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# CORROSION IN AEROSOLS

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# Corrosion in Aerosols

**Corrosion pitfalls in aerosols cut by knowledge of electrochemical forces involved; extensive tests, and choice of right propellant, solvent, active ingredient, packaging medium, are key to stability.**

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ALTHOUGH there are hundreds of successful aerosol products, many potential aerosols never reach the market and others fail after they are marketed. These products fail for a variety of reasons. Product instability, low market potential, lack of appeal, cost, and container corrosion are some of the factors that can be involved. There are many examples of container corrosion in aerosols and, in many instances, this corrosion can be explained on the basis of the fundamental principles governing corrosion. It is the purpose of this paper to discuss some of these corrosive aerosol systems along with some of the theory that applies to these systems.

The possibility of container corrosion must be considered whenever any aerosol product is packaged in a metal container. If an aerosol product leaks from the container, either before it reaches the consumer or during its use by the customer, the product was not tested sufficiently and should never have been placed on the market. Leakage is an extreme example of corrosion, but even very light container corrosion can mean failure in the case of a sensitive product.

There are a number of ways in which the possibility of packaging a corrosive product in a metal container can be lessened. One is to carry out adequate storage stability tests. Another is to take advantage of the knowledge that is already available in the aerosol field. Aerosol loaders and suppliers, such as the valve, container, and propellant manufacturers, have accumulated over the years a vast amount of information about the storage stability of many aerosol products. Quite often, the stability of an aerosol product can be predicted with considerable accuracy from the results already available with similar products. A third way is to become familiar with the aerosol systems known to cause corrosion and to understand why these systems do cause corrosion.

Most corrosion reactions are electrochemical in nature. Such a reaction involves the presence of an electrolyte, two electrodes, and a potential difference between the electrodes. An electrolyte is a liquid which contains ions and therefore is capable of conducting an electric current. Water is an example of an electrolyte, and the more ions it contains, the better a conductor it is. The electrodes may be formed by two different metals, such as tin and iron in tinplate containers; or the electrodes may consist of two different locations on the same piece of metal. In either case, it is necessary to have a path, such as a piece of wire, for the electricity to flow between the electrodes in order to complete the circuit. (2).

In electrochemical corrosion, a number of reactions take place: positively charged metallic ions leave the anode and enter the electrolyte solution. This is the action that results in erosion and ultimate destruction of the metal that is the anode. At the same time, the electrons that are left as a result of the loss of the positively charged metallic ions to the solution, flow from the anode to the cathode through the path described above. After the electrons reach the cathode, they meet and neutralize positively charged hydrogen ions which travel through the electrolyte to the cathode. The neutralization of the hydrogen ions by the electrons in the cathode forms hydrogen molecules. All of these reactions occur simultaneously, but most of the corrosion in the system occurs at the anode where the metal ions go into solution.

### *Common Corrosive Systems in Tinplate*

Many materials or combinations of materials will cause corrosion in tinplate containers. Some of these are so obviously corrosive to metal that they would never be considered for aerosol products. Others, however, cause corrosion only under certain conditions. The present discussion is concerned primarily with examples of the latter group. The particular materials or mixtures of materials which will be covered are:

1. Aerosol Propellants
   a. Propellant 11 and anhydrous ethyl alcohol;
   b. Propellant 11 and aque-

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ous ethyl alcohol;
c. Propellant 11 and water;
d. Propellant 21 and alkalies; and
e. Propellant 22 and alkalies:
2. Chlorinated solvents;
3. Surface active agents ;
4. Water; and
5. Acids, salts and alkalies

### *Aerosol Propellants*

There is a wide variety of propellants available for the aerosol industry. These propellants differ considerably in their stability, and therefore their reactivity. Some of these materials, such as propellant 12 and propellant 114, are so stable that they can be used in almost any type of aerosol product without fear of decomposition. Others, such as propellant 11, will react under specific circumstances to give corrosive reaction products. These specific circumstances are discussed in the paragraphs below.

### *Propellant 11*

Propellant 11 is one of the most useful of the propellants. Its low toxicity, nonflammability, and high solvent power make it desirable for many aerosol products. Propellant 11 is particularly useful as a vapor pressure depressant for propellant 12. However, propellant 11 can react in alcoholic, aqueous alcoholic, or aqueous systems to give products that cause corrosion.

*Reaction of propellant 11 with anhydrous ethyl alcohol:* Aerosol products such as hair sprays, room deodorants, colognes, etc. are generally formulated with combinations of propellant 11 and anhydrous ethyl alcohol. Since these products account for the major proportion of aerosols on the market, it is obvious that, in general, they have not presented much of a corrosion problem. However, under certain conditions, such as very low air concentration in the product, propellant 11 can react with ethyl alcohol via a free radical mechanism to give acetaldehyde, hydrogen chloride, and propellant 21 as the primary reaction products.

$$CCl_3F + C_2H_5OH \rightarrow CH_2CHO + HCl + CHCl_2F$$

Secondary reactions form acetal, ethyl chloride and water. The presence of acid, moisture, and tinplate establish the conditions necessary for electrochemical corrosion. When the concentration of hydrogen ions becomes sufficiently high, corrosion of the container occurs with the formation of hydrogen molecules and the evolution of hydrogen gas at the cathodic area. (2).

Oxygen has a profound effect upon the free radical reaction between propellant 11 and ethyl alcohol. Low concentrations of oxygen catalyze the reaction because oxygen is a free radical catalyst. However, high concentrations of oxygen have an opposite effect and inhibit the reaction as a result of the formation of inactive peroxy compounds which interrupt the chain reaction. (1).

Besides its effect upon the free radical reaction, oxygen also is known to play a complex role in corrosive systems in general. For example, oxygen acts as a cathode depolarizer. Thus, when acids react with metals, hydrogen accumulates at the cathode and slows down the corrosion because the accumulated hydrogen acts as a barrier and prevents other hydrogen ions from reaching the cathode. This process is called polarization. Oxygen can react with the accumulated hydrogen to form water or hydrogen peroxide and hydroxyl ions (2) and the removal of the hydrogen from the cathode allows the corrosion reaction to continue. This effect of oxygen is called cathodic depolarization. Due to these reactions, high concentrations of oxygen tend to promote corrosion.

Tin and iron are very close in their electrochemical behavior and slight variations in the environment can determine which metal is the cathode and which is the anode. The concentration of oxygen is one of the environmental variables that determines the polarity of the system and this is another important effect of oxygen (8). The reason that the polarity of the tinplate system is so important is this: In tinplate containers, the area of tin exposed to the product is very large, compared with the area of the iron, which is available only as a result of imperfections or pinholes in the tinplate. When a cathode with a large area is coupled to an anode with a small area, severe electrochemical corrosion can occur because the polarizing hydrogen which plates out on the cathode is spread out over a relatively large area and is easily removed by reaction with oxygen (2). Corrosion is promoted when hydrogen is removed by depolarization.

In electrochemical corrosion, the attack on the metal occurs primarily at the anode. Therefore, if the tin in a tinplate container is cathodic and the iron is anodic, the corrosive attack is concentrated on the relatively small areas of the iron. This results in pinholing. In addition, the unfavorable relationship of the large cathodic area of the tin and the small anodic area of the iron increases the rate of corrosion so that pinholing can occur within a short time. However, if the tin is anodic and the iron is cathodic then the tin is attacked initially instead of the iron and generalized detinning occurs rather than pinholing. Also, the favorable relationship of the small area of the cathode (the iron) to the large area of the anode (the tin) promotes a low rate of corrosion.

At low concentrations of oxygen in tinplate containers, the tin is anodic and the iron is cathodic. This is why tinplate containers have been so successful for packaging food products. Low concentrations of oxygen tend to inhibit corrosion because of the effect upon polarity of the system. At higher concentrations of oxygen,

the tin becomes cathodic and the iron anodic, which promotes corrosion.

Therefore, the effects of oxygen upon the free radical reaction between propellant 11 and ethyl alcohol and upon the electrochemical characteristics of the system tend to oppose each other as far as corrosion is concerned. The effects are summarized in Table 1.

**Table 1. Effect of oxygen concentration**

| | |
|---|---|
| **Low Oxygen Concentration** | |
| 1. Free radical reaction catalyzed | — promotes corrosion |
| 2. Little cathodic depolarization | — retards corrosion |
| 3. Tin anodic and iron cathodic | — retards corrosion |
| **High Oxygen Concentration** | |
| 1. Free radical reaction inhibited | — retards corrosion |
| 2. Cathodic depolarization | — promotes corrosion |
| 3. Tin cathodic and iron anodic | — promotes corrosion |

The fact that considerable corrosion occurs with propellant 11-ethyl alcohol combinations at low oxygen concentrations and very little occurs at high oxygen concentrations shows that the most important factor is the free radical reaction. Apparently, the polarizing activity of hydrogen at the cathode is not sufficient to prevent corrosion at low oxygen concentrations. once the free radical reaction has been initiated. The corrosion is usually of the generalized detinning type which indicates that the tin is anodic. At high oxygen concentrations, the depolarizing effect of oxygen is of little consequence since the free radical reaction has been inhibited and there is no acid formed which will attack the tinplate. Therefore, there is no hydrogen for the oxygen to react with.

Nitromethane has been found to be an effective stabilizer for the free radical reaction and to prevent it, in most cases, regardless of the concentration of oxygen (9). Therefore, it is not necessary to attempt to control the oxygen concentration in an aerosol product in order to minimize the reaction between propellant 11 and alcohol.

The combination of ethyl alcohol and propellant 11 is an excellent example of two components that are noncorrosive themselves but will react to form corrosive products.

## Aqueous Systems

*Oil-in-Water Emulsions and Three-phase Systems*

Many aerosol products, such as window cleaners, starch sprays, furniture polishes, etc., are formulated either as oil-in-water emulsions, where the propellant is emulsified in the aqueous phase or as three-phase systems. Propellant 11 has never been used in these aqueous systems because it reacts with water to form salts and acidic products. The resultant high conductivity of the aqueous phase in contact with the metals provides all the conditions necessary for electrochemical corrosion.

Present evidence (10) indicates that the reaction between propellant 11 and water in tinplate containers is catalyzed by the metals present. The reaction products include propellant 21, fluorocarbon 112, and acidic materials. The reactions that have been postulated to account for these products are (M = metal):

$$2\ CCl_3F + M \longrightarrow CCl_2F\ CCl_2F + M\ Cl_2$$

$$2\ CCl_3F + 2H_2O - 2M \longrightarrow CH\ Cl_2F + M\ Cl_2 + M\ (OH)_2$$

$$CCl_3F + H_2O \longrightarrow \begin{matrix} F \\ \diagdown \\ C=O \\ \diagup \\ Cl \end{matrix} + 2HCl \longrightarrow CO_2 + HF + HCl \quad (H_2O)$$

The propellant 11-water combination causes considerable corrosion in metallic containers; but water alone, without propellant 11, also causes corrosion, although less than the propellant 11-water combination. Nitromethane is a fairly effective corrosion inhibitor for water alone or for the propellant 11-water combination. (10) This suggests that nitromethane acts as a corrosion inhibitor by adsorption on the metal surfaces. Actually, most inhibitors are considered to function as chemically or physically adsorbed films which change the electrochemical nature of the system or act as barriers to corrosion processes (7). Polar compounds are reported to work in this manner by acting as barriers to corrosion by water and it seems very likely that this is how nitromethane functions in this system (7). From another point of view, the decomposition of halogen compounds by combinations of metal and moisture is considered to be free radical in nature (11) and nitromethane may also be functioning as a free radical inhibitor.

## Water-in-Oil Emulsions

When products that require a fine spray, such as room deodorants, are formulated as aqueous products, water-in-oil emulsions are used where the water is dispersed in the propellant. In contrast to its instability in oil-in-water emulsions, propellant 11 is usually sufficiently stable in water-in-oil systems. (12)

There are several reasons for the increased stability of propellant 11 in these systems. The water droplets in a water-in-oil emulsion are surrounded by an interfacial film of the oil soluble surfactant and this film prevents the water from coming into contact with the metal surface. As previously mentioned, the metal is the catalyst for the propellant 11-water reaction and the absence of contact undoubtedly prevents the reaction.

Electrochemical reactions leading to corrosion are minimized in the water-in-oil emulsions also because the continuous phase of the emulsion is organic and is much less conductive than the

aqueous phase of the oil-in-water emulsions. The surfactants used for the water-in-oil emulsions are nonionic and do not impart a charge to the dispersed water droplets. The conditions in a water-in-oil emulsion therefore are not favorable for the flow of electric current and this minimizes the tendency toward corrosion.

In order for the water-in-oil emulsions to remain noncorrosive to metal, it is necessary for the emulsion itself to be stable as far as coalescence of the dispersed water droplets is concerned. If the water droplets coalesce during storage and form a separate, continuous water phase, this would be essentially the same as a three-phase system and the separate water layer would be conducting. However, if the droplets maintain their individuality even though the emulsion creams, the conditions required for a noncorrosive system will remain.

### Aqueous Alcohol Systems

The substitution of 95% ethyl alcohol for anhydrous ethyl alcohol in products such as hair sprays has been suggested in order to obtain a cost savings. Combinations of 95% ethyl alcohol and propellant 11 are more corrosive than combinations with anhydrous alcohol because the propellant 11 can react with the ethyl alcohol and the water. However, nitromethane is a fairly effective inhibitor for both of these reactions and combinations of 95% ethyl alcohol with propellant 11 containing nitromethane have been shown to have appreciable stability in metal containers.

Combinations of 95% ethyl alcohol and propellant 11 with nitromethane as an inhibitor are almost as stable as combinations of anhydrous ethyl alcohol with unstabilized propellant 11 as far as corrosion in metal containers is concerned. The effect exerted by the other components of the aerosol upon the reaction of propellant 11 with alcohol and water will usually determine whether or not a product with 95% ethyl alcohol can be marketed. During the study of the stabilization of anhydrous ethyl alcohol and propellant 11 by nitromethane, it was observed that in certain cases, the active ingredients in the aerosol products also inhibited the free radical reaction. In other instances, the ingredients had a catalytic effect. If the product itself has an inhibiting effect, it may lend itself to be packaged in combination with propellant 11 containing nitromethane and 95% ethyl alcohol.

Combinations of propellant 11 with 20% and 90% ethyl alcohol were found to be too corrosive to be packaged in metal containers, even with nitromethane present. It is interesting that nitromethane is an effective inhibitor for propellant 11-ethyl alcohol systems or propellant 11-water systems but is ineffective for aqueous ethyl alcohol combinations containing appreciable concentrations of water.

Aqueous alcohol solutions alone are probably fairly corrosive, even without propellant 11 present. Aqueous ethyl alcohol foams, containing high proportions of alcohol, have received a considerable amount of attention during the last few years. In general, these products attack, metal containers, even when propellant 12/ propellant 114 combinations are used.

### Propellant 21

Propellant 21 has aroused considerable interest among aerosol fillers as a result of its excellent solvent properties. Stable in acidic and neutral aqueous systems, it decomposes rapidly under alkaline conditions. (13) The salts resulting from the decomposition in alkaline systems undoubtedly would lead to increased corrosion, but this is academic since propellant 21 would not be used in alkaline conditions. There is no evidence that propellant 21 will react with ethyl alcohol *via* a free radical reaction as does propellant 11.

### Propellant 22

Propellant 22 has found only limited application in aerosol products, partially as a result of its high pressure. Like propellant 21, it is stable under acidic or neutral conditions but decomposes in alkaline systems. (14)

### Chlorinated Solvents

As a general rule, chlorinated compounds, such as methylene chloride, methyl chloroform, and trichlorethylene, tend to be unstable in the presence of metals, water, and air. Many of these solvents will decompose with formation of acidic products and this can cause corrosion problems during bulk shipment or in nonaerosol applications that involve the cleaning of metal components. A wide variety of additives have been tested as potential stabilizers to prevent decomposition of the chlorinated solvents. A number of patents have been issued disclosing stabilizers found to be effective in preventing the decomposition of the solvents. (15) Most of the common commercial chlorinated solvents now contain stabilizers.

In aerosols, chlorinated compounds serve as solvents for active ingredients and as vapor pressure depressants. Methylene chloride appears to be quite stable and has been used as a component of aerosols formulated both as oil-in-water and water-in-oil emulsions. Methyl chloroform is less stable and can cause corrosion in aqueous systems.

PAST unsuccessful attempts to package aerosol shampoos based upon anionic detergents such as sodium lauryl sulfate, are well known. (16) Although these products appeared to have satisfactory storage stability at elevated temperatures, they caused perforation of metal containers within a short time at room temperature. As a result, aerosols formulated with aqueous solutions of anionic detergents are usually viewed with suspicion, as far as stability in metal aerosol containers is concerned.

Sodium alkyl sulfates ionize in solution and contain inorganic salts as impurities which also ionize. Solutions of sodium alkyl sulfates therefore are electrolytes with very high corrosive potentialties. Corrosion caused by these surfactants was of the pinholing type, and, as previously mentioned, it was comparatively rapid. The corrosive nature of the sodium lauryl sulfate systems at room temperature has been explained very effectively by Root, who demonstrated that in these systems, the tin is cathodic and the iron anodic. (17) Therefore, corrosion was localized at the relatively minute areas where the iron was exposed and pinholing occurred rather than generalized detinning. This system is an excellent example of the severe corrosion that can occur when a cathode with a large area (the tin) is coupled with an anode with a small area (the iron).

The reason why the sodium lauryl sulfate shampoos appeared to be stable at elevated temperatures may have been that the polarity of the system was reversed, at the higher temperatures, with tin becoming the anode and iron the cathode. As previously mentioned, iron and tin are close in their electrochemical properties, and it is very possible that variations in temperature might cause a reversal of polarity. If, at the higher temperatures, tin was the anode and iron the cathode, the product would be expected to be much more stable. Root examined several other shampoo formulations in which the tin was found to be anodic and the iron cathodic. These formulations were reported to have fairly good storage stability at room temperature. There are many examples known where the storage stability results at elevated temperature fail to correlate with room temperature tests. (23, 24, 25). Results of storage tests at elevated temperatures must therefore be viewed with a certain amount of caution. Again, it is possible, that in many of these cases, the lack of correlation between the room temperature tests and those at the accelerated temperatures may be due to a reversal of polarity at the different temperatures.

Aerosol shaving lathers provide an interesting example of a stable system with surfactant present. These products generally are formulated with triethanolamine salts of fatty acids as the emulsifying agents. Although these surfactants are anionic, the rate of corrosion of the shaving lathers is acceptably low and the products have been a tremendous success. There probably are a number of factors involved in the low corrosion rate of the triethanolamine salts. In these systems, the tin may be anodic and the iron cathodic, which would slow down corrosion considerably. Because triethanolamine salts are salts of a weak acid and a weak base, they tend to hydrolyze in solution rather than ionize. They do not contain inorganic salts as impurities. Therefore solutions of triethanolamine salts would be much weaker electrolytes than solutions of sodium alkyl sulfates and corrosion would be correspondingly less.

Still another factor may be that the tin and iron salts of fatty acids are insoluble in the aqueous phase. LaQue and Copson (4) have pointed out that corrosion is reduced when the corrosion products are insoluble. The salts of the alkyl sulfates would be expected to be soluble. West (18) has suggested that fatty acid soaps should be considered as corrosion inhibitors.

Nonionic surfactants in general appear to cause much less corrosion than the anionic agents. This may be due to the decreased conductance of the aqueous solutions, and possibly the system is anodic with respect to tin.

Cationic agents, such as quaternary ammonium compounds, have been used as germicidal agents in aerosol products. These compounds have been reported to cause corrosion in aqueous solutions. (19)

### *Water In Aerosols*

The use of water as one of the major components in aerosol products has steadily increased during the past few years and water based aerosols now are common. Water has the advantages of being inexpensive, nontoxic, and a good solvent. Unfortunately, since water contains ions, it meets one of the first requirements for corrosion, which is the presence of an electrolyte. The corrosivity of water varies, depending upon the amount and type of dissolved solids and gases. (3) This, in turn, depends upon the source of the water. The corrosive activity of distilled water is low compared with that of sea water with its high concentration of salts. The fact that dissolved gases influence the corrosive properties of water is shown by the attack on steel by water containing appreciable concentrations of carbon dioxide.

If the storage stability of a water based product in a metal container is to be determined, it is important that the test pack is prepared with the same type of water that will be used in production. The storage stability of samples prepared in the laboratory with distilled water may be quite different from that prepared with deionized or tap water. Also, storage stability of a product prepared in one location may be entirely different from that of samples prepared in a different location, as a result of the difference in the composition of the water.

### Acids, Salts, and Alkalies

The hydrogen ion is one of the principal factors in causing corrosion as a result of its reaction at the cathode to form hydrogen. (4) Therefore, one would expect the rate of corrosion to be a function of the acidity of the solution. As far as iron is concerned, the dividing line between rapid and slow corrosion is reported to occur at a pH of 4.5. Free acids can attack metals in a number of ways. They can react with metals directly or they promote corrosion by removing protective films from metals.

The corrosiveness of salt solutions is well known, particularly by those who have parked their cars by the seashore for extended periods. The solutions of most salts are good electrolytes and this provides an environment suitable for galvanic corrosion. The corrosiveness of a solution will vary considerably, depending upon the type of salts present. Acid, salts, such as some of the aluminum salts that have been used in antiperspirant products, can be quite corrosive in aqueous solution.

Generally alkalies are less corrosive than acids, but quite often they produce cathodic films which then cause anodic attack at localized areas. This results in a pitting type of corrosion. (3)

### Corrosivity in Aluminum

Aluminum is a good example of a metal that becomes passive as a result of the formation of a protective oxide film. This oxide film forms readily in the presence of oxygen or oxidizing agents and its thickness can be increased by anodizing. Aluminum is quite resistant to many normally corrosive materials because of this protective oxide film. The film is reported to be stable under most conditions in the pH range of from 4.5 to 8.5. (5)

Aluminum containers are used extensively in Europe and in some countries the majority of aerosol products is packaged in aluminum, indicating that many aerosol products have a satisfactory storage life in this medium. In the United States, aluminum containers have been used to only a minor extent partially because they have been more expensive than tinplate containers. Consequently, there is much less information available in the United States on the compatibility of aerosol products in aluminum containers than there is for tinplate containers. However, aluminum containers offer a number of advantages and storage stability tests with aluminum have therefore been carried out at intervals for a number of years.

These studies have shown that combinations of propellants with alcohols can cause extensive corrosion of aluminum. This is unfortunate because a large proportion of aerosol products are formulated with these combinations. Thus, mixtures of anhydrous ethyl alcohol with propellant 114 have been observed to cause perforation during storage at elevated temperatures. (17) In this case the attack is attributed to the formation of the aluminum alcoholate and hydrogen. The possibility of a rapid reaction in the presence of anhydrous alcohols and propellants with a build-up of pressure from the hydrogen was also noted in tests carried out in the presence of aluminum strips. (22) The addition of about 1.5% to 2.0% water had a strong inhibiting effect upon the corrosion; the water apparently inhibited the formation of the alcoholate, which is favored by anhydrous conditions. (17, 20) Raising the concentration of water beyond the 2% level increased the rate of corrosion.

Combinations of ethyl alcohol and propellant 11 caused very rapid corrosion of aluminum containers and there was no concentration of water found which offered sufficient protection against the attack. Ethyl alcohol and propellant 11 can react to form acidic materials and it was assumed that these attacked the protective oxide film on the aluminum and exposed the metal itself to corrosive attack.

Distilled water in aluminum containers caused heavy corrosion and build-up of pressure when the containers were pressurized either with nitrogen or with nitrous oxide. The storage stability appeared to be satisfactory, however, when the containers were pressurized with carbon dioxide. (21) Root studied the storage stability of three shaving lather formulations in aluminum containers and reported that two of the formulations had satisfactory shelf life in the containers while the third caused considerable corrosion. (17)

There seems to be little doubt that the use of aluminum containers for aerosol products will continue to increase as more information about the behavior of aerosol products in these containers becomes available. However, the studies that have been carried out indicate that some combinations, such as mixtures of propellant 11 and alcohols, will have to be avoided.■

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