection of the ferrous metal surfaces is obtained without the difficulties, pointed out above, encountered at lower or higher potentials.

In employing the above described means for preventing corrosion of ferrous metals by aqueous ammonium nitrate solutions the cathode immersed in the solution may be of any electrically conducting material suitably resistant to attack by the solution. For example, aluminum is a suitably inert metal for the cathode in ammoniacal, acid or in neutral ammonium nitrate solutions.

Despite the relatively low potential applied to the electrodes in contact with the solution, protection of the surfaces of large containers for the solution, such as tank cars or storage tanks or the commercial equipment of plants in which the solutions are produced or used, is obtained without having to unduly complicate the size and arrangement of the cathode in the container. Protection of the ferrous metal anode surfaces may be obtained with cathodes of very small surface area as compared with the anode surface area and unsymmetrically disposed with respect to the anode surfaces.

Making the container for the ammonium nitrate solution the anode under the conditions described above has been found to prevent or suitably reduce the rate of corrosion of the ferrous metal of which the container is constructed below the level of liquid in the tank. Corrosion may start, however, at surfaces above the liquid level and then extend downwardly to and below the liquid level. This is particularly liable to happen in the case of tank cars in which upper surfaces may be intermittently wetted with the solution when the car is in transit. Corrosion of the container surfaces above the liquid level which may be wetted by the liquid may be inhibited by having present in the ammonium nitrate solution an inhibitor.

To counteract the tendency of the surfaces at and above the liquid level to corrode, we prefer to paint these surfaces with a corrosion-resistant paint after giving the metal surface an oxidizing passivating treatment in addition to making the surfaces in contact with the solution an anode under the above described conditions. Any paint resistant to attack by an aqueous ammonium nitrate solution may be employed. A chlorinated rubber paint, marketed under the trade name "Tornesit" has been found particularly good for use in a container for ammoniacal ammonium nitrate solutions. Other chlorinated rubber paints, such as that sold under the trade name "Densol" or a coat of red lead primer under a coat of a chlorinated rubber paint sold under the trade name "Socony White" may be used. The entire interior surface of the tank car may, of course, be painted in the above manner or the painted area may be confined to a strip immediately above and below the normal liquid level. This painting of the tank not only prevents corrosion immediately above the solution level but reduces the amount of current which must be supplied in anodically protecting the metal surfaces by the process of our invention.

The simplicity of the apparatus required for protecting ferrous metal surfaces against attack by ammonium nitrate solutions in accordance with the process of our invention makes that process peculiarly adaptable for use in connection with the tank cars in which aqueous ammonium nitrate solutions are shipped. The accompanying drawing illustrates diagrammatically an apparatus suitable for carrying out the process of this invention in such a tank car.

With reference to the drawing the numeral 1

indicates the tank for containing the solution. As is customary in tank cars for liquids, the tank is provided with a dome 2 through which an unloading pipe 3 passes to a sump in the bottom of the tank. This tank is carried by a conventional running gear 4 which also carries a storage battery 5. This storage battery may comprise one or more 2-volt lead storage cells or about 1.5 volt Edison alkaline batteries for delivery of a direct electric current at a potential of 2 or 1.5 volts, respectively. An aluminum rod 6 extends through a fitting 9 in dome 2 electrically insulating the pipe from the dome and reaches downwardly through the interior of tank 1 to near the bottom of the tank. Preferably a second aluminum rod 7 extends for some distance along the bottom of the tank and very close thereto. Rod 7 is in electrical contact with pipe 6 and is supported on electrically insulating supports 8. While pipe 7 is not a necessary feature of the apparatus, it is desirable to insure protection of the tank when practically empty of solution. The positive terminal of battery 5 is electrically connected with tank 1 either directly, as shown in the drawing, or through the running gear of the car. The negative terminal of battery 5 is electrically connected with the top of rod 6. Thus, when an aqueous ammoniacal solution of ammonium nitrate is introduced into tank 1 an electric circuit is completed through the solution in contact with tank 1 as anode and rod 6, and rod 7 when this rod is included in the apparatus, as cathode. A potential difference of 2 or 1.5 volts is thus applied to the anode and cathode, depending upon the type of battery used.

In employing the process of this invention for preventing corrosion, an ordinary tank car supplied for shipping an ammonium nitrate solution was provided with an aluminum cathode consisting of a rod passing through an insulator in the dome in the car with the lower end of the rod extending within six inches of the bottom of the car. This rod corresponded to rod 6 in the apparatus of the drawing. As a source of direct electric current two lead storage batteries connected in parallel were used. The positive terminals of the batteries were connected with the dome of the car and the negative terminals with rod 6.

The interior of the car was first steamed at temperatures above 85° C. and then air was admitted to dry the car while it remained at these elevated temperatures. The dome was then painted on the inside with two coats of "Tornesit." The car was filled with solution containing 60% ammonium nitrate, 20% ammonia and 20% water to which had been added 0.1% ammonium thiocyanate and 0.5% As_2O_3 as an arsenite. The car was filled until the level of liquid rose into the dome of the car.

While the car was being filled, the current passing between the car tank and aluminum cathode rose to a maximum of about 17 amperes. Twenty-four hours after loading the current had decreased to about 6 amperes and it continued to drop thereafter so that the daily average current had dropped to 0.7 ampere on the 12th day. With a surface area of the tank in contact with the solution of about 850 square feet, this was equal to about 0.0008 ampere of current per square foot of anode area. At the end of twelve days, the battery was disconnected from the car and for a period of ten days the tank remained passive and unattacked by the solution. On longer standing, however, the solution started to corrode the interior surfaces of the tank.

As shown by this example, in utilizing the process of our invention it is not necessary, although in some cases it may be convenient and desirable, to continuously maintain an electric potential on the container as an anode to prevent corrosion of the container surfaces. Contact of the ferrous metal surfaces as anode with the ammonium nitrate solution under the conditions described above for carrying out a specific procedure embodying our invention develops in the passive surfaces of the ferrous metal a passivity of a nature such that it will persist for a period of time after the source of electric current is disconnected from the container and prevent substantial corrosion of the container for some time after the electric current has ceased to flow. Accordingly, our invention contemplates a procedure in which a container which is to be filled and later emptied of the ammonium nitrate solution, as in shipping the solution in a tank car, is made the anode in contact with the solution for a period of time to develop this passivity which will persist and will itself prevent attack of the metal by the solution. The electric current is then cut off from the container, although the solution is still left in contact with the surfaces thereof. Or a container for the ammonium nitrate solution may be connected as anode to a source of electric current to develop this resistance to attack by the solution and thereafter the electric potential is applied to the container only intermittently, as needed to maintain the passivity of the container surface against attack by the solution.

The procedure of the above example may be varied in numerous ways without departing from the scope of our invention. For example, the interior of the tank car may be dried out in a current of air, without first steaming it, and then painted with "Densol." Under these conditions, after filling the car with the ammoniacal solution of the above example, the average daily current passing at 2 volts potential applied to the tank was 7 amperes for the first day. By the tenth day the current had fallen to a daily average of about 3 amperes. When the car was unloaded on the 16th day inspection of the surfaces of the tank showed no corrosion.

Hot rolled, mild steel which, as pointed out above, was corroded by a solution containing 60% ammonium nitrate, 20% ammonia and 20% water at the rate of 0.5 inch penetration per year showed no corrosion when first passivated and then made the anode in contact with the solution with a potential of 1 volt applied to the anode and a cathode in contact with the solution. The average current passing was only 0.00035 ampere per square foot of anode surface. In contact with the same solution containing 0.1% ammonium thiocyanate, this steel was corroded at the rate of 0.011 inch penetration per year but when passivated and made the anode at an applied potential of 1 volt with respect to a cathode immersed in the solution no corrosion occurred. The average current passing was 0.00011 ampere per square foot of anode surface. A saturated aqueous solution of ammonium nitrate containing 0.1% nitric acid corroded this steel at the rate of 0.047 inch penetration per year. When the steel was made the anode in contact with the solution with a potential of 1.2 volts applied to the electrodes the rate of corrosion dropped to 0.004 inch penetration per year

with an average current passing of 0.0039 ampere per square foot of anode surface. These applied potentials of 1 to 1.2 volts were effective to substantially prevent corrosion of previously passivated anodes with areas much smaller than the interior of tank cars used for shipping ammoniacal solutions of ammonium nitrate. The applied potentials which are effective to prevent corrosion of the anode surfaces depend upon the type and size of the equipment in which surfaces are to be protected by the process of this invention since the interfacial potential drop between anode and solution depends upon the size and arrangement of the anode and cathode. The material used for the cathode may have an influence upon the range of suitable potentials applied to the electrodes. Generally, the larger the apparatus, the higher the applied potentials which will be used, although in all cases the applied potentials are low, e. g., the 1.25 to 2.2 volts which are suitable for tank cars.

The process of protecting a ferrous metal against corrosion by an aqueous ammonium nitrate solution disclosed and claimed in this application is disclosed and claimed in our copending application Serial No. 380,995, filed February 28, 1941, for protecting ferrous metals against corrosion by aqueous solutions of electrolytes which liberate oxygen at the anode when electrolytically decomposed.

We claim:

1. The process for protecting passivated surfaces of a ferrous metal against corrosion by an aqueous ammonium nitrate solution contacted therewith, said ferrous metal being one subject to corrosion by said solution, which comprises making said ferrous metal the anode in an electric circuit completed through said solution in contact with said passivated surfaces of the metal, an inert cathod in said solution and a source of direct current at a voltage which is below that at which said solution is electrolytically decomposed in contact with said anode and cathode and is above those at which the flow of current increases the rate of corrosion of said surfaces by the solution in contact therewith as compared with the rate of corrosion of the same surfaces in contact with the solution without the application thereto of an electrical potential.

2. The process for protecting a ferrous metal against corrosion by an aqueous ammonium nitrate solution contacted therewith, said metal being one subject to corrosion by said solution, which comprises passivating the surface of said metal which is to be contacted with said solution by treating said surfaces with an oxidizing agent, thereafter placing said surfaces in contact with said solution and making said surfaces the anode in an electric circuit completed through said solution in contact with said metal, a cathode in said solution and a source of direct current at a voltage which is below that at which said solution is electrolytically decomposed in contact with said anode and cathode and is above those at which the flow of current increases the rate of corrosion of said surfaces by the solution in contact therewith as compared with the rate of corrosion of the same surfaces in contact with the solution without the application thereto of an electrical potential.

3. The process for protecting against corrosion by an aqueous solution of ammonium nitrate a container for said solution in which surfaces exposed to contact with the solution are composed of a passivated ferrous metal which is subject to corrosion by the solution, which comprises immersing an inert electrode in the solution in said container, electrically connecting the negative and positive terminals of a source of direct electric current to said electrode and to said ferrous metal, respectively, thereby making said passivated ferrous metal surfaces the anode in an electric circuit completed through said solution, and by means of said source of electric current maintaining between said ferrous metal and said inert electrode a potential difference which is below that at which said solution is electrolytically decomposed in contact with said ferrous metal surfaces and said inert electrode and is above those at which the flow of current increases the rate of corrosion of the ferrous metal surfaces by the solution in contact therewith as compared with the rate of corrosion of the same surfaces in contact with the solution without the application thereto of an electrical potential.

4. The process for protecting the ferrous metal surfaces of a container for an aqueous solution of ammonium nitrate against corrosion thereby, said ferrous metal being one subject to corrosion by said solution, which comprises passivating said surfaces by treating it with an oxidizing agent, thereafter contacting said surfaces with said solution and immersing in the solution an aluminum electrode of small size and unsymmetrically disposed with respect to said surfaces of ferrous metal, electrically connecting the negative and positive terminals of a source of direct electric current to said electrode and to said ferrous metal, respectively, thereby making said ferrous metal surfaces the anode in the electric circuit completed through said solution, and by means of said source of electric current maintaining between said ferrous metal and said aluminum electrode a potential difference below that at which said solution is electrically decomposed in contact with said ferrous metal and aluminum electrode and above those at which the flow of current increases the rate of corrosion of the ferrous metal surfaces by the solution as compared with the rate of corrosion of the same surfaces in contact with the solution without the application of the potential.

5. The process for protecting a ferrous metal against corrosion by an aqueous ammonium nitrate solution contacted therewith, said metal being one subject to corrosion by said solution, which comprises subjecting the surfaces of said metal which are to be contacted by said solution to an oxidizing passivating treatment, coating thus treated surfaces with a paint resistant to attack by said solution, and making the thus treated and coated ferrous metal the anode in an electric circuit completed through said solution in contact with said surfaces, a cathode in said solution and a source of direct current at a voltage which is below that at which said solution is electrolytically decomposed in contact with said ferrous metal surfaces and said cathode and is above those at which the flow of current increases the rate of corrosion of said metal by the solution in contact therewith as compared with the rate of corrosion of the same surfaces in contact with the solution without the application thereto of an electrical potential.

6. The process for protecting a container for an aqueous solution of ammonium nitrate against corrosion thereby, said container having interior surfaces of a ferrous metal subject to corrosion by said solution which comprises subjecting said interior surfaces of said container

to an oxidizing passivating treatment, coating thus treated surfaces adjacent to and above the level of liquid to be placed in said container with a paint resistant to attack by said solution, introducing said solution into the thus treated container, immersing an electrode in the solution in said container, electrically connecting the negative and positive terminals of a source of direct electric current to said electrode and to said ferrous metal, respectively, thereby making the ferrous metal of the container which is in contact with said solution the anode in the electric circuit completed through said solution, and by means of said source of electric current maintaining between said ferrous metal and said electrode a potential difference below that at which said solution is electrically decomposed in contact with said ferrous metal and said electrode and above those at which the flow of current increases the rate of corrosion of the ferrous metal surfaces by the solution in contact therewith as compared with the rate of corrosion of the same surfaces in contact with the solution without the application of the potential.

7. The process for preventing corrosion of a tank car during shipment therein of an aqueous ammoniacal solution of ammonium nitrate, the surfaces of said tank car exposed to contact with said solution being composed of a ferrous metal subject to corrosion by the solution, which comprises passivating said ferrous metal surfaces by treatment with an oxidizing agent, introducing said solution into said tank car and into contact with the thus passified surfaces, electrically connecting said ferrous metal surfaces to the positive terminal and electrically connecting an aluminum cathode immersed in the solution in the tank car to the negative terminal of a source of direct current at a voltage of 1.25 to 2.2 volts thereby making said ferrous metal surfaces of said tank car the anode in a circuit completed through said solution and the cathode and substantially preventing corrosion of the ferrous metal surfaces by said solution.

8. The process for preventing corrosion of a tank car during shipment therein of an aqueous ammoniacal solution of ammonium nitrate, said tank car having surfaces exposed to contact with said solution which are composed of a ferrous metal subject to corrosion by the solution, which comprises passivating said surfaces by treatment with an oxidizing agent, introducing said solution into said tank car the surfaces of which have been thus passified, immersing in the solution in said tank car an aluminum electrode of small size and unsymmetrically disposed with respect to said surfaces of ferrous metal, and electrically connecting the negative and positive terminals of a source of direct electric current at a potential of 1.25 to 2.2 volts to said electrode and to said ferrous metal, respectively, thereby making said ferrous metal surfaces the anode in the electric circuit completed through said solution and preventing corrosion of the ferrous metal in contact with said solution.

9. The process for preventing corrosion of a tank car during shipment therein of an aqueous ammoniacal solution of ammonium nitrate, said tank car having surfaces exposed to contact with said solution which are composed of a ferrous metal subject to corrosion by the solution, which comprises subjecting said surfaces to an oxidizing passivating treatment, coating thus treated surfaces adjacent to and above the surface level of liquid to be placed in said tank car with a paint resistant to attack by said solution, electrically connecting said ferrous metal to the positive terminal of a source of direct current at a potential of 1.25 to 2.2 volts, and completing an electric circuit including said ferrous metal as an anode in contact with said solution and an aluminum cathode immersed in the solution and electrically connected with the negative terminal of said source of direct current and preventing corrosion of the ferrous metal in contact with said solution.

10. The process for loading and preparing a tank car for shipment therein of an aqueous ammoniacal solution of ammonium nitrate, said tank car having surfaces of ferrous metal to be exposed to contact with said solution, which metal is subject to corrosion by said solution, and said tank car being provided with an aluminum electrode adapted to be immersed in the solution, which comprises passivating said surfaces by treating them with an oxidizing agent, electrically connecting said electrode and said ferrous metal of the tank car to the negative and positive terminals, respectively, of a source of direct electric current at a potential of 1.25 to 2.2 volts, introducing said solution into said tank car to complete an electric circuit through the solution between said electrode and the ferrous metal surfaces of the tank car and after introduction of said solution into the tank car maintaining said electrode and surfaces of the tank car electrically connected to said source of current for a substantial period of time long enough to impart to said ferrous metal surfaces a passivity against corrosion by said solution which persists when the metal surfaces of the tank car are electrically disconnected from said source of electric current.

11. The process for protecting passivated surfaces of a ferrous metal against corrosion by an aqueous ammonium nitrate solution contacted therewith, said ferrous metal being one subject to corrosion by said solution, which comprises making said ferrous metal the anode in an electric circuit completed through said solution in contact with said passivated surfaces of the metal, an inert cathode in said solution and a source of direct current at a voltage which is below that at which said solution is electrolytically decomposed in contact with said anode and cathode and is above those at which the flow of current increases the rate of corrosion of said surfaces of ferrous metal by the solution in contact therewith as compared with the rate of corrosion of the same surfaces in contact with the solution without the application thereto of an electrical potential, and incorporating in said solution an inhibitor of corrosion of said ferrous metal by the solution.

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