REACTION OF PROPELLANT 11 WITH WATER

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Reaction of water with propellant 11 at 130°F. shown to be catalyzed by metal. Products of reaction, propellant 21, fluorocarbon 112 and acid, indicate three different reactions occur.

Reaction of Propellant 11 with Water

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THE stability of trichloromonofluoromethane (propellant 11) in the presence of water and metals has been of considerable interest to both the aerosol and refrigeration industries. In the aerosol industry, the instability of propellant 11 in the presence of water and metals has limited its use in aqueous based aerosol products. In the refrigeration industry, the stability of propellant 11 is of concern because of the possibility of corrosion to the metal components in the refrigerating system and the effect of acidic reaction products on the insulation.

As a result of these considerations, the stability of combinations of Propellant 11, metals and water has been the subject of several investigations. Parmelee and Downing (1) aged combinations of steel, aluminum, water and propellant 11 in both the absence and presence of air. The corrosion that was observed on steel was considered to be due principally to rusting, but it was reported that hydrolysis of the propellant ultimately occurred. The presence of air increased the corrosion of the steel.

Church and Mayer (2) aged combinations of propellant 11 and various metals in cylindrical pressure bombs at temperatures up to 150°F. Moisture contents in the mixtures were varied from 0.001% to over 1.0% and the air content was varied up to 4.0 volume per cent. These conditions were chosen in an attempt to simulate the conditions which might be encountered in industrial refrigeration systems.

Church and Mayer postulated three possible decomposition reactions of propellant 11 in the presence of water and metal as follows: (M=metal).

$$2CCl_3F + M \rightarrow MCl_2 + CCl_2FCCl_2F \quad (1)$$

$$CCl_3F + H_2O \rightarrow CCl_2FOH + HCl \;\downarrow\; \quad \mathrm{F{-}C(=O){-}Cl} + HCl \quad (2)$$

$$2CCl_3F + 2H_2O + 2M \rightarrow 2CHCl_2F + MCl_2 + M(OH)_2 \quad (3)$$

Reaction 1, which produces a metallic chloride and tetrachlorodifluoroethane (fluorocarbon 112), is similar to a Wurtz reaction. In Reaction 2, propellant 11 is decomposed by water to form a hydrogen halide and an analog of phosgene. In Reaction 3, propellant 11 is reduced to dichloromonofluoromethane (propellant 21) by the action of metal and water. Reaction 3 is similar to the reaction that is used for the preparation of chloroform in which carbon tetrachloride is reduced with iron and water.

Church and Mayer determined the rates of decomposition of propellant 11 under various conditions by analyzing the reaction products for chloride ion. They were unable to find any fluoride ion with a method they reported could detect concentrations of fluoride ion as low as 20 ppm. They also were unable to detect any hydrogen halide in the vapor phase. The concentration of organic decomposition products in the reaction mixtures was too low for identification, but on the basis of their rate data, Church and Mayer favored reaction 1.

Among aerosol loaders, combinations of propellant 11 and anhydrous ethyl alcohol, in products such as hair sprays, were known to have caused container corrosion at various times. Initially, this was attributed to the hydrolysis of the propellant 11 by the low concentrations of water present in the ethyl alcohol. Later, it was shown that, under certain conditions, propellant 11 reacted with anhydrous ethyl alcohol to produce acetaldehyde, hydrogen chloride, and dichloromonofluoromethane as the major reaction products (3).

$$CCl_3F + CH_3CH_2OH \rightarrow CH_3CHO + HCl + CHCl_2F \quad (4)$$

The acid formed in this reaction was responsible for the corrosion observed in the containers. Reaction 4 was shown to be a free radical reaction and the mechanism of the reaction was elucidated. In a subsequent investigation of potential stabilizers for the reaction, nitromethane was found to be an excellent inhibitor for the reaction (3).

The availability of propellant 11 containing nitromethane as an inhibitor ("Freon-11" S) eliminated corrosion problems resulting from the reaction of propellant 11 with ethyl alcohol. Interest then shifted to the possibility of using propellant 11 in combination with 95% ethyl alcohol instead of anhydrous ethyl alcohol because of the cost saving that could be achieved by the substitution.

In view of the previous work on the reaction of propellant 11 with anhydrous ethyl alcohol, and the corrosion studies of Parmelee and Downing which showed that propellant 11 reacted with water alone under certain conditions, it would be expected that, in combinations of propellant 11 and 95% ethyl alcohol, propellant 11 would react with both the ethyl alcohol and the water. In any investigation involving the reaction of propellant 11 with 95% ethyl alcohol, it was important to be able to determine the relative rates of reaction of propellant 11 with ethyl alcohol and with water. It was conceivable that the overall hydrolysis reactions of propellant 11 with water could be represented by the following equation:

$$CCl_3F + 2H_2O \rightarrow CO_2 + 3HCl + HF \qquad (5)$$

It was known that, for all practical purposes, Reaction 4 represented the reaction of propellant 11 with ethyl alcohol. If the assumption that Reaction 5 represented the overall reaction of propellant 11 with water was valid, then, when mixtures of propellant 11 and 95% ethyl alcohol were reacted, the relative rates of Reaction 4 and Reaction 5 could be determined by analyzing the reaction mixture for propellant 21 and for acid. The concentration of propellant 21 in the reaction mixture would indicate the extent to which Reaction 4 took place, while the excess of acid (or halide ion) above that formed in Reaction 4 would indicate to what extent Reaction 5 occured.

Before proceeding to investigate the problem on this basis, however, a preliminary check was made to determine if propellant 21 could be formed in the reaction of propellant 11 with water. Mixtures of propellant 11 and water were stored in tinplate containers with blank caps for one week at 130°F. The propellant 11 layer was analyzed by gas chromatography and found to contain an appreciable concentration of propellant 21. The fact that propellant 21 was formed in the reaction between water and propellant 11 in metal containers eliminated the possibility of distinguishing between Reaction 4 and Reaction 5 by analyzing the reaction mixture for propellant 21 and acid. An investigation of the reaction between propellant 11 and water alone in the presence of metals therefore was initiated, not only to clarify the mechanism, but also in the hope that this might lead to compounds or methods which might retard the reaction.

Summary and Conclusions

Combinations of propellant 11, water, and metals were stored at 130°F for various periods of time. The reaction mixtures were analyzed and found to contain propellant 21, fluorocarbon 112, acid and, in some cases, quantities of insoluble solids assumed to be metallic salts or hydroxides. The formation of propellant 21 and fluorocarbon 112 indicated that the following two reactions probably occurred:

$$2CCl_3F + M \rightarrow MCl_2 + CCl_2FCCl_2F \qquad (1)$$

$$2CCl_3F + 2H_2O + 2M \rightarrow 2CHCl_2F + MCl_2 + M(OH)_2 \qquad (3)$$

The presence of acid in some of the mixtures indicated that either Reaction 2, Reaction 5 or some other acid-forming reaction took place.

The decomposition of propellant 11 by water is catalyzed by the metal. In the absence of metal, propellant 11 does not appear to react with water.

Water alone causes considerable corrosion in aerosol tinplate containers. Combinations of water and propellant 11 cause more corrosion than water alone as a result of the reaction of water with propellant 11. Nitromethane not only retards the corrosion from water alone but also retards the corrosion caused by mixtures of water and propellant 11. One of the functions of nitromethane as a corrosion inhibitor for water or water propellant 11 systems appears to be a deactivator for the metal. This prevents the metal from acting as a catalyst for the reaction between water and propellant 11.

Nitromethane was evaluated alone as a corrosion inhibitor and also in combination with a variety of other known corrosion inhibitors. The combination of nitromethane/sodium benzoate appeared to be the most effective. This combination was tested in three aqueous based aerosol products, a window cleaner, a starch formulation and a snow formulation. The nitromethane/sodium benzoate combination was effective in retarding the corrosion caused by the aerosol window cleaner during storage at 130°F. but caused adverse color changes in the other two aerosol products. This indicates that the inhibitor combination must be tested thoroughly in each formulation before large scale use is considered.

Experimental

1. Reaction of propellant 11 with Water

A. In the absence of metal catalysts

In the absence of metal catalysts, propellant 11 does not appear to react with water. Thus, in water saturated with propellant 11 at atmospheric pressure, the hydrolysis rate was too low to measure after one year at room temperature. (4). In the presence of steel, the hydro-

Table I. Reaction of Propellant 11 with Water in Glass-with Metal

One Week at 130°F.

Mixture No.	Water/Propellant 11 Ratio (Wt. %)	Metal* Present	Air Present	Concentration of Reaction Products In Organic Phase (Wt. %) Propellant 21	Fluorocarbon 112	pH of Aqueous Phase	Appearance of Reaction Mixture
1	70/30	No	Yes	None	None	4.3	Colorless
2	70/30	Yes	Yes	1.43%	0.09%	4.9	The aqueous phase was grey and there was a considerable amount of brown solid present
3	70/30	Yes	No	1.43%	0.04%	4.6	Both layers were almost colorless but there was some grey solid present

*Metal strips from a sideseam tinplate aerosol container.

lysis rate became appreciable and could be determined by analytical methods. In unpublished work carried out at the "Freon" Products Laboratory, homogeneous solutions of propellant 11 in dioxane containing up to 2.5% water were aged in glass for 18 months at 167° F. Judging by the fact that the chloride ion concentration did not increase during the aging period, there was no hydrolysis of the propellant 11 during that time.

B. In the presence of metal

Experiments to determine the effect of both metal and air upon the reaction between propellant 11 and water were carried out by sealing water/propellant 11 (70/30) mixtures in glass flasks and aging the flasks for one week at 130°F. One of the samples was prepared without any added metal; the other two samples were prepared with added strips of metal from a sideseam, tinplate aerosol container. The strips were sufficiently long so that part of the strips extended into the vapor phase. One of the flasks containing the metal strips was cooled in ice water and sealed in order to trap air in the flask. This would indicate the stability of propellant in the presence of water, metals and air. The other sample was cooled and evacuated to remove air before sealing. This would indicate the stability of propellant in the presence of water and metals without any air present.

After the storage tests were completed, the organic phases were analyzed by gas chromatography. The results of the analyses, along with visual observations of the reaction mixtures, are given in Table I. The data show that propellant 21 and fluorocarbon 112 were formed during the metal catalyzed reaction of propellant 11 with water. No other unknown reaction products were found during the gas chromatographic analysis of the mixtures. The catalytic effect of the metal strips (which included the soldered sideseam) from the tinplate container upon the reaction was very pronounced. In the absence of the metal, no reaction between water and propellant 11 occurred.

The effect of air was not very conclusive, judging from the present data, but there was some indication that the rate of reaction was increased by the air present. Reaction Mixture No. 2 had more solid material present and the mixture was more discolored than Reaction Mixture No. 3 Also, the concentration of fluorocarbon 112 in Reaction Mixture No. 2 was higher than that in Reaction Mixture No. 3.

The formation of fluorocarbon 112 and propellant 21 can be accounted for by Reaction 1 and Reaction 3, respectively. The molar concentration of propellant 21 in Reaction Mixture No. 2 was about 31 times greater than that of fluorocarbon 112. It might be assumed from this that Reaction 3 occurred at a rate about 31 times greater than Reaction 1. However, this conclusion would also assume that neither propellant 21 nor fluorocarbon 112 themselves reacted further once they are formed. It is known that propellant 21 will react in the presence of steel and water (4) and it is likely that fluorocarbon 112 would also react. Therefore, the relative concentrations of propellant 21 and fluorocarbon 112 in the mixture do not necessarily indicate the relative rates of Reaction 1 and Reaction 3.

The similarity of Reaction 3 to the preparation of chloroform by the reduction of carbon tetrachloride with iron and water has been mentioned. This was confirmed by adding a small quantity of carbon tetrachloride to propellant 11 and aging the mixture in the presence of water in metal containers. Chloroform was identified as one of the reaction products.

The pH of the three aqueous phases was about the same. It is possible that no acid was formed during the reaction. In this respect, the results would be similar to those reported by Church and Mayer. A second possibility is that acid was formed and reacted completely with the metal present to form metallic chlorides.

In another experiment, mixtures of water and propellant 11 were aged in drawn tinplate aerosol containers with blank caps for one week at 130°F. Under these conditions, there was no solder present in the reaction mixture. According to titration of the aqueous phase with base, there were twice as many mols of acid as there were mols of propellant 21 in the mixture. The presence of the acid indicated that either Reaction 2, Reaction 5, or decomposition reactions involving propellant 21 or fluorocarbon 112 occurred. Church and Mayer were unable to find any fluoride ion in the reaction mixture, which would eliminate Reaction 5. Neither were they able to find any hydrogen halide in the vapor phase which would tend to eliminate Reaction 2. However, it is a little dangerous to extrapolate their results, which were obtained with relatively low concentrations of water, to the present experiments.

II. *Comparative Corrosive Effects of Water Alone and Water/Propellant 11 Mixtures in Aerosol Tinplate Containers.* The preceding work has shown that a number of reactions occurred during the storage of mixtures of propellant 11 and water in metal containers. Considerable corrosion occurred in the container during the storage tests. Before attempting to find compounds which might retard the corrosion, however, it was necessary to determine how much of the corrosion was caused by water alone and how much was caused by the products of the reaction between water and propellant 11.

In order to obtain this information, samples with water alone and samples with mixtures of water and propellant 11 were aged in tinplate containers with blank caps for periods up to eight weeks at 130°F. The corrosion observed in the containers was judged by the rating system shown in Table II.

The data obtained from three different experiments are listed in Table III. These data show that water alone caused considerable corrosion in the containers. Mixtures of water and propellant 11 caused more corrosion than water alone, as would be expected. Therefore, any effective corrosion inhibitor for water/propellant 11 systems in metal containers would have to include an inhibitor for re-

Table II. Corrosion Rating System

Corrosion Rating	Description
0	No corrosion
0-1	No corrosion, but slight darkening either liquid or vapor phase or slight ring at interface
1	Generalized corrosion either liquid phase or vapor phase—spotty detinning
1-2	Generalized corrosion both liquid phase and vapor phase — spotty detinning
2	Complete detinning either liquid phase or vapor phase
2-3	Complete detinning either liquid phase or vapor phase with spotty detinning in other phase
3	Complete detinning both liquid phase and vapor phase
3-4	Complete detinning either liquid phase or vapor phase with rusting and spotty detinning in other phase
4	Complete detinning both liquid phase and vapor phase with spotty rusting
4-5	Complete rusting throughout container
5	Container leaked

tarding the corrosion of metal by water alone. If this could be achieved, the reaction between water and propellant 11 might also be retarded since the data had shown very clearly that the reaction between water and propellant 11 was catalyzed by metal.

III. *Corrosion Inhibitors for Water and Water/Propellant Mixtures.* The investigation of the reaction of water with propellant 11 had shown that the reaction was catalyzed by metal. The data had also shown that water alone caused corrosion in metal containers. The search for suitable corrosion inhibitors, therefore, was concentrated on compounds which would inhibit metals against corrosion by aqueous systems or compounds which would inhibit the corrosion of metals by halogenated compounds.

The use of nitromethane for stabilizing halogenated compounds against decomposition by metals has been revealed in patents (5). It has also been disclosed as a stabilizer for retarding the corrosion of metals by aqueous antifreeze solutions (6). The use of nitromethane in combination with propylene oxide as a corrosion inhibitor for aqueous based aerosols has also been disclosed recently (7).

Nitromethane was tested alone and in combination with other known corrosion inhibitors. Tests were carried out at 130°F. in tinplate containers with water alone and with mixtures of water and propellant 11. The most effective inhibitor combination for reducing the corrosion either from water alone or from mixtures of water and propellant 11 was a nitromethane/sodium benzoate combination. The corrosion occurring in metal containers with various combinations of water, propellant 11, nitromethane, and sodium benzoate is shown by the data in Table IV, using the corrosion rating system given in Table II.

The data in Table IV show that nitromethane was very effective alone as an inhibitor for retarding the corrosion from water or from water/propellant 11 mixtures. Sodium benzoate was ineffective in retarding the corrosion from water alone but was somewhat helpful in retarding the corrosion from water/propellant 11 mixtures.

The preceding experiments

Table III. Corrosive Effects of Water and Water/Propellant 11 Mixtures in Tinplate Containers

	Composition of Water/ Propellant 11 Mixture (Wt. %)		Corrosion after Storage at 130°F		
Run No.	Water	Propellant 11	2 Weeks	4 Weeks	8 Weeks
1	100	0	2	3-4	3-4
1	70	30	3-4	4	5
2	100	0	2	3-4	3-4
2	70	30	2	3-4	4-5
3	100	0	3-4	3-4	3-4
3	70	30	3-4	4-5	5

Table IV. Effect of Nitromethane /Sodium Benzoate Combinations in Retarding Corrosion from Water/Propellant 11

Composition of Mixture (Parts by Wt.)				Corrosion in Metal Containers after Storage		
Water	Sodium Benzoate	Nitro-methane	Propellant 11	2 Weeks	4 Weeks	8 Weeks
105.0	—	—	—	2	3-4	3-4
105.0	—	0.3	—	1	1-2	1-2
104.7	0.3	—	—	3-4	3-4	3-4
104.7	0.3	0.3	—	0	0-1	0-1
105.0	—	—	45.0	3-4	4	5
105.0	—	0.3	44.7	1	1	1
104.7	0.3	—	45.0	2	4	4
104.7	0.3	0.3	44.7	0-1	1	1

had shown that the reaction between water and propellant 11 was catalyzed by metals. Propellant 21 and acid were among the reaction products. Since nitromethane was found to be a corrosion inhibitor for the water/propellant 11 system, it should also retard the formation of propellant 21 and acid when the mixtures were stored in metal containers.

This was demonstrated by aging mixtures of water with propellant 11 or propellant 11 containing 0.3% nitromethane ("Freon-11" S) in tinplate containers for 16 hours and 24 hours at 194°F. The acidity of the aqueous phases was determined by titration and the propellant 11 layer was analyzed on the gas chromatograph to determine the concentration of propellant 21. The results are given in Table V. The data show that nitromethane reduces the formation of both acid and propellant 21.

Nitromethane may function as an inhibitor in several ways. For example, it may be adsorbed on the tinplate and thus deactivate the metal. Balezin and Chistyakov (8) evaluated various inhibitors for retarding the corrosion of carbon steel by carbon tetrachloride and carbon tetrachloride and water combinations. They reported that benzoic acid, one of the inhibitors studied, functions by adsorption on the steel surface and that this apparently is followed by a reaction of the benzoic acid with the steel. The fact that nitromethane retards corrosion by water alone suggests that it is adsorbed on the metal.

Alkyl halides are also apparently able to react with metals to produce organic radicals (9). These can yield coupled symmetrical products. This could explain the formation of fluorocarbon 112. In this case, nitromethane could act either by deactivating the metal or by reacting with the organic free radicals that were produced. In some cases, metal reductions may give rise to organic radicals (9). Again, nitromethane could act by adsorption on the metallic surface or by reacting with the organic radical itself.

The mechanism by which sodium benzoate functions as a corrosion inhibitor apparently is not known (10).

IV. Evaluation of the Nitromethane/Sodium Benzoate Inhibitor Combination with Aqueous Based Aerosol Products. The effectiveness of nitromethane alone or nitromethane in combination with sodium benzoate in retarding the corrosion from water alone or from water/propellant 11 mixtures had been demonstrated. In order to determine if the combination was also effective for actual aqueous based products, the nitromethane/sodium benzoate combination was tested in three aqueous based aerosol products, a window cleaner, a starch formulation, and an aqueous based snow formulation. The snow was formulated with propellant 12 only and the stabilizers were tested for retarding the corrosion due to water alone.

The concentrates used for the three aqueous based products had the following compositions:

Aerosol Starch Concentrate

	Wt. %
Amaizo #513 Starch	4.00
10% Tributyl tin oxide in "Triton" X-45	0.02
Glycerine	0.30
Water	95.68

Aerosol Window Cleaner Concentrate

	Wt. %
"Triton" X-100	0.1
"Emcol" 5138A	0.1
Odorless Mineral Spirits	9.0
Isopropyl Alcohol	18.8
Water	72.0

Aerosol Snow Concentrate

	Wt. %
Stearic acid	4.0
"Emcol" 14	1.5
Myristic acid	4.0
"Lucite" 46 Acrylic Resin	4.0
Methylene chloride	39.0
Water	47.5

The compositions of the aerosols and the results of the storage stability tests with the aqueous based formulations are given in Tables VI, VII, and VIII. The results indicate that the nitromethane/sodium benzoate combina-

Table V. Stabilization of Water/Propellant 11 Reaction at 194°F with Nitromethane in Metal Containers

	Composition of Mixture	
Aged 16 Hours at 194°F	Water/Propellant 11 (70/30)	Water/"Freon-11" S (70/30)
Normality of aqueous phase	0.11	0.0006
% Propellant 21 in Propellant 11	0.29%	None detected
Aged 24 Hours at 194°F		
Normality of aqueous phase	0.54	0.001
% Propellant 21 in Propellant 11	0.31%	None detected

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tion was useful in only one out of the three formulations. Definite stabilization against corrosion was observed with both the window cleaner formulation and the snow formulation. However, in the latter case, the nitromethane/sodium benzoate combination caused the snow to become orange during storage. With the starch formulation, the nitromethane/sodium benzoate combination did not stabilize the system against corrosion and the inhibitor combination had an adverse effect upon the color of the formulation.

These results indicate that the combination of nitromethane/sodium benzoate can be useful but that it must be tested thoroughly. In some cases, the stabilizers apparently react with some of the components of the aerosol. In other cases, the corrosion caused by the aqueous based formulation apparently is not due to water but to some component in the formulation that is not inhibited by the nitromethane/sodium benzoate combination.

Summary

The reaction of water with propellant 11 at 130°F was shown to be catalyzed by metal. The products of the reaction were propellant 21, fluorocarbon 112 and acid. These reaction products indicate that at least three different reactions occur.

Nitromethane alone or in combination with sodium benzoate stabilizes aerosol tinplate containers against corrosion either by water alone or by water/propellent 11 mixtures. The data indicate that the stabilizing effect of nitromethane is due to deactivation of the metal by nitromethane or deactivation of the organic radicals that might be produced.

The combination of nitromethane and sodium benzoate was tested in three aqueous based aerosol formulations, a window cleaner, a starch, and a snow. The inhibitor combination was useful only with the window cleaner. In the other two formulations, the inhibitors had an adverse effect.

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Table VI. Storage Stability of Aerosol Based Aerosol Snow Formulations

Concentrate	Composition (Parts by Wt.) Nitro-methane	Sodium Benzoate	Propellant 12	Corrosion after Storage at 130°F 2 Weeks	4 Weeks	8 Weeks
74.0	0.5	0.5	25.0	0-1	0-1	1
75.0	—	—	25.0	2	2	2

The nitromethane/sodium benzoate combination caused the color of the snow to become orange after storage for 8 weeks at 130° F.

Table VII. Storage Stability of Aerosol Window Cleaner Formulations

Concentrate	Composition (Parts by Weight) Nitro-methane	Sodium Benzoate	Propellant 12	Propellant 12/ Propellant 11 (50/50)	Corrosion after Storage at 130°F 2 Weeks	4 Weeks	8 Weeks
95.0	—	—	5.0	—	3-4	3-4	3-4
94.5	0.5	—	5.0	—	0-1	2	2
94.5	—	0.5	5.0	—	0-1	0-1	1
94.0	0.5	0.5	5.0	—	0-1	0-1	0-1
90.0	—	—	—	10.0	3-4	3-4	3-4
89.5	0.5	—	—	10.0	2	3-4	3-4
89.5	—	0.5	—	10.0	2	1-2	5
89.0	0.5	0.5	—	10.0	0-1	0-1	1

All containers had a corrosion rating of 0-1 after storage for one year at room temperature

Table VIII. Storage Stability of Aerosol Starch Formulations

Concentrate	Composition (Parts by Weight) Nitro-methane	Sodium Benzoate	Propellant 12/ Propellant 114 (40/60)	Propellant 12/ Propellant 11 (50/50)	Corrosion after Storage 4 Weeks at 130°F	1 Year at Room Temperature
95.0	—	—	5.0	—	3-4	3
94.0	0.5	0.5	5.0	—	2	3-4
95.0	—	—	—	5.0	3-4	3
94.0	0.5	0.5	—	5.0	3-4	3-4

Concentrates with the nitromethane/sodium benzoate combination turned black during storage

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