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The Refrigeration-11 Plant— An Evaluation of Residue Formation and Chemical Cleaning Methods

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→ Residue formation in naval Refrigeration-11 plants is a problem. The formation of these residues is attributed to the chemical reaction of the refrigerant with the compressor oil. This reaction produces hydrochloric acid, which is free to react with the oil and the metal components of the system. Spectroscopic analysis confirms the presence of the metals in the residue, and free chloride ions were found in used compressor oil in amounts that increased with time of operation of the plant. A number of potential solubilizing agents for the residues was studied. Of these, →

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20. (Continued)

ethylenediaminetetraacetic acid (EDTA) and Oakite 32 (a proprietary cleaning formulation) were found to solubilize 90 to 95% of the residue with minimum effects on metal components of the system. ←

CONTENTS

THE REFRIGERATION-11 PLANT—AN EVALUATION OF RESIDUE FORMATION AND CHEMICAL CLEANING METHODS

Refrigeration-11 (R-11) plants perform their cooling function, like all refrigeration systems, by allowing a compressed liquid refrigerant ($CFCl_3$) to vaporize in a specific area. After this the vapor is condensed back to a liquid in the compressor so that it can be vaporized again, to perform its continuous cooling function. During this cycle a small amount of compressor lubricating oil is normally carried along with the refrigerant.

Prolonged operation of the R-11 plants results in repeated formation of large quantities of solids within the refrigeration system. This has become a matter of concern to the Navy. The unwanted residue is associated with the stability of the refrigerant and has caused the R-11 plants to become inoperative. The residue then must be purged from the plant.

Formation of this residue may be due to chemical reactions of the refrigerant with oil, air, and moisture that find their way into the system, and the incompatibility of the refrigerant and its decomposition products (particularly halogen acids) with the metal components of the system.

Borchardt [1] has shown that if the temperature during the compression cycle is high enough, a chemical reaction between the fluorocarbon refrigerant and the compressor oil forms products that resemble degraded oil—sludge, varnish, and coke. Parmelee [2] showed that, of a variety of fluorocarbon refrigerants studied, R-11 was the least stable in the presence of various metals used in the refrigeration system and in the absence of compressor oils.

Eiseman [3] observed that, when the aluminum oxide coating on an aluminum impeller was removed by friction in a malfunctioning system, it was possible for the refrigerant to react with the impeller to produce aluminum fluoride, aluminum chloride, and carbon. However, Borchardt [1] concluded that this reaction was only a signal to the chemical reaction of the refrigerant and compressor oil, which resulted in the formation of hydrochloric acid (HCl).

Spauschus and Doderer [4] demonstrated that R-22 ($CHClF_2$), hydrochloric acid, and other products are produced when R-12 (CCl_2F_2) reacts with oil by the following general equation:

$$\begin{aligned} &\frac{\text{R-12}}{CCl_2F_2} + \frac{\text{Oil}}{RCH_2CH_2R'} \rightarrow \frac{\text{R-22}}{CHClF_2} + \frac{\text{Chlorinated Oil}}{RCH_2CHClR'} \\ &\rightarrow \frac{\text{Acid}}{HCl} + \frac{\text{Unsaturated Oil}}{RCH = CHR'} \;\rceil \\ &\text{Varnish, Sludge, and Coke} \longleftarrow \text{Polymerization} \end{aligned} \tag{1}$$

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Thus, degradation of R-12, as shown, results in the formation of highly corrosive hydrochloric acid, which in turn could attack metallic components of the refrigeration plant. According to Borchardt [1], R-11 decomposes to form R-12 by

$$\frac{\text{R-11}}{2CCl_3F} \rightarrow \frac{\text{R-12}}{CCl_2F_2} + CCl_4. \qquad (2)$$

The R-12 produced could continue to react according to Eq. (1) to produce HCl and other products.

On the basis of the above information, we undertook to assess R-11 degradation in the plants and to evaluate various cleaning solutions and methods for removing the residue. To this end, samples of refrigeration oil, new and used R-11, and the deposits or residue from the plant were evaluated.

EXAMINATION OF RESIDUE FROM R-11 PLANT

An impeller (Fig. 1) from an R-11 plant showed alternate layers of red and gray deposits, indicating possible formation of FeO, Fe_2O_3, and Fe_3O_4. A heterogenous rusty brown residue was obtained by physically scraping the deposits from various parts of the R-11 plant (Fig. 2).

Analysis by emission spectroscopy (Table 1) showed that the scrapings from the void space walls in the plant were mainly iron, whereas scrapings from the general plant area and the impeller contained, in addition to iron, significant quantities of aluminum and copper. Further, an elemental analysis of the scrapings from the general plant area for carbon, nitrogen, hydrogen, and residue contents indicated a percentage composition of 3.5, 0.3, 1.1, and 83.3, respectively.

EXAMINATION OF R-11

Samples of gas and liquid R-11 were taken from a plant after 354 h of operation. The gas phase portion of the sample was analyzed by gas chromatography-mass spectrometry (GC-MS), and the liquid phase of each refrigerant was tested for chloride ion by microcoulometry [5].

Sampling points in the plant are listed in Table 2, and the results of the analyses are given in Table 3. As shown in Table 3, no chloride ions were detected in the liquid phase. Analysis of the gas phase indicated that the unused refrigerant (sample number 5) in general contained lower levels of contamination than the used samples.

Because of the low concentration and response of the unknown components of the samples mass spectrometry could not identify these contaminants. However, a "fingerprint" and total response of the unidentified contaminants were obtained by gas chromatography. The total responses are listed under the column labeled "unknown" and are expressed as methane equivalent in milligrams per cubic meter.

Fig. 1 — Impeller with buildup of solid residue

Up to 10 unidentified contaminants were found in the used refrigerants, and 6 of these were found in the unused sample 5. If the air, as indicated in Table 3, was not introduced at the time of sampling, then the presence of air in an operating plant would hasten the degradation of the refrigerant [2]. Water at low concentration was detected by both gas chromatography and mass spectrometry, but its quantity was not determined.

R-12 (CCl_2F_2), R-21 ($CHCl_2F$), and carbon dioxide (CO_2) were detected in the samples. The presence of R-12 can be accounted for by Eq. (2). Depending on the chemical mechanism, R-21 could be produced by

$$H + \frac{\text{R-11}}{CCl_3F} \rightarrow \frac{\text{R-21}}{CHCl_2F} + Cl \qquad (3)$$

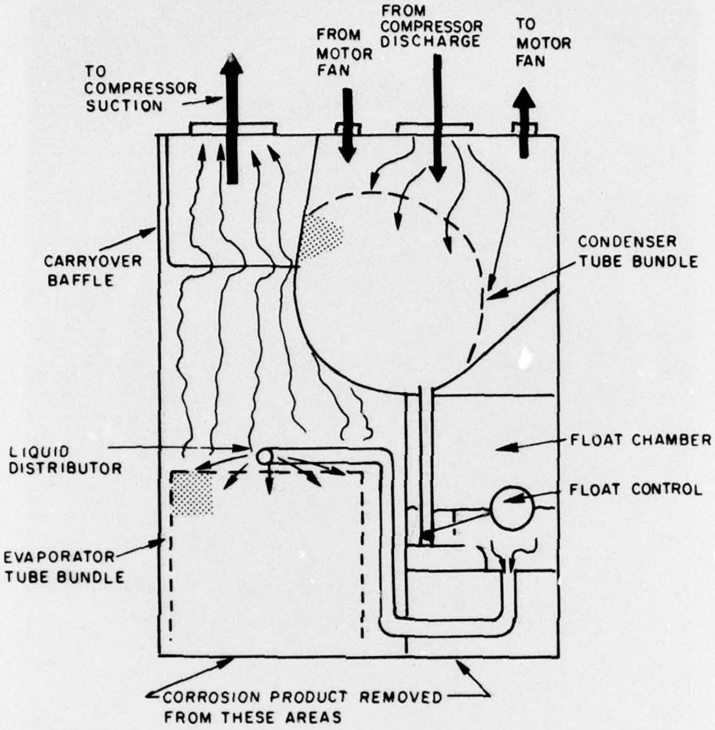

Fig. 2 — Cross section of triduty shell

or by a reaction similar to that shown in Eq. (1) to obtain

$$\frac{\text{R-11}}{CCl_3F} + \frac{\text{Oil}}{RCH_2CH_2R'} \rightarrow \frac{\text{R-21}}{CHCl_2F} + \frac{\text{Chlorinated Oil}}{RH_2CHClR'} . \qquad (4)$$

The presence of the chlorinated oil could then result in production of HCl, as shown in Eq. (1).

The R-11 might degrade in the presence of water as follows:

$$CCl_3F + H_20 \rightarrow CCl_2F0H + HCl \qquad (5)$$

$$CCl_2F0H + H_2O \rightarrow 2HCl + HF + CO_2. \qquad (6)$$

This would account for the high CO_2 concentrations in the samples (Table 3), assuming that ambient CO_2 did not leak into the samples during sampling.

Table 1 — Elemental Analysis on Typical Residue Found in Refrigerant-11 Plant (Relative Percentages)

Component	Impeller Deposit	General Plant Scrapings	Scrapings from Void Space Walls	Scrapings from Void Space Bubble
Iron	Major	Major	Major	Major
Aluminum	1-3	1-3	0.8-1.2	0.001-0.005
Nickel	0.005-0.01	0.005-0.01	0.005-0.01	0.005-0.01
Copper	1-3	3-5	0.3-0.5	0.01-0.05
Titanium	0.001-0.005	0.01-0.05	0.005-0.01	0.001-0.005
Silver	0.0001-0.0005	0.001-0.0005	0.0001-0.0005	0.001-0.005
Vanadium	0.0001-0.0005	0.0001-0.005	0.001-0.005	0.001-0.005
Molybdenum	0.1-0.3	0.1-0.3	0.1-0.3	0.08-0.12
Silicon	0.3-0.5	0.8-1.2	0.3-0.5	0.3-0.5
Manganese	0.3-0.5	0.3-0.5	0.3-0.5	0.3-0.5
Magnesium	0.01-0.05	0.01-0.05	0.0001-0.0005	0.01-0.05
Chromium	0.01-0.05	0.01-0.05	0.05-0.10	0.0001-0.0005
Calcium	0.001-0.005	0.01-0.05	0.01-0.05	0.01-0.05
Zinc	0.001-0.005	0.01-0.05	—	—
Sodium, Strontium	0.01-0.05	0.01-0.05	0.01-0.05	0.001-0.005
Cobalt, Barium	0.001-0.005	0.001-0.005	0.001-0.005	0.001-0.005

Table 2 — Refrigerant-11 Sample Descriptions from Plant A

Sample No.	Sample Date	Sample Description
1	May 9, 1974	Sampling point 5; Evaporator gas
2	May 9, 1974	Sampling point 3; Condenser gas
3	May 7, 1974	Sampling point RAC-47 Purge tank, liquid
4	May 9, 1974	Sampling point 2; Purge tank
5	May 9, 1974	Sampling point 3; Condenser gas
6	Unknown	New Freon 11, source unknown, liquid

Table 3 — Analysis of R-11 Samples from Plant A

Sample No.	Air (%)	Chloride Ion (ppm)	CH_4 (ppm)	R-12 (%)	R-21* (%)	CO_2 (%)	Unknown† (mg/m^3)
1	8 ± 0.8	N.D.‡	1 ± 0.5	0.37 ± 0.03	0.07 ± 0.007	2.2 ± 0.2	10 ± 1
2	3 ± 0.3	N.D.	20 ± 2	2.1 ± 0.02	0.22 ± 0.02	1.0 ± 0.1	105 ± 11
3	3 ± 0.3	N.D.	18 ± 2	2.1 ± 0.02	0.12 ± 0.01	1.0 ± 0.1	15 ± 2
4	10 ± 1.0	N.D.	85 ± 9	0.92 ± 0.09	0.33 ± 0.03	1.0 ± 0.1	36 ± 4
5	3 ± 0.3	N.D.	1 ± 0.5	0.14 ± 0.01	0.02 ± 0.002	0.4 ± 0.04	N.M.‖

*Calculated as R-12 equivalents.
†Reported as methane equivalents; probably oxygenated compounds.
‡N.D.—Not detected (limit of sensitivity, 0.1 ppm by volume).
‖Not measured.

As seen in Table 3, the contaminants R-12, R-21, and CO_2 are produced as a result of the degradation of R-11. Sampling and detection of these contaminants in an operating R-11 plant, for example by gas chromatography [6], could be used in assessing the degradation of the refrigerant before complete plant failure. The buildup of these contaminants would be a sensitive indication of the decomposition of the R-11 as well as of the production of hydrochloric acid.

EVALUATION OF REFRIGERATION OIL

As discussed previously, one of the products formed in the reaction of the refrigerant with the compressor oil is hydrochloric acid (HCl) [4]. New and used compressor oil from the R-11 plant were compared for chloride ion concentrations by extracting the samples with water and then analyzing the water phase with a microcoulometer detector [5]. With this method, organically bound chloride does not interfere with the inorganic chloride in the oil. However, inorganic chloride contamination in the oil, from such sources as seawater, would be additive. Whether chloride contamination is due to degradation of the compressor oil resulting in the production of HCl or is from some other source, it would degrade the operation of the R-11 plant.

In Table 4, used oil samples 3 to 6 displayed higher levels of chloride ions than the unused oil samples (1-2). Sample 2, although labeled as new oil, had a considerably higher level of chloride ions than sample 1. This was later confirmed to be due to contamination of sample 2. The source of contamination was not established.

It appears from the limited data presented in Table 4 that chloride ion levels increase with time of plant operation.

Table 4 — R-11 Plant Oil Samples

Oil Sample	Total Chloride (μg HC1/m1 oil)*	Remarks
1. New Oil, Octagon May 1973 Lot C 2699	0.7	Unused oil
2. New Oil, Davis Lot C 2621 Feb. 1973	2.0	DSA600-73-C-1402 EES 620659 B
3. Plant B Aug. 4, 1975	3.3	36 h plant operation
4. Plant B Aug. 2, 1974	7.7	420 h plant operation
5. Plant B SMMS A	14.5	No dates or time given
6. Oil from Charleston Naval Shipyard SMM 53 Unit 1 Date 4293	17.6	Origin not known

*Microcoulometric values are ±10%.

EVALUATION OF CLEANING SOLUTIONS

Dynamic Test

Various chemicals were evaluated for their relative effectiveness in solubilizing the R-11 plant residue and for their compatibility with metal components of the plant. Although these tests were not designed to yield quantitative engineering data, they were useful in selecting the most promising chemical solution or method.

In the first phase of this study, weighed portions of the residue were put into a stirred solution for various temperatures and time. At the end of each reaction the solution was filtered. The residue was then dried and reweighed to determine the percent of residue that went into solution. The solvents studied in this manner were critric acid and ethylenediaminetetraacetic acid (EDTA).

In its shipyard cleaning procedure, the Navy now uses a buffered citric acid cleaning solution. This treatment was tested by adding weighed portions of the deposit to a citric acid solution buffered to pH 4.5 and heated to 60°C for 1 h. As shown in Table 5, Method 1, 33% of the deposit dissolved. Lowering the initial pH of the solution to 3.1 improved the cleaning effectiveness slightly, 37% dissolved (Method 2). After lowering the pH and maintaining it at 2.5, 78% of the deposit dissolved (Method 3). For the latter experiment, the pH was adjusted at the end of 1 h, and the solution was heated and

Table 5 — Effectiveness of Various Refrigeration Plant Cleaning Agents

Method	Cleaning Agent	Initial pH	Final pH	Amount in Solution (%)	Comments
1	Ammonium citrate	4.5	5.5	33	Heated and stirred at 60°C for 1 h.
2	Ammonium citrate	3.1	3.5	37	Same as above.
3	Ammonium citrate (controlled pH)*	2.4	2.5	78	Heated and stirred at 70°C for 2 h. pH readjusted after 1 h.
4	Citric acid	2.2	3.6	41	Heated and stirred at 60°C for 1 h.
5	Citric acid with prior degreasing wash	2.2	2.9	40	Same as above.
6	Citric acid (controlled pH)*	2.2	2.4	77	Heated and stirred at 60°C for 2 h. pH readjusted after 1 h.
7	EDTA, pH adjusted with ammonium hydroxide	5.3	7.8	28	Heated and stirred at 60°C for 1 h.
8	EDTA, pH adjusted with ammonium hydroxide (lower pH than above)	3.7	7.3	34	Same as above.
9	EDTA, pH adjusted with ammonium hydroxide (controlled pH)*	3.7	2.8	81	Heated and stirred at 70°C for 3 h. pH readjusted after 1 and 2 h.
10	EDTA, Tetrasodium salt	10.8	10.8	16	Heated and stirred at 60°C for 1 h.
11	EDTA, Disodium salt	4.6	8.4	28	Same as above.
12	EDTA, Slurry	2.7	2.4	91	Heated and stirred at 70°C for 2 h. pH readjusted after 1 h.

*Acid was added during test to lower the pH.

stirred for 2 h. As can be seen by comparing the three methods, the lowered pH had a marked effect on the cleaning action, but continued heating and adjusting of the pH had little effect on increasing the solubility of the deposit beyond 78%.

Unbuffered citric acid (pH 2.2) with 1 h of heating resulted in 41% of the deposit going into solution (Method 4). Prior cleaning of the deposit with a degreasing solvent, trichloroethene (Method 5), did not increase the cleaning action; only 40% of the solid dissolved. Citric acid controlled at a pH of approximately 2.2 by adding acid during the test dissolved 77% of the deposit (Method 6). Thus, it can be seen that the present shipyard cleaning method of using the buffered pH solution of 4.5 is not as effective in solubilizing the plant residue as a more acidic solution.

EDTA presently used in the Fleet for dissolving boiler scale [7], was also found to effectively dissolve the R-11 plant residue. The R-11 plant deposit was added to an EDTA solution adjusted with ammonium hydroxide to pH 5. Heating this mixture for 1 h at 60°C resulted in only 29% of the solid dissolving (Method 7).

An EDTA solution adjusted with ammonium hydroxide to pH 3.7 resulted in 34% of the deposit dissolving after 1 h (Method 8). Controlling the pH at about 2.5 resulted in 81% of the deposit dissolving after 3 h (Method 9). Again, the reduced pH has a pronounced effect on the cleaning action of the solution.

EDTA as the tetrasodium salt, with pH 10.8 (Method 10), or as the disodium salt, pH 4.6 (Method 11), resulted in 16% and 28% of the deposit dissolving, respectively. An EDTA solution maintained at a pH of about 2.5 by adding more acid resulted in 91% of the deposit being dissolved after 2 h of heating (Method 12). At this low pH the EDTA is only slightly soluble, and the cleaning solution would have to be used as a slurry [8]. As long as the solid is present the pH will remain low. The solid EDTA can be removed from the system by increasing the pH of the solution and flushing with water.

Static Test

The second phase of this study was the evaluation of the two previously mentioned chemical reagents, citric acid and EDTA, and proprietary cleaning formulations Oakite 32 (Oakite Products, Inc., Berkeley Heights, N.J. 07922), Gamlen's Metal Brightner GP, (Gamlen Chemical, Sybron Corp., Norfolk, Va. 23510), and Zep Rust Remover (Zep Manufacturing Co., Atlanta, Ga. 30301) for their compatibility with the metal components in the R-11 plant in the presence of plant residue. This evaluation was conducted in the same manner as described previously, except that the solution was not stirred. This procedure was used to assess the effect of a cleaning solution standing in the plant. Again, we were looking for a way to compare and screen formulations for more extensive engineering testing under real conditions.

Metals representative of R-11 plant components were cut into small pieces. Each type of metal was used such that the weight percent composition corresponded to the approximate composition used in the plant: 50% carbon steel type 515, 20% red brass, 18% copper-nickel, 9% copper, and 3% silver alloy. A representative amount of plant residue, 3% by weight, was heated with the metals in the cleaning solutions at temperatures of 60°, 70°, and 80°C for 24 h. Under these conditions the steel exhibited the

Table 6 — Cleaning Formulation Tested

Cleaning Agent	Percent Weight Loss of Steel (Percent Residue Dissolved)[*]			
	24 Hours			48 Hours
	60°C	70°C	80°C	80°C
Citric Acid	10 (75)	8 (82)	10 (84)	22 (84)
EDTA	5 (84)	7 (90)	12 (91)	12 (91)
Oakite 32	2 (87)	4 (92)	6 (95)	—
Gamlen Metal Brightner GP	—	—	11[†]	—
Zep	0.9 (85)	31 (88)	44 (82)	—

[*] These values are ±2 percentage points.

[†] Because the formulation is apparently phosphoric acid, the residue was transformed to insoluble iron phosphate.

greatest loss of weight and was used as a criterion for attack on the R-11 system (Table 6). All the other metal components exhibited less than 0.5% weight loss at the temperatures tested. The percentage of residue dissolved from the R-11 plant is indicated in parentheses. Only Gamlen Metal Brightner GP failed to remove unwanted residue. This was attributed to the phosphoric acid content of the formulation and the insolubility of iron phosphates. Zep showed the highest attack of the steel at elevated temperatures, and the lowest at the lowest temperature.

The initial pH of both the citric acid and EDTA solutions was around 2.0. The pH of the EDTA solution increased to 5.9 after 20 h at 80°C, and additional EDTA was added to lower the pH to 2.2. After this solution was heated for 4 additional hours at 80°C, the pH remained at 2.2. The citric acid solution reacted similarly. However, after the addition of citric acid the pH again increased to a value of 3.5 after 4 h, indicating that the acid was still being consumed. This was verified by repeating the experiment and allowing the reaction to continue for 48 h.

As indicated in Table 6, using citric acid increased the dissolution of the steel but not of the residue. With EDTA, there was no change in the dissolution of either steel or residue within the 48-h period.

It is apparent from Table 6 that the Oakite 32 dissolved the most residue and at the same time attacked the steel the least. This proprietary formulation was tested at a concentration of 30% by volume. The initial pH of the solution was less than 1.0, indicating a strongly acidic solution. After 24 h the pH was 2.5.

SUMMARY AND CONCLUSIONS

1. It is apparent from the tests conducted, as well as from the available literature, that residue formation in the R-11 plant is preceded by the chemical reaction of the refrigerant with oil in the system. This reaction results in the formation of highly corrosive hydrochloric acid and possibly hydrofluoric acid, which are free to attack the metal components of the plant.

2. The formation and detection of R-21 and R-12 by degradation of R-11 could be used to assess the condition of the refrigerant plant before complete failure. The Total Hydrocarbon Analyzer now used on board most nuclear submarines could be used for this purpose.

3. Analysis of the residue and the deposits on an R-11 plant impeller indicated high concentrations of aluminum, copper, and iron.

4. The present shipyard method of solubilizing the residue with a buffered citric acid solution was less effective than using the same acid without buffering. However, the more acid solution reacts more strongly with steel plant components.

5. Both EDTA, used as a slurry, and Oakite 32 solutions solubilized 90 to 95% of the plant residue; the latter formulation was less reactive with metal plant components.

6. It appears that a cleaning solution containing Oakite 32 would most effectively solubilize the plant residue and would also be the most compatible cleaner for the metal components in the R-11 plant. However, a large-scale engineering test should be made to confirm this conclusion. Further, the EDTA slurry should be included in such a test for comparison.

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