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ATM C&D PANEL/EREP COOLING SYSTEM CONTAMINATION PROBLEM

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This report presents the history of a preflight contamination problem that occurred in the ATM C&D Panel/EREP Cooling System on the Skylab, the studies that were made by the Materials Division to determine the cause of the problem, and corrective actions that were made prior to lift-off.

The results of all the observations, analyses and laboratory testing indicated that the contamination came from one or more of the EREP Tape Recorder coldplates and was caused by some abnormal electrolytic action, either during bench testing or in the spacecraft. Studies indicate that no such electrolytic action is likely to occur under normal operating conditions.

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SUMMARY

This report presents the history of the preflight contamination problem that occurred in the ATM C&D Panel/EREP Cooling System on the Skylab, the studies that were made by the Materials Division to determine the cause and corrective actions that were made prior to liftoff.

The investigation consisted of a re-examination of the cooling fluid inhibitor system, an examination of all components that could possibly be observed with complete chemical analysis of any contamination found and laboratory studies to try to duplicate the contamination found in the system. A complete analysis of the contamination found in the filter system was made as the starting point of the investigation.

The results of all the observations, analyses and laboratory testing indicated that the contamination came from one or more of the EREP Tape Recorder coldplates and was caused by some abnormal electrolytic action either during a bench type test or in the spacecraft. Studies indicated that no such electrolytic action is likely to occur under normal operating conditions.

INTRODUCTION

The Apollo Telescope Mount (ATM) Control and Display (C&D) Panel and Earth Resources Experiment Package (EREP) Cooling System provides cooling to the two components by circulating an inhibited water coolant. The ATM C&D panel system contains essentially pumps, valves, heat exchanger, filter, coldplate and necessary tubing. The EREP system consists of primary and secondary tape recorder coldplates, Control and Display coldplate and S-192 electronic package coldplate. The EREP is located in the Multiple Docking Adapter (MDA) and the remainder of the system is in the Airlock Module (AM). A schematic of this system is shown in Figure 1.

The system had operated successfully for some 14 months on standby and in testing, and then during a 5 week period four months prior to lift-off the filter of the system became very contaminated. Therefore, the purpose of this investigation was to determine the cause of this problem and demonstrate a solution prior to flight. Several NASA Centers as well as several Skylab contractors worked on this problem and some of the history came from information obtained from these sources.

This report, however, is based generally on work conducted by MSFC Astronautics Laboratory, Materials Division personnel. Mr. Ralph Higgins and Mr. John Barnes participated in the analysis of the contaminated filter at MDAC-E in St. Louis, Mo., and Mr. Higgins also participated in the work conducted on Tape Recorder No. 5 at Ampex in Redwood City, California. Mr. Eli Nelson and Mr. James Lowery conducted the electrolytic phase of the study, and Mr. Jim Sulcer and Mr. Harold Ryan made the analytical analyses of all inhouse studies. Mr. O. Y. Reece participated in the examination of Tape Recorder No. 3, C&D coldplate and tubing located on the spacecraft and obtained much of the history of this problem from KSC.

TYPE I COOLANT

The water coolant for the system contains, by weight, 2% dipotassium hydrogen phosphate, 0.2% sodium tetraborate ($Na_2B_4O_7$) as corrosion inhibitors and 500 parts per million (ppm) Roccal (50% concentration) as a biocide. This is known as Type I coolant. The Roccal is a 50% aqueous solution of Benzalkonium chloride. Benzalkonium chloride is a mixture of Alkyl dimethylbenzylammanium chlorides.

The selection of the inhibitor system was dictated by several requirements other than the control of corrosion in the system. It was determined that the coolant must be non-toxic, compatible with the biocide, and compatible with the vapor-liquid separator because the same coolant must be used in the Suit Cooling Loop system. The use of the same coolant in the two systems would have reduced the qualification programs since several components are common in both systems. However, this plan had to be changed when it was found that the liquid-gas separator would not function in the presence of Roccal.

NASA/MSFC and MMC Materials and Processes personnel recommended using Sodium Chromate (Na_2CrO_4) and Sodium Dichromate ($Na_2Cr_2O_7$) as the inhibitor system for the coolant, however, the chromates would not meet all of the previously mentioned requirements. A system composed of 2% dipotassium hydrogen phosphate and 0.2% sodium tetraborate was recommended by Union Carbide Corporation engineers and MDAC-E Materials and Processes engineers. Although it was recognized that other inhibitors would be more satisfactory from the standpoint of inhibiting corrosion, tests indicated that this inhibitor system would be adequate.

Dipotassium hydrogen phosphate is an effective corrosion inhibitor when used in concentrations of 0.2% or higher. It is compatible with Roccal and is also approved for human consumption by the Food and Drug Administration. It is a sacrifical type inhibitor and becomes less effective as the hydrogen ion concentration is increased. (Ref. 1).

Borax based compounds, such as sodium tetraborate, are employed extensively as corrosion inhibitors. Borax is used in MIL-C-11755 ethylene glycol anti-freeze solution. This material is not effective with aluminum systems unless used with additives such as Sodium Mercaptobenzothizole (NaMBT) or phosphates. Sodium borate used with K_2HPO_4 is known to be an adequate corrosion inhibitor in ferrous/aluminum water systems. Federal Specification O-I-490A, entitled Inhibitor, Corrosion, Liquid Cooling System, includes sodium borate and phosphates for use in liquid cooled engines. (Ref. 1)

The pH of this Type I coolant is in the 9.2-9.3 range. As a general rule, the protective oxide film on aluminum alloy is stable in aqueous solutions in the pH range of 4.5 to 8.5. There are, however, certain acid and alkaline solutions in which aluminum is highly resistant to attack. A few examples are glacial acetic acid, concentrated nitiric acid, sodium disilicate and concentrated ammonium hydroxide. For this reason, the corrosivity of an environment cannot be determined solely by pH because the nature of the individual ions in the solution may be the controlling factor rather than the degree of acidity or alkalinity. (Ref. 2).

OPERATING HISTORY

The cooling system contains mainly 304L stainless steel (tubing) and 6061 aluminum (cold plates and tubing). The C&D panel cold plate has a chemical conversion coating (MIL-C-5541). Some of the EREP coldplates are coated and others are bare. A complete list of the materials in this system can be found in Appendix I.

The system was installed in the Skylab and was refilled with coolant for approximately 4 months during which it had some 438 hours of operation. The filter (S/N 9) was removed from the system in January 1973 and was found to be relatively clean. The filter had actually been in the system for 14 months, about 10 months before being moved to KSC. Figure 2 shows a complete filter assembly.

The replacement filter (S/N 24) was installed in the system and was subjected to only 35 hours of system operation during a five week period. The filter was examined and found to be about 80% covered with contaminants. It was estimated that the weight of the contaminants was from 2 to 5 grams. The contaminants on the filter did not cause a pressure drop nor was the flow restricted in any way.

The next filter (S/N 25) was installed in the system after the contaminated filter was removed. The system was exposed to the coolant for 2 weeks during which there was approximately 90 hours of operation. About 13 milligrams of material were removed from the filter. This small amount of contamination could have been generated during this period or it could have been some of the old contamination that had remained in the system. Analysis of this material identified it to be the same as that on No. 24 filter.

In order to determine the source of the contamination, data were compiled by all involved NASA Centers and contractors. These data indicated what EREP equipment was present in the system and what time each was operating during the KSC system testing. From these studies, the coldplates associated with the Tape Recorders became the prime suspect because they were the only items that contained any contamination when physically examined. The coldplates were also about the only aluminum items that had not been coated. Looking into these systems on the Skylab was very difficult. However, Tape Recorder No. 3 was examined and found to have some contamination, the ATM C&D panel and the stainless steel tubing was found to be clean. The contamination found on the tape recorder was not sufficient to cause a problem. Tape

Recorder No. 5 coldplate which was on the spacecraft during the time of the contaminated filter (S/N 24) was found to have some contamination. This Recorder was located at Ampex at the time of the inspection. Tape Recorder No. 2 was examined at MMC and found to be only slightly contaminated, considerably less than No. 5. See the next section on "Component Inspection and Analytical Studies" page five for a complete evaluation of the coldplates on the various tape recorders.

The tape recorder coldplates are not electrically grounded in the same manner as are other electrical components in the spacecraft. This could allow stray currents to enter the cooling system and could cause corrosion by electrolytic action.

It had also been determined several months ago by both MSFC and MMC that if the Type I solution was heated to 355°K (180°F) in the presence of aluminum, large amounts of white precipitate would form.

Since it was known that higher temperatures and stray currents could have been present on some of the components during testing (both in the system and individual bench tests), these two abnormalities could have produced the contamination in the system. It was generally agreed that the normal corrosion process of the system could not have contaminated the system in a 5 week period, when the normal corrosion process had produced only such a small amount of contamination during a 14 month period.

COMPONENT INSPECTION & ANALYTICAL STUDIES

The contaminated filter (S/N 24) removed from the cooling system after only 5 weeks of operation was the first indication of an abnormality in the system. This contamination was studied carefully in an effort to determine the source. The filter element was reported to be about 80% covered (no photographs were taken until after KSC had removed some of the contamination for analysis) and contained an estimated 2 to 5 grams of material. Although the filter appeared to be grossly contaminated, the flow in the system had not decreased nor was there any pressure drop across the filter.

KSC removed some of the contamination from the filter and sent the remainder to MDAC-E for a more complete analysis. Figure 3 shows the condition of the filter when it reached MDAC-E. Results of this analytical work indicated that the contamination was composed of metal phosphates, primarily aluminum and potassium. Minor components of the contamination may have included oxides or hydroxides, although this was not verified. Material Division personnel were present during the analytical study of the contamination at MDAC-E. The details of the finding of the analytical work on the contamination are given in Appendix II.

The analytical results obtained by the Materials Division compared with the results of MDAC and KSC. One additional material identified by the Materials Division was carbon. The amount found was 1.3% by weight and it probably came from the Roccal. This proved to be important in later work when efforts were made to reproduce the contamination in the laboratory.

The first component other than the filter to be inspected was Tape Recorder No. 5. This inspection showed some contamination to be present; however, the amount was small and very little pitting was noted. This recorder was examined more carefully later in the study. This recorder was not on the vehicle when it was examined.

The next component to be examined was Tape Recorder No. 3. At the time it was opened, this recorder was the spare recorder in the system. Approximately 200 ml of coolant were obtained from the recorder when it was opened. This fluid was analyzed. An infrared spectrogram of the residue filtered from the 200 ml matched that from the contaminated filter. Chemical analysis of the residue showed the following:

Phosphate	46.5% by wt.
Aluminum	13.7%
Potassium	22.8%

One of the important findings of this operation was that the pH of this fluid was 10.5. This was the first abnormality found in the system after the gross contamination was found in the filter (S/N 24).

When this condition was found in the system, it was decided to conduct a full system test. The system was drained and a charge of new solution and a new filter were put into the system. After about 5 minutes of running, the system was sampled. After 5 hours the system was again sampled and then after about 11 more hours in which the EREP system was operated part time, a final sample was taken. The filter was removed for examination. A well defined procedure for analyzing the samples was worked out at MSFC with personnel from MSFC, JSC, Martin and Ames Research Center. This procedure is given in Appendix III.

The overall results of this test indicated that the system was clean and that contamination was not being generated to any great extent. Contaminant on the filter at the end of the total test was 0.00055 grams and in the filter housing there was an additional 0.0032 grams of contaminant. The total weight of contamination from the filter after being washed was 0.0835 grams. This weight included mainly the inhibitors in the solution that were left on the filter when it dried. A complete report of the analysis of this test is given in Appendix IV.

Early in the study, EREP Tape Recorder No. 5 was examined at Ampex as discussed above. This recorder had been in the spacecraft during the time that the first two filters (S/N 9 & 24) had been in the system. An inspection of the cooling tubes (coldplate) was made by JSC, MDAC and Ampex personnel. At the steel/aluminum inlet and outlet connections there was an uneven coating of white flaky contaminant. There was no indication of pitting. A fiberscope was used to inspect a short distance into the aluminum tubing. There was an even coating of white contamination tightly adhered to the surface.

When it became known that stray electrical currents were associated with the tape recorders, the No. 5 tape recorder was again examined. At this time, personnel from MSFC, JSC, Martin, MDAC-E and Ampex participated. The purpose of this examination was to obtain electrical resistance measurements of the recorder coolant lines and fittings, and to inspect the lines and fittings more closely for contamination. Typical results are shown in Figure 4. A drawing of the tape recorder coldplate is shown in Figure 5. The results of this investigation were as follows:

1. Extremely low resistance measurements were found at all stainless steel fitting to aluminum tubing joints. The measurements ranged from 0.06 to 0.95 milliohms, generally they were from 0.1 to 0.2 milliohms.

2. Visual examination was made of about 1.52 meters (5 ft.) of the 4.68 meters (15.35 ft.) of aluminum tubing making up the coldplate.

3. Most of the contamination found was at the inlet and outlet to the recorder (only a few milligrams).

4. Nickel Voishan washers are used only on the inlet and outlet lines of the coldplate. These are used only if the joints leak.

5. A small amount of white powdery coating was found inside all openings, some loose and some tightly adherent. It was more concentrated at fitting joints and tube bends. Deposit thickness was estimated to be 0.005 to 0.01 cm (0.002 to 0.004 inch) thick in some areas.

6. Superficial etching was noted at the interface of the stainless steel to aluminum joints. The etched areas were bright and shiny and appeared to be more than 0.00254 cm (0.001 inch) deep. This was not observed until the deposit was removed by rubbing with a dry cotton swab.

The conclusion drawn from this examination was that this coldplate probably contributed to the contamination problem, but in retrospect it does not appear to have been the only contributor.

The last equipment examined in the effort to locate the cause of the contamination was the coldplate of Tape Recorder No. 2. This recorder had

10 hours of recording time, and had been installed for a duration of 2 months. The report on this examination indicated that this was the cleanest of 3 tape recorders examined. Prior to the examination, the recorder had been drained, flushed with demineralized (D. I.) water, purged with nitrogen and then vacuum dried for 18-20 hours. The observer was able to see 10 cm (4 inches) into the outlet line. There was no buildup at the stainless steel aluminum interface as had been seen on the other recorders. The inlet tube was observed for distance of 30.5 cm (12 inches). Nothing was visible at the union and no buildup was seen at the interface. There was a very light, white deposit one-half way around the tube. This may have been water or inhibitor residue.

LABORATORY STUDIES

Corrosion studies using Type I solution were conducted by this Division about two years ago. Bare 6061 aluminum panels were exposed for 18 months with only minimal corrosion resulting which led to the conclusion that the inhibitor system was satisfactory. The amount of aluminum loss on the specimens was equivalent to 0.032 mils/year (0.000081 cm/yr.). In a shorter 8 months test, a 2014-T6 aluminum - 302 CRES couple showed no visible corrosion or accelerated attack at the interface.

Other studies of this type were conducted by the Martin Company and by the McDonnell Douglas Company with essentially the same results.

Probably the most comprehensive test was the Endurance Test conducted on the actual system by the McDonnell Douglas Company. This was a 12 month test in which the fluid was circulated continuously. The EREP package was not in the system at this time but the same area of aluminum in the package was present in the test system in the form of 6061 aluminum tubing. This constituted a satisfactory simulation from the standpoint of corrosion testing.

At the end of this 12 month test there was no evidence of corrosion or corrosion products. The chemical analysis of the fluid indicated that no aluminum went into solution.

1. TEMPERATURE EFFECTS

When the contamination was found in the system in March 1973 after only five weeks of service, this Division started a detailed study of the problem. At first the emphasis was placed on the fact, learned several months ago, that a temperature of $355^{o}K$ ($180^{o}F$) would cause rapid precipitation of the solution if aluminum was present. Since the contamination had appeared to form rapidly, this seemed a logical approach.

The initial test in this series consisted of placing 96.8 sq. cm (15 sq. inches) of 6061 aluminum in 450 cc of Type I solution. The solution was maintained at 352°K (175°F) for 65 hours. The 6061 aluminum panel had been cleaned and etched prior to exposure. After 65 hours, the test was stopped and the solution allowed to cool. No suspended matter was evident when the solution was hot; however, after reaching room temperature the coolant contained a fine precipitate which resembled a colloidal suspension. The suspension was broken by boiling the coolant, and the suspension was allowed to settle. The residue was filtered, dried and analyzed. The total weight of the residue (dried) was 0.040 grams. The elemental analysis showed 10.2% aluminum, 7% potassium and 29.4% PO_4 based on P. The carbon content of this particular sample was not analyzed but similar tests had a carbon content of about 30%. The x-ray diffraction analysis did not show a pattern. This material did not resemble the contaminant that was taken from No. 24 filter in appearance nor was the chemical analysis similar. As shown earlier, analysis of the filter showed 14% aluminum, 21% potassium, 45% PO_4 based on P and 1.3% carbon. This may have been due to the ratio of aluminum to the volume of solution. In this test it was 4.65 cc of solution per cm^2 of aluminum. This gave a residue production rate of 6.35×10^{-5} gm/cm²/hr at 352°K (175°F).

The next test was designed to produce sufficient residue to allow a more complete chemical analysis. A large area of 6061 aluminum (231 sq. cm) was put in 900 cc of Type I coolant and heated at 349°K (170°F) for 21 hours. This produced 0.1113 grams of precipitate at a ratio of 3.88 cc of solution per cm^2 of aluminum. The analysis of this precipitate showed the material to be essentially the same as the material produced in the previous test. This sample was analyzed for carbon and was found to contain 31.4% by weight. This was much greater than the slightly over 1% found in the contamination on No. 24 filter. The carbon came from the Roccal used in the solution as a biocide.

A series of tests were then made to determine at what temperature a significant amount of residue would form. The first tests were conducted at 322°K, 328°K and 333°K (120°F, 130°F, 140°F). The effect of a galvanic couple in the system was also investigated in this series of tests. The tests were conducted in duplicate. One set consisted of 5 specimens, 3.8 cm x 10 cm (1.5 inches x 4.0 inches) immersed in 900 cc of Type I solution and the other set was the same except each of the five specimens (total area - 387 cm^2) had a 2.54 cm square piece of 304L CRES bolted to the center of each specimen. This duplicated the stainless steel fittings which are in the tape recorder coldplates. All of the specimens were degreased, cleaned and etched in an acid cleaner. These duplicate sets were heated in a constant temperature water bath for 15 hours.

At the conclusion of the test, all of the solutions appeared somewhat opaque. The solution became very milky upon cooling to room temperature as shown in Figure 6. Table I and Figure 7 give the results of these tests. The spectrographic analysis of the residue from the solution formed at 333^{o}K (140^{o}F) with the aluminum specimens showed the following:

Major: Al
Minor: K, P, B, Na, Mg
Trace: Fe, Cr, Si, Cu, Mn

Two other series of tests were made identical to the above test except the times were reduced to 4 hours and 2 hours. The specimens were cleaned and etched prior to these tests. Tables II and III and Figures 8 and 9 show the resulting weight loss of these specimens. The rate of attack is reduced with time even at the higher temperatures, as shown in Figure 10.

2. SURFACE AREA EFFECTS

In order to more closely simulate conditions in the actual Skylab system, studies were conducted using the exact ratio of surface area of 6061 aluminum to volume of Type I coolant as found in the EREP Recorder coldplate. Sufficient specimens of the size used in previous tests were placed in a special glass container and heated to various temperatures as was done in the initial studies. The aluminum specimens had a total combined surface area of 2,516 cm^2 (390 sq. in.). A solution volume of 500 cc of Type I coolant was used, which gave the same surface area-to-volume ratio that is in the EREP Recorder Coldplate. Again the worst type of conditions were used by cleaning and etching the aluminum prior to exposure to the coolant. This fresh surface would simulate a system that had not previously been exposed to the inhibitors and thus did not have a passive film. Tests were conducted at 322^{o}F (120^{o}F), 328^{o}K (130^{o}F) and 333^{o}K (140^{o}F).

The results of these studies are shown in Table IV and Figure 11. The residue produced was not significantly greater at 333^{o}K (140^{o}F) than that produced with a much smaller (6.5 times less) amount of aluminum. However, it can be noted that the temperature had much less effect on the amount of residue produced than it did with the studies in which a smaller area of aluminum (390 sq. cm) was used. The studies using the smaller amount of aluminum (390 sq. cm) produced 7.8 mg at 322^{o}K (120^{o}F) while the tests using the larger amount of aluminum produced 49.5 mg at the same temperature or 6.35 times more. Since the area of aluminum was 6.5 greater, this would be the expected result. However, the larger amount of aluminum produced only 56.8 mg. of residue at 333^{o}K (140^{o}F) or only 1.146 times that produced at 322^{o}K (120^{o}F) under the same conditions. Using the smaller amount of aluminum, 5.78 times more residue was produced at 333^{o}K (140^{o}F) than was produced at 322^{o}K (120^{o}F).

An 18 hour test at 333°K (140°F) was conducted exactly as the above test (simulating the EREP recorder coldplates) to determine the amount and type of residue that would be produced. The specimens were cleaned but not re-etched as was done on all previous tests.

The results of this test are shown in Table IV. Due to the coating of the specimens by the inhibitors the amount formed in 18 hours at 333°K (140°F) was even less than that formed in 4 hours at 322°K (120°F) - 44.9 mg vs. 49.5 mg. This more closely represents the condition of the system after the aluminum tubing of the bare aluminum coldplate has been exposed to the solution for a few hours.

3. TEMPERATURE CYCLING EFFECTS

Additional studies were made using the simulated coldplate approach at lower temperatures to try to determine at what temperature the residue starts to form. Using the same procedures as the previous tests, studies were made at 312°K (100°F) and 316°K (110°F). The solutions were cooled to room temperature every 15 minutes and observed for residue. Nothing was noted for the first hour and 45 minutes. However, at the end of two hours both solutions had some residue. The 312°K (100°F) solution contained 8.96 milligrams of precipitate (dried) and the solution heated to 316°K (110°F) had 6.67 milligrams (dried). This was sufficient precipitate to give the solution a very very slight milky appearance.

Another test of the same type as has just been described consisted of exposing the 6061 aluminum specimens to Type I solution for 3 hours at 312°K (100°F) and 316°K (110°F). At the end of two hours exposure, the solutions were cooled to room temperature and visual observation was made. It was questionable whether or not any visible precipitation had formed. Chemical analysis showed the solutions contained 11.9 ppm aluminum (312°K) and 23.1 ppm aluminum (316°K) after two hours.

The tests were resumed for another hour at the designated temperatures. After a total of three hours, the solutions appeared slightly milky. The solution maintained at 312°K (100°F) had 8.4 mg. of residue (dried) and the solution maintained at 316°K (110°F) had 5.9 milligrams (dried). The reason for the slightly higher residue content at the lower temperature is not known; however, both are rather insignificant amounts considering that 2516 sq. cm of aluminum was exposed.

The results of all of the tests conducted by heating the Type I solution in the presence of 6061 aluminum has shown that an extremely fine precipitate will be formed. However, the precipitate or residue that was formed by the heating methods did not resemble the material taken from the filter in the coolant system either in appearance or chemical analysis.

4. ELECTROLYSIS EFFECTS

Investigations of the electrical grounding of the components of the coolant loop indicated that the tape recorders are the only items that are not grounded to the Skylab frame. The recorders are grounded to the coolant loop and this could allow current to flow through the coolant, which could cause excessive corrosion of the aluminum by electrolysis. Tests were set up to determine the effect of electroylsis of this Type I solution. Two electrodes, both aluminum or one aluminum and one stainless steel were placed in a beaker of Type I solution with a potential across the electrodes. The aluminum had to be the anode for the residue to be produced.

Several studies were made by passing a current into the Type I solution. The first test consisted of using 3.8 cm x 10 cm (1.5 inches x 4 inches) aluminum electrodes in a 600 cc beaker filled with 540 cc of Type I coolant. The electrodes were immersed approximately 3/4 of their area into the coolant. The electrodes were about 1 cm apart. Approximately 28 volts DC (to simulate Skylab conditions) was applied for about 4 hours.

A considerable amount of residue was formed (251 milligrams) in the 4 hour test. This material visually resembled the residue that had been seen on the filter (No. 24) removed from the coolant system. The chemical analysis of the dried residue also very closely matched that found in the actual system. The elemental analysis of the dried residue is as follows:

Al	14.7% by wt.
K	13.4%
PO_4	46.0% (based on P)
C	6.2%

Many tests of this type were conducted and it was found that large amounts of the residue could be formed in a rather short time. One test operating at 100 volts produced 287 milligrams in one hour. It was also found that if the electrodes are completely immersed in the coolant as shown in the right beaker of Figure 12, no residue would be formed. However, if there are irregularities in the electrode as shown in the left beaker of Figure 12, the residue will form. Note the cavity made by bending the anode is completely filled with residue. In this particular study, 28 volts were used in each test.

5. EFFECTS OF pH

As stated earlier in the History section of this report, one of the Tape Recorder Coldplates (No. 3) was found to contain Type I solution that had a pH of 10.5. It was found in these electrolysis studies that over a period of time (about 24 hours) the pH of the solution would approach 10.5. This pH rise appeared to be essentially time dependant. The voltage was varied from 28 to 100 volts and the time to reach 10.5 was about 24 to 36 hours. A test was conducted to obtain some of the electrolysis residue that was produced over a period of time sufficient to give a high pH. The test was set up using two 6061 aluminum electrodes and solution volume of 740 cc. The test was conducted for about 24 hours and the potential was 28 volts. The pH of the Type I solution at the end of the test was 10.35. The weight of the residue was 2.00039 grams. The analysis of the residue was as follows:

Al	9.81%	by wt.
K	11.68%	
PO_4	48.8%	(based on P)

The inhibitors were depleted somewhat by this reaction as would be expected. The following Table shows the amount of depletion:

	Before, %	After, %
$Na_2B_4O_7$	0.197	0.186
K_2HPO_4	2.030	1.760

6. SIMULATED COLDPLATE TEST

The last part of this series of studies was made on a simulated Tape Recorder Coldplate. The coldplate was fabricated using the same size and type 6061 aluminum tubing (Figure 13), 0.95 cm (3/8 inch) in diameter with a 0.089 cm (.035 inch) wall thickness. The length of 6061 aluminum tubing was 457 cm (180 inches) with 5 stainless steel couplings and 107 cm (42.1 inches) of 304 CRES tubing. The only difference in this coldplate and an actual coldplate was the geometrical configuration.

The purpose of this study was to determine if the residue that could be created in beakers and electrodes could be made in an actual coldplate. The pH of the Type I solution was 9.2 and the fluid was heated to 306°K (90°F). The coolant was pumped at 31.8 kilograms (70 pounds) per hour which is the same as the actual system flow rate. The resistance of the system with the fluid pumping was 0.037 ohms, at 1 volt and 19.5 amps. The test was run for one hour and 25 minutes, and all the tubing and couplings were dismantled and inspected. There were some very light white deposits at CRES to aluminum couplings. No deposit or residue was seen in the reservoir.

The next test in this series was essentially the same as the first except it ran for 69 hours. There was a very slight amount of white deposits at the CRES to aluminum joints and the solution appeared to have a very slight amount of precipitate.

Since no appreciable amount of residue was being formed in the simulated coldplate, more resistance was introduced into the system by applying Teflon tape to insulate two of the couplings (one on the CRES side, the other on the aluminum side). The resistance of the system was increased to 8 ohms. The test was conducted at 28 volts and the current varied from 7 1/2 to 10 milliamps. The Type I coolant was heated to 306°K (90°F) as in the other tests. This test was conducted for 1 hour and 35 minutes. There was no evidence of any formation of precipitate or residue, but there was some evidence of pitting on the aluminum fitting. There was only very slight evidence of pitting on any of the actual Tape Recorder coldplates that were examined although the area that could be viewed was small. These particular tests and the apparent condition of the coldplates indicate that the effect of an impressed current along the coolant loop will not significantly accelerate the corrosion process in the absence of a high resistance joint; however, the effect of stray currents are well known and can cause rapid corrosion under some conditions.

CONCLUSIONS

The following conclusions can be drawn from these studies:

1. The inspections of the various components of the actual cooling system failed to find the exact source of the contamination.

2. The acceptability of the Type I solution was verified if the normal operating temperature of 299°K (78°F) is maintained (no precipitate formed).

3. The precipitate or residue that is formed during a heating process does not resemble either visually or chemically the residue found on the contaminated filter.

4. The material produced by electrolysis did appear to be like the residue or precipitate on the contaminated filter and matched the chemical analysis closely. This material, however, could not be precisely reproduced under simulated coldplate conditions.

The above listed facts obtained from this study indicate that it is highly probable that the contamination found on the flight filter was produced by an abnormal electrolytic reaction in one of the EREP tape recorder coldplates, either during a bench type test or while the component was on the spacecraft.

REFERENCES

1. "Study of Corrosion Inhibitors for the Airlock Module LCG and ATM Controls and Display Panel Cooling Systems" SCD Group Technical Note Airlock No. E453-82, McDonnell Douglas Astronautics Company Eastern Division.

2. "Aluminum" American Society for Metals, Metals Park, Ohio, Volume 1, "Properties, Physical Metallurgy and Phase Diagrams" pp 220-221, Edited by Kent Van Horn, 1967.

3. Contamination in the ATM C&D Panel and EREP Cooling System, Airlock Project Design Note E451-5, April 20, 1973, prepared by S. Shrage, McDonnell Douglas Astronautics Company - East, St. Louis, Mo.

TABLE I
Exposure of 6061 Aluminum And CRES Specimens To Type I Solution For 15 Hours At Various Temperatures

Analysis	322°K (120°F) Specimens		328°K (130°F) Specimens		333°K (140°F) Specimens	
	Al	Al + SS	Al	Al + SS	Al	Al + SS
pH Solution	9.26	9.24	9.29	9.29	9.29	9.28
Al Conc. in solution (ppm)						
● Before filtration	35	30	47.5	37.5	63.8	55.0
● After filtration	35	28	44.5	35.0	53.0	47.5
Wt. of Al removed from Specimens (milligrams) (based on soln. analysis)	31.5	27.0	42.8	33.8	57.4	49.5
Wt. Loss of Specimen (mg) (actual weighing)	16.9	13.0	21.3	15.7	28.9	21.8
Wt. of Residue, dried (milligrams)	13.9	2.0	33.9	8.0	75.9	43.3
Wt. of Residue per Hour (milligrams)	0.93	0.13	2.26	0.53	5.06	2.88

TABLE II
Exposure Of 6061 Aluminum And CRES Specimens To Type I Solution For 4 Hours At Various Temperatures

Analysis	322°K (120°F) Specimens		328°K (130°F) Specimens		333°K (140°F) Specimens	
	Al	Al + SS	Al	Al + SS	Al	Al + SS
pH Solution	9.25	9.25	9.25	9.25	9.26	9.25
Al conc. in solution (ppm)						
● Before filtration	19.4	14.4	28.8	25.0	37.5	32.0
● After filtration	18.0	13.5	26.5	23.5	37.5	35.5
Wt. of Al removed from Specimens (milligrams) (based on soln. analysis)	17.5	13.0	25.9	22.5	33.8	33.8
Wt. Loss of Specimen(mg) (actual weighing)	2.8	5.0	15.3	10.5	18.8	16.9
Wt. of Residue, dried (milligrams)	7.8	7.6	15.2	6.0	45.0	15.5
Wt. of Residue per Hour (milligrams)	1.95	1.90	3.80	1.50	11.3	3.88

TABLE III
Exposure of 6061 Aluminum And CRES Specimens To Type I Solution For 2 Hours At Various Temperatures

Analysis	322°K (120°F)		328°K (130°F)		333°K (140°F)	
	Specimen		Specimen		Specimen	
	Al	Al + SS	Al	Al + SS	Al	Al + SS
pH Solution	9.22	9.20	9.22	9.22	9.22	9.22
Al conc. in solution (ppm)						
• Before filtration	6.25	6.25	11.25	10.63	16.88	14.00
• After filtration	5.00	7.00	9.00	13.00	20.00	21.00
Wt. of Al removed from specimens (milligrams) (based on soln. analysis)	5.6	5.6	10.1	9.6	15.2	18.0
Wt. Loss of Specimen (mg) (actual weighing)	1.3	-	6.8	3.8	10.8	7.8
Wt. of Residue, dried (milligrams)	19.5	4.3	47.4	14.9	75.9	34.1
Wt. of Residue per Hour (milligrams)	9.75	2.15	23.7	7.45	37.95	17.05

TABLE IV
Exposure Of 6061 Aluminum To Type I Solution For 4 And 18 Hours Simulating EREP Recorder Coldplate* At Various Temperatures

Analysis	4 Hr. Test			18 Hr. Test
	322°K (120°F)	328°K (130°F)	333°K (140°F)	333°K (140°F)
pH Solution	9.35	9.35	9.35	9.30
Al conc. in soln. (ppm)				
• Before filtration	70.6	92.5	100.0	78.1
• After filtration	62.0	83.5	87.5	72.0
Wt. of Al removed from specimens (milligrams) (based on solution analysis)	35.3	46.3	50.0	39.1
Wt. Loss of specimen (mg) (actual weighing)	12.6	23.7	29.9	24.6
Wt. of Residue, dried, (mg)	49.5	56.7	56.8	44.9
Wt. of Residue, per Hr. (mg)	12.4	14.2	14.2	2.5

* 6061 aluminum surface area 2,516 sq. cm (390 sq. in.) immersed in 500 cc of Type I solution.

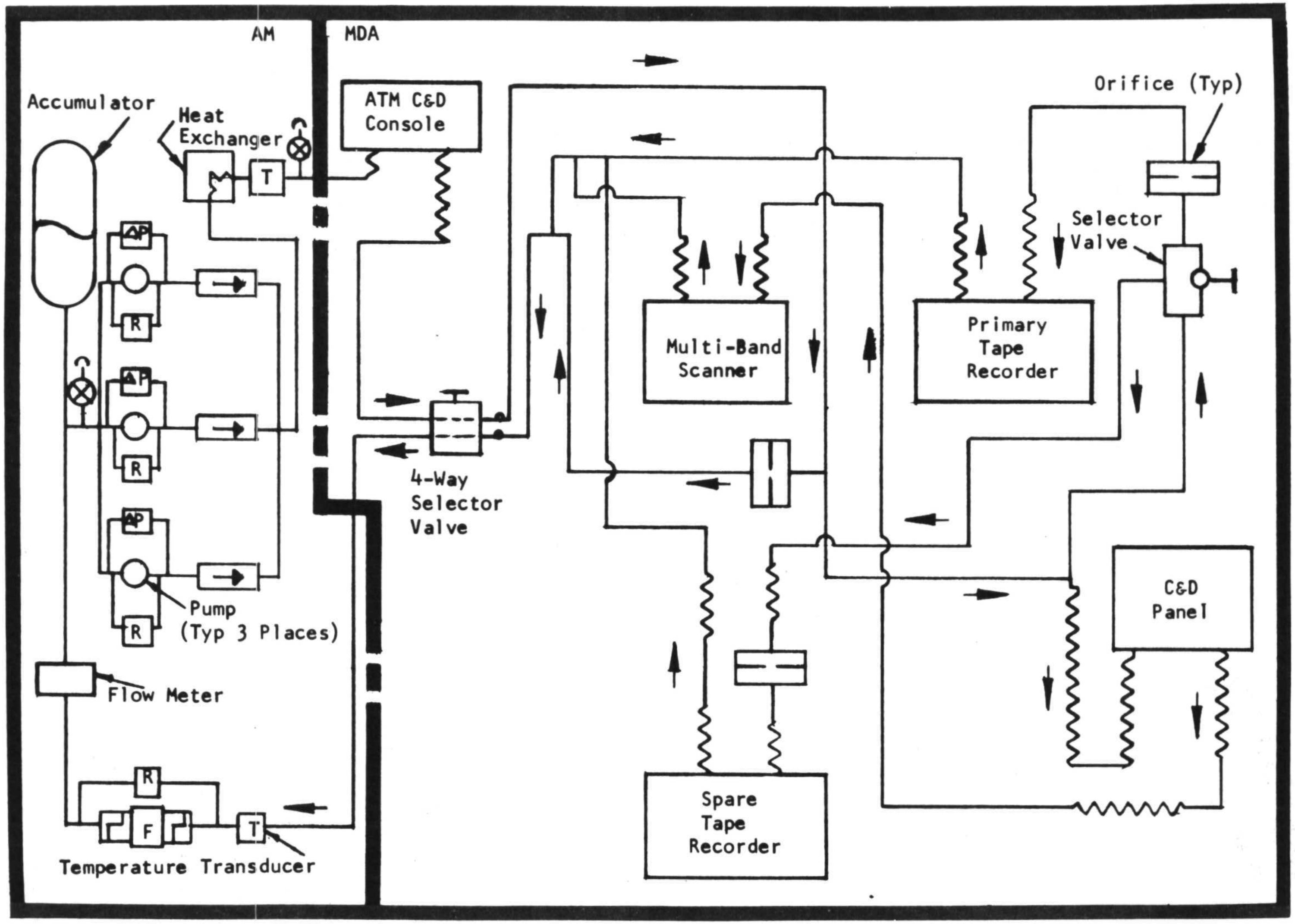


FIGURE 1 - SCHEMATIC OF ATM C&D PANEL/EREP COOLING SYSTEM

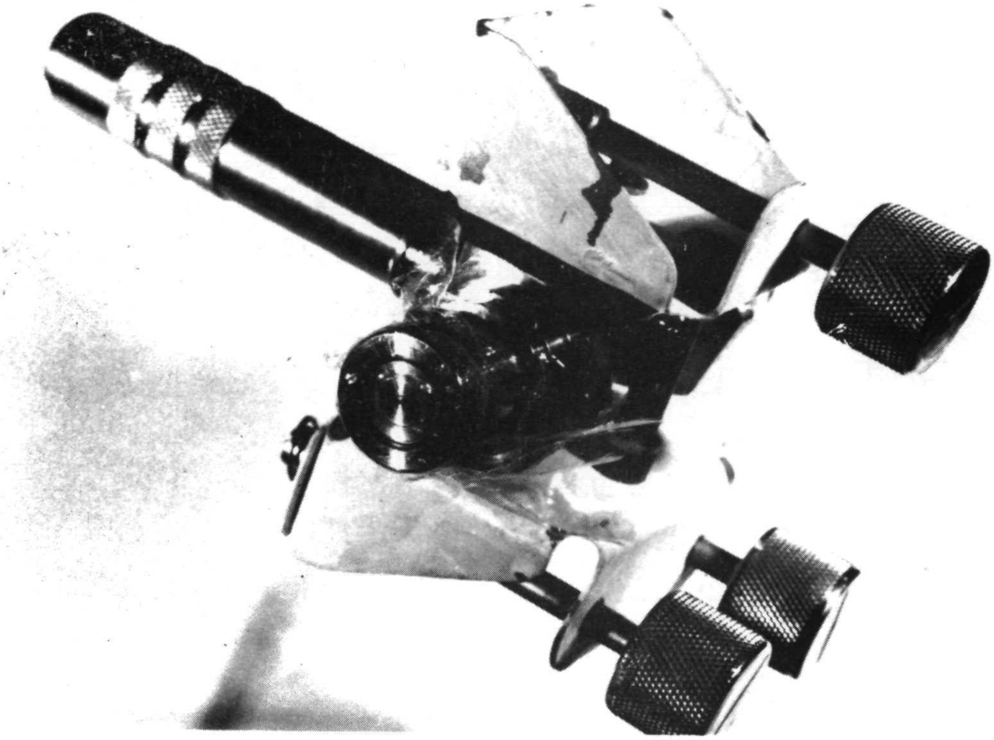


FIGURE 2 - FILTER ASSEMBLY

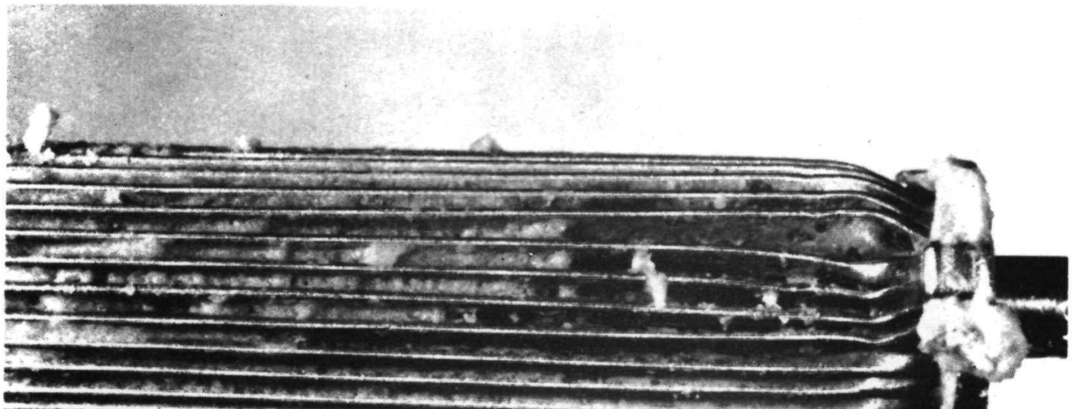


FIGURE 3 - CONTAMINATED FILTER S/N 24 (SOME MATERIAL REMOVED)

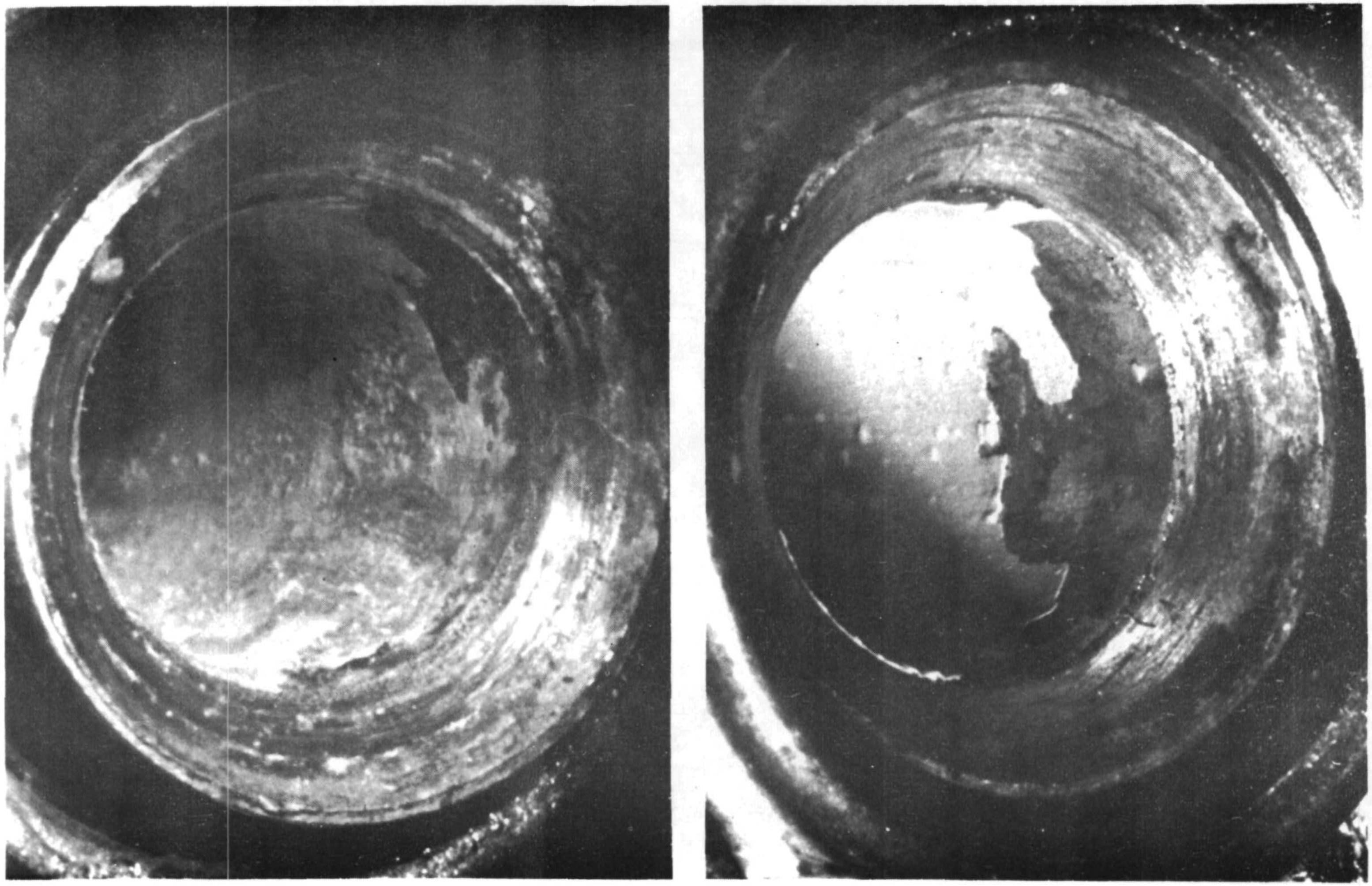


FIGURE 4 - CONTAMINATION IN COOLING COIL OF EREP TAPE RECORDER NO. 5

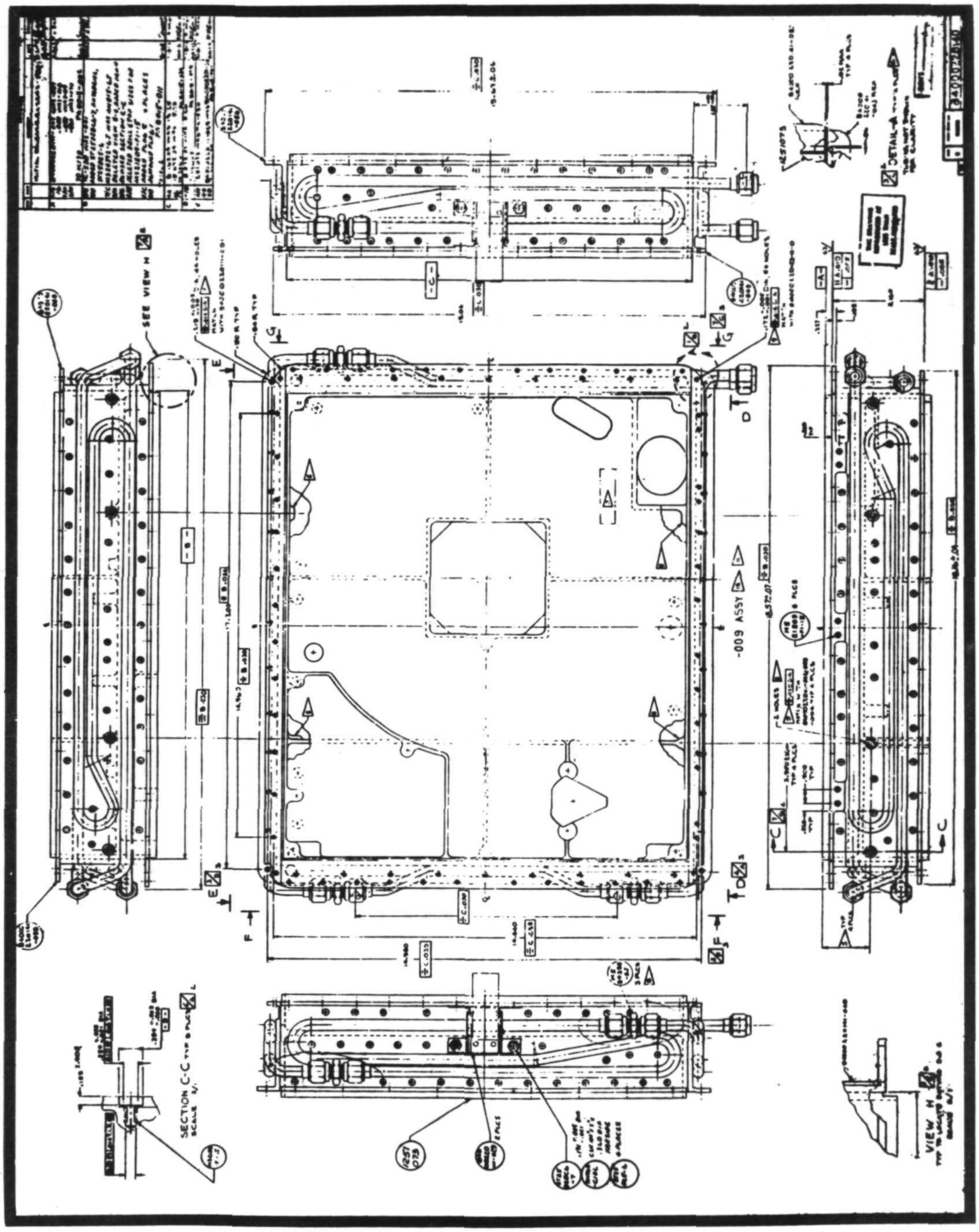
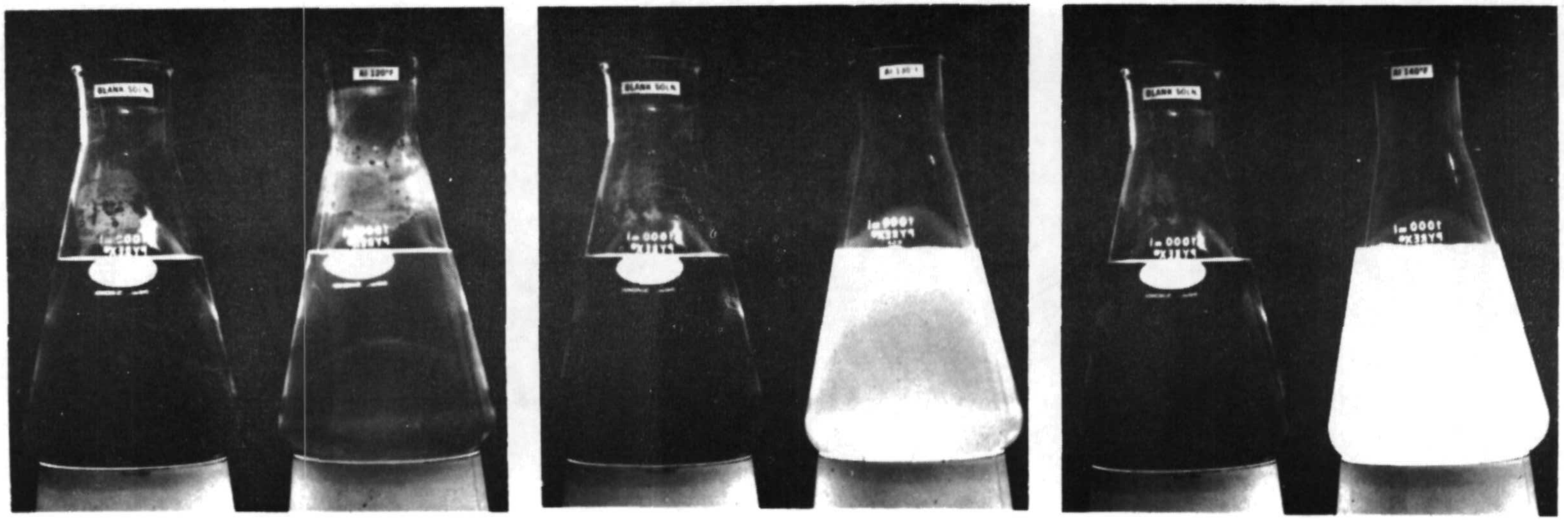


FIGURE 5 - SKETCH OF EREP TAPE RECORDER COLDPLATE



322°K (120°F)

328°K (130°F)

333°K (140°F)

FIGURE 6 - TYPE I SOLUTION HEATED TO VARIOUS TEMPERATURES FOR 15 HOURS WITH 6061 ALUMINUM SPECIMENS

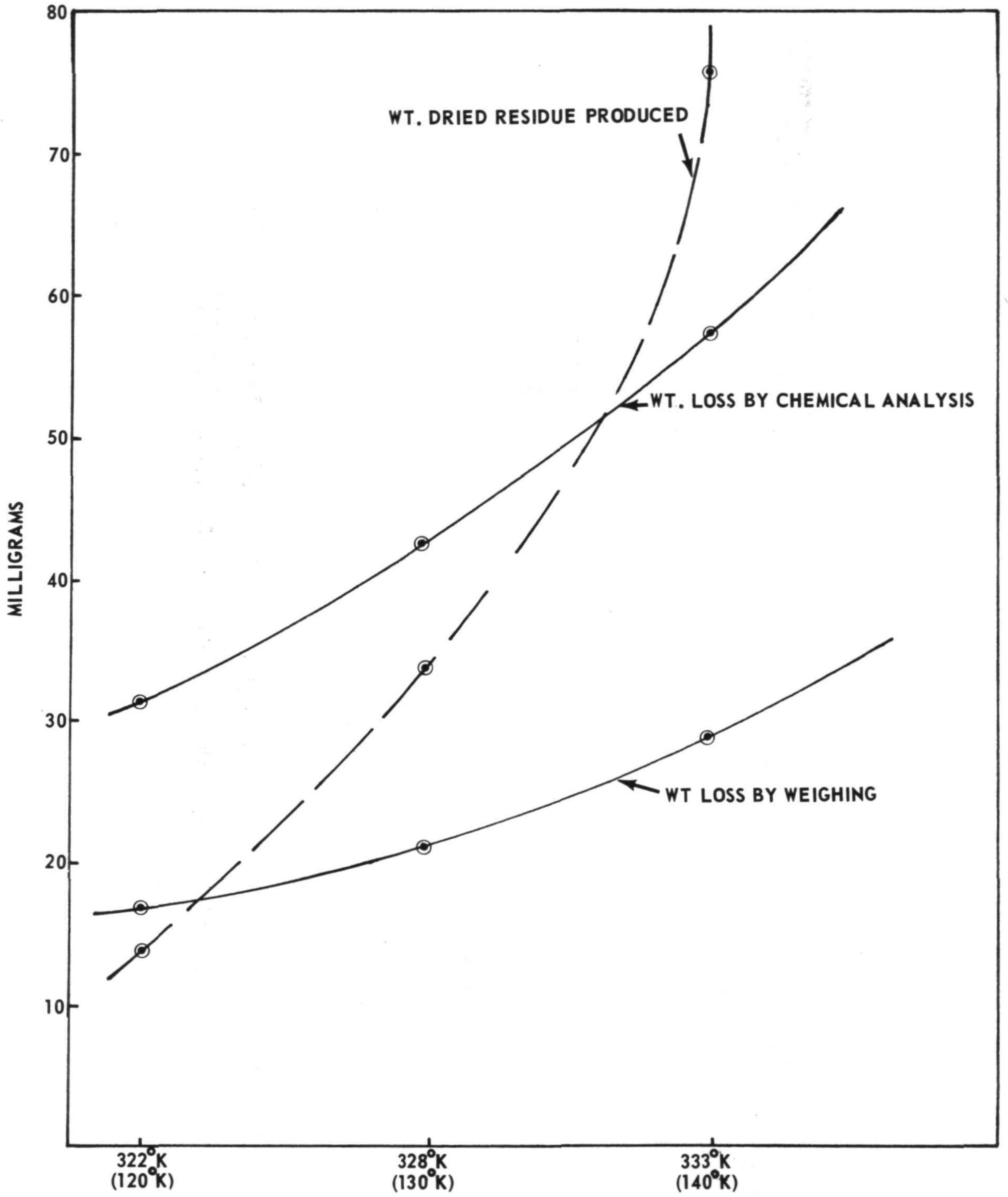


FIGURE 7 EFFECT OF EXPOSING 6061 ALUMINUM IN TYPE 1 SOLUTION FOR 15 HOURS AT VARIOUS TEMPERATURES

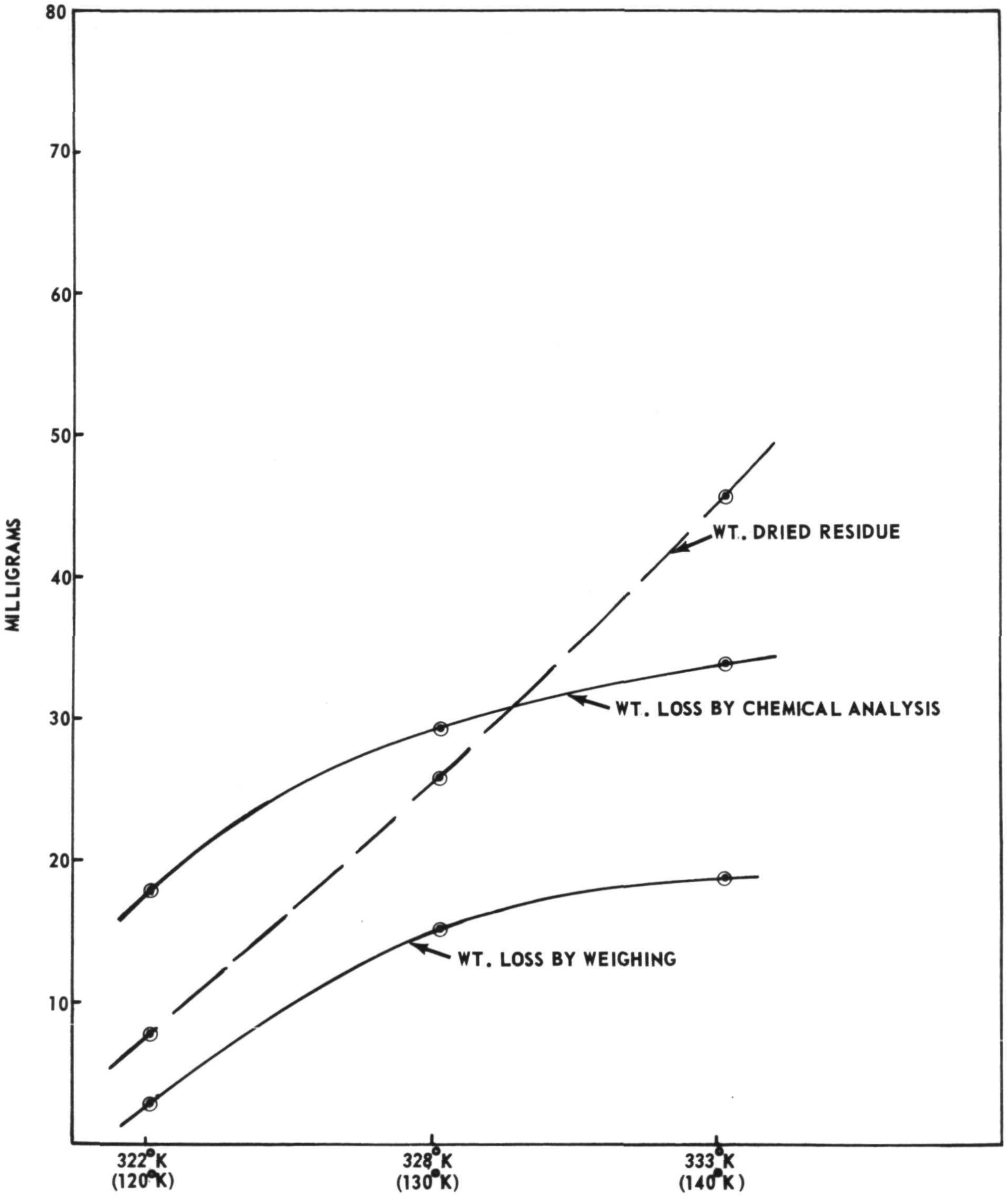


FIGURE 8 EFFECT OF EXPOSING 6061 ALUMINUM IN TYPE 1 SOLUTION FOR 4 HOURS AT VARIOUS TEMPERATURES

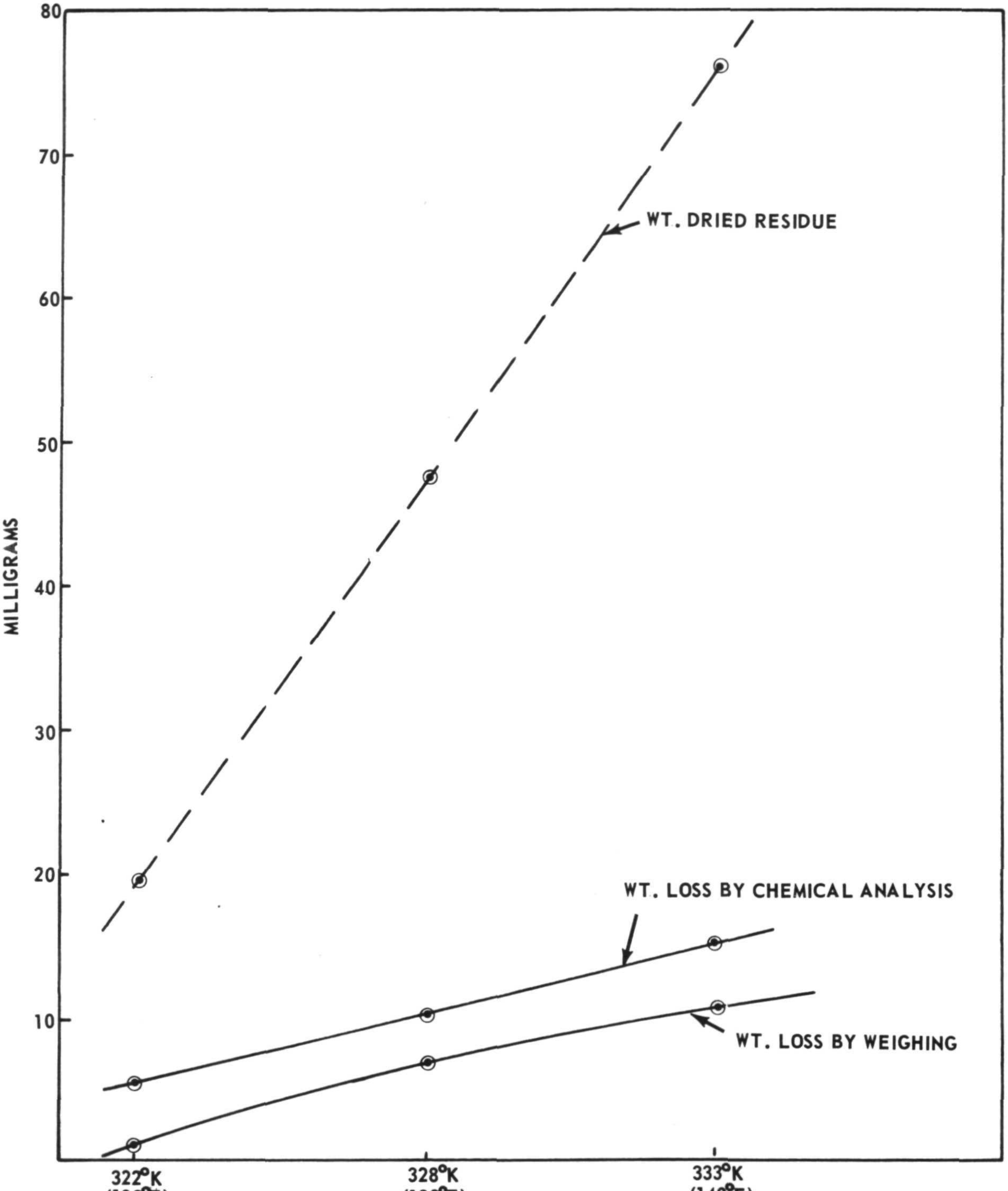


FIGURE 9 EFFECT OF EXPOSING 6061 ALUMINUM IN TYPE 1 SOLUTION FOR 2 HOURS AT VARIOUS TEMPERATURES

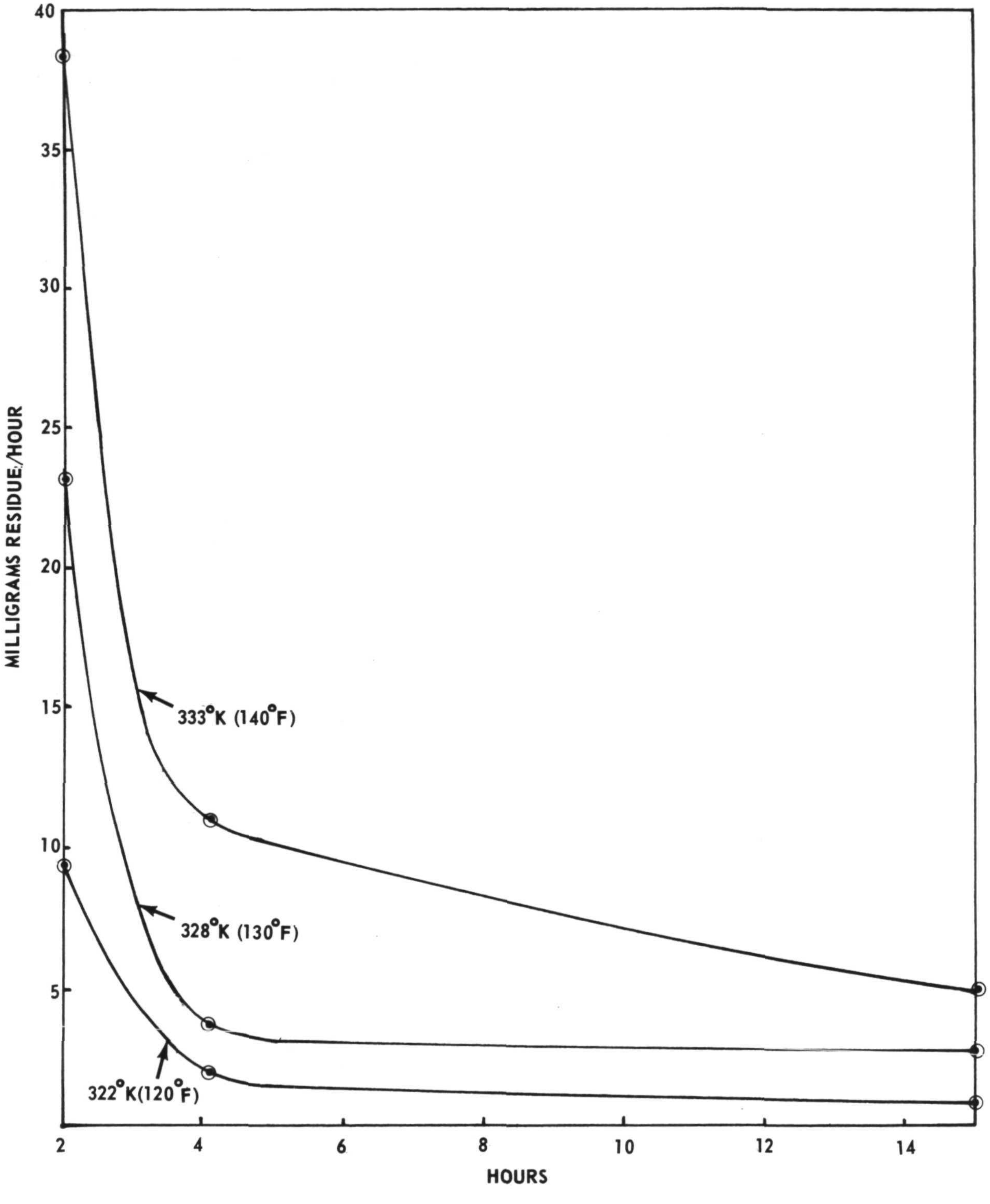


FIGURE 10 RATE OF RESIDUE FORMED PER HOUR FROM EXPOSING 6061 ALUMINUM TO TYPE 1 SOLUTION AT ELEVATED TEMPERATURES

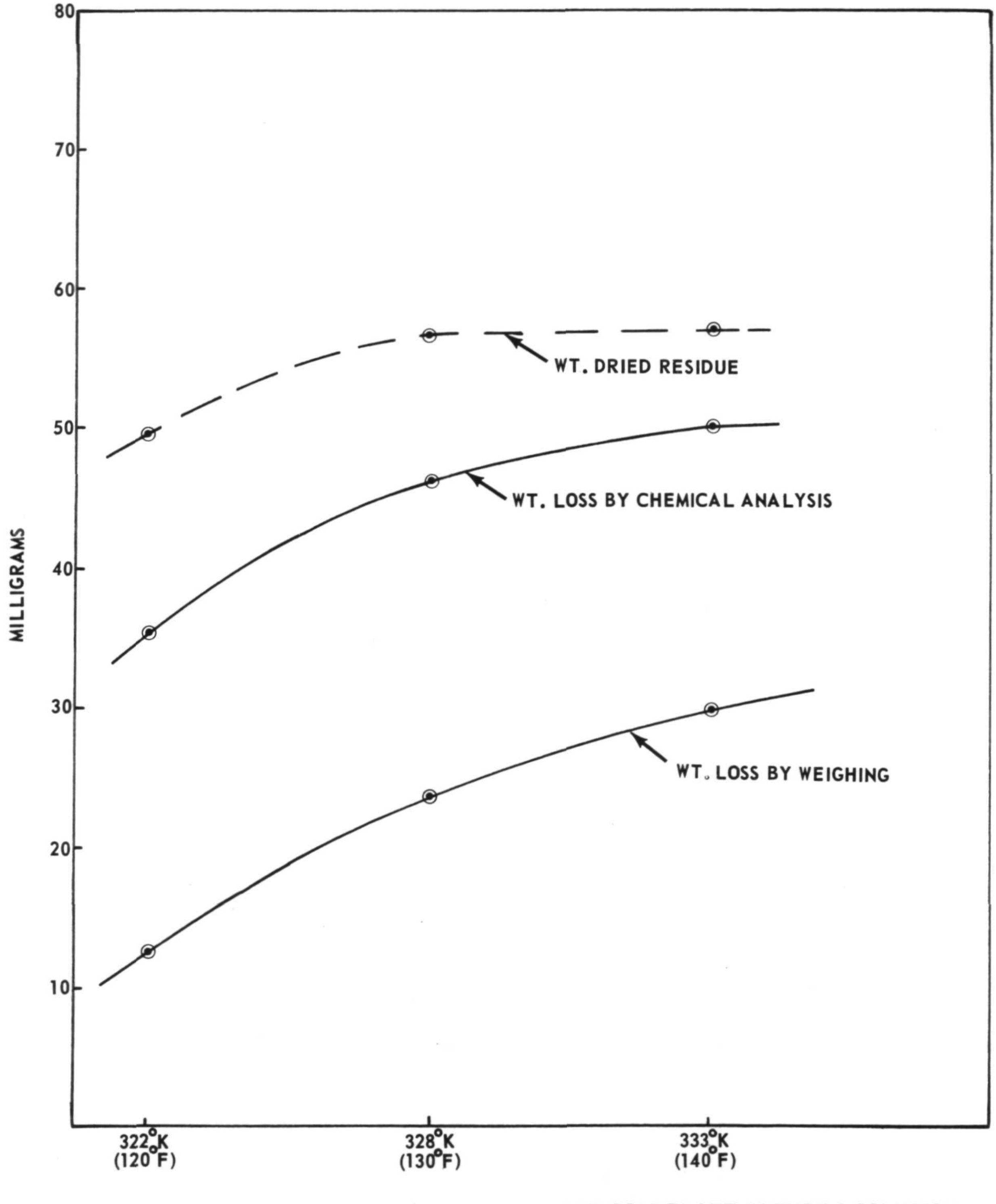


FIGURE 11 EFFECT OF EXPOSING SIMULATED EREP COLDPLATE IN TYPE 1 SOLUTION FOR 4 HOURS AT VARIOUS TEMPERATURES

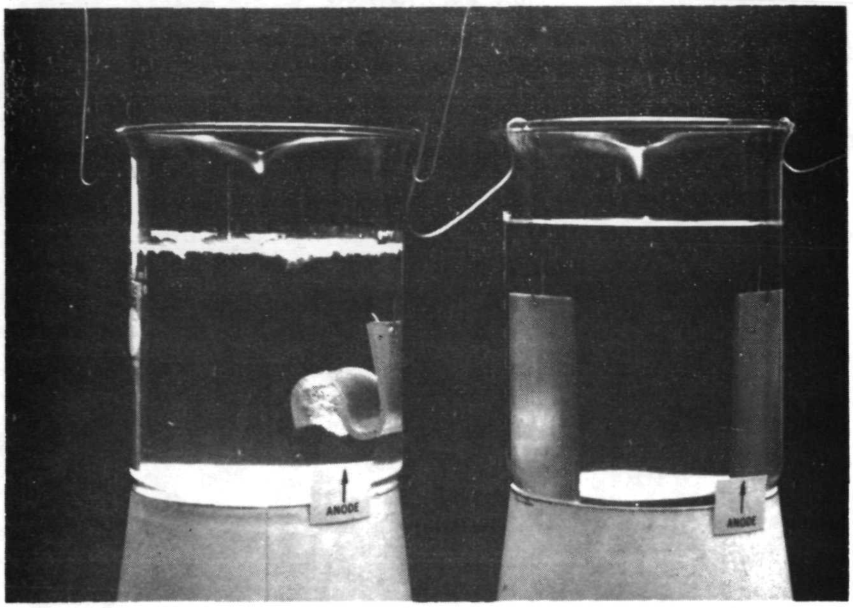


FIGURE 12 - EFFECT OF ELECTRODE LOCATION AND CONFIGURATION ON THE FORMATION OF RESIDUE BY ELECTROLYSIS

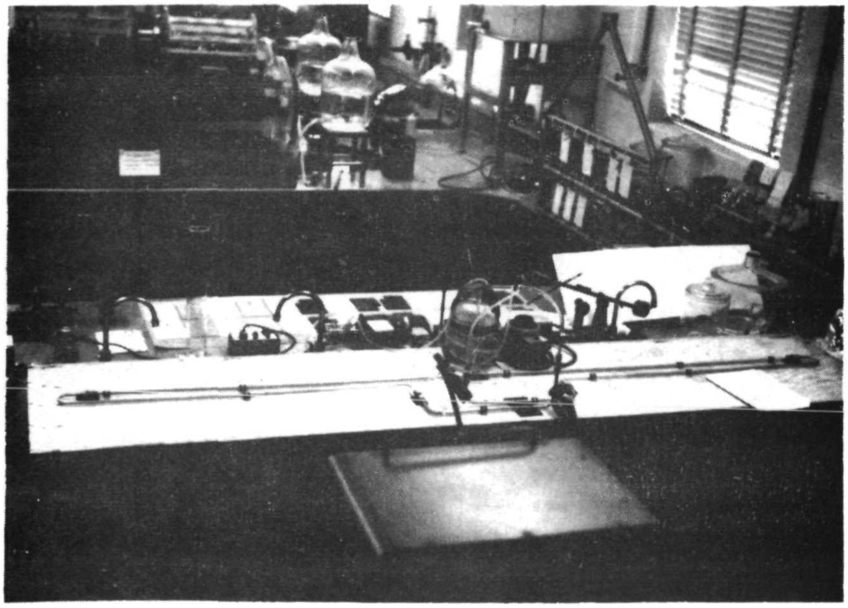


FIGURE 13 - SIMULATED TAPE RECORDER COLDPLATE

APPENDIX I

ATM C&D PANEL & EREP COOLING SYSTEM MATERIAL

Suit And EREP Cooling System

MDA - Part No.	Title	Non-Metals	Metals
82000004320	Tube Assembly		304 CRES
8420	" "		304 CRES
9320	" "		304 CRES
84000220140	" "		304 CRES
			304 CRES
220289	Orifice Union		347 CRES
220600	Tube Assembly	Fluovel/Viton	6061 Al
		Viton	718 Al
			304 CRES
PD32000048	Tube Assembly		321 CRES
			347 CRES
PD4700181	Valve Body		6061 Al
F1024	Gamah Fittings		321 CRES
F1034	" "		321 CRES
	" Seal		6061 Al
ST47D52	Valve Body & Plug	Viton	303 CRES
ST32D11	Tube Assembly		321 CRES
			347 CRES
ST55D22	O-Ring	Fluovel/Viton	
ST55D37	"	" "	6061 Al
ST57D58	Elbow Swivel Fitting		316 CRES
59026D015	O-Ring	Fluovel/Viton	
55F3N	Insert		Nickel
852315	Selector Block	Teflon	
MC237-C6	Adapter Fitting		304 CRES
MS24389	Tee		304 CRES
MS24392	Union		304 CRES
MS24393	Bulkhead Fitting		304 CRES
MS24394	" Elbow		304 CRES
MS24396	Elbow		304 CRES
MS24399	Reducer		304 CRES
MS24402	Tee		CRES

APPENDIX I CONTINUED

Airlock Module Cooling System

Part No.	Title	Non-Metals	Metals
52-83700-1205	Heat Exchanger		347 CRES Nickel
52-83700-1207	Heat Exchanger, Reqm		347 CRES Nickel
52-83700-1211	Water Tank	Epoxy-Polyurethane (EMS 369)	302 CRES
		Epoxy-Polyurethane Adhesive (EMS 363)	304 CRES
		Viton A	
52-83708-127	Quick Disconnect	Buna-N N-398	316 CRES
-129	" "	" "	316 CRES
-131	" "	" "	316 CRES
-151	" "	" "	316 CRES
52-88705-495 -719	Differential Pressure Transducer		302 CRES
61B830011	Water Relief	Parco 1235-70 Silicone	302 CRES
		Teflon	303 CRES
61C830060	Water Check Valve	Ethylene Propylene Rubber	303 CRES
61C830066	Water Filter Assembly	Viton A	303 CRES Chrome Plated
61C830068	Flexible Hose		321 CRES
61C830069	Water Pump		Chrome Plated
		Buna-N	302 CRES
			302 CRES
			304 CRES
			410 CRES
			416 CRES
			420 CRES
			416 CRES
			420 CRES
			17-4PH
			911 Al
			Gold
			Silver Solder
			Colmonoy #6
			Colmonoy #70

APPENDIX II

ANALYSIS OF CONTAMINATED FILTER NO. 24

1. Wet Residue Dried over desiccant - 29% Loss

2. Wet Residue Dried in oven at 110°C. - 49.5% Loss

3. Emission Spectrographic Analysis of oven dried residue:

   Major Elements - Al, K, P
   Minor Trace Elements - Mg, B, Ni, Fe, Cu, Zn

4. Atomic Absorption Analysis of oven dried residue:

   Percent By Weight

K - 20.8	Ni - 0.076	Zn - 0.014
Al - 14.4	Fe - 0.028	Na - 0.113
Mg - 0.034	Cu - 0.019	B - 1.0

5. Water Solubility of oven dried residue:

   Insolubles - 76.6 %
   Solubles - 23.4%

6. Atomic Absorption Analysis (water insolubles dried):

   K 13% by weight
   Al 17% by weight

7. Phosphates Analyses:

Original oven dried residue	43.5 % PO_4 by wt.
Water insolubles oven dried	52.8% PO_4

8. Infrared Analysis:

   Spectra of oven dried residue and water insolubles were very similar and contained strong bands for P-O, but neither spectrum would give positive identification (possibly due to mixtures). Two characteristic bands for K_2HPO_4 (coolant additive) did not appear in the unknown spectra.

9. X-Ray Diffraction Analysis:

   Best interpretation of pattern received indicates the presence of $K_3H_3(PO_4)_2 \cdot 2H_2O$. Although it is known that an aluminum compound is present, x-ray diffraction did not verify the presence of aluminum metal, aluminum hydroxide, aluminum oxide, or aluminum phosphate.

APPENDIX III

TYPE 1 SOLUTION SAMPLE DIAGNOSTIC PLAN

1. Determine delta pH of samples (include control) refer to H ion.
2. Use potentiostat at 290°K (70°F)
3. Compare turbidity - photograph (Nessler turbidity meter or equivalent).
4. Filter with .45 millipore filter, dry and weigh.
   - a. Control solution, filter, dry, weigh.
5. Analyze filtrate (liquid).
   - a. Set aside 50% - chill to 278°K (40°F) for 7 days - filter for precipitate and weigh.
   - b. Analyze (50%), use atomic absorption, look for Al, K, PO_4.
6. Analyze precipitant for 4 above, using X-ray fluorescence for Al, K, P and compare with Parker's photo as reference.

ATM/EREP COOLANT LOOP FILTER ANALYSIS

1. Photograph element after removal from housing.
2. Dry element in desiccator weigh (con't to const. wt.).
3. Mechanically remove contaminant (tapping/shaking) - weigh.
4. Analyze as procedure spec in filtrate analysis (X-ray fluorescence).
5. Wash element with 15% HCl solution.
6. Dry element - desiccant - weigh (to constant weight).
7. Compare 2 above with 6 above.

REPORT OF FINDINGS

1. Is there indication of an anomolous reaction?
2. If so, what is predicted effort?
   - a. How much contaminant will be generated in 75 hours 289°K (60°F)?
   - b. How much contaminant attributed to extraneous current flow (from tape recorder as measured)?
   - c. Is there problem of filter loading with residue, if so how long would it take?
   - d. Is there problem of filter leaking (eat through) ? If so, in what time frame?

APPENDIX III CONTINUED

ATM/EREP COOLANT LOOP

CONTAMINATION MEETING 4/11/73

ATTENDANCE LIST

1.	R. R. Orrell	MMA/Denver	
2.	A. L. Madyda	MSFC	SL-AL/MDA
3.	M. Piccone	MMA/Denver	
4.	J. R. Nunnelley	MSFC	S&E-ASTN-MC
5.	John W. Barnes	MSFC	S&E-ASTN-MC
6.	Sydney Shrage	MDAC-E	232-7351
7.	Glenn Salisbury	MDAC-E	232-6018
8.	C. Cataldo	MSFC	S&E-ASTN-MX
9.	Ralph Higgins	MSFC	S&E-ASTN-MMC
10.	Eli E. Nelson	MSFC	S&E-ASTN-MMC
11.	Gleason Williamson	MSFC	S&E-ASTN-MMC
12.	James E. Curry	MSFC	S&E-ASTN-MN
13.	John Parker	Ames Research Center	
14.	J. E. Kingsbury	MSFC	S&E-ASTN-DIR
15.	F. Uptagrafft	MSFC	S&E-ASTN-MX

APPENDIX IV

Analyses of Coolant Samples and Residues Associated With ATM C&D Cooling Loop Test Run While Using Filter Number 30

Coolant sample No. 1. (228 cc) - Sampled from S/N 7 can on 4-9-73 and represents coolant that was used in system during run using filter #30.

pH - 9.3

Turbidity - Compares favorably with good water that had been filtered through 0.45 filter.

Filtration of 100 cc through a 0.45 millipore pad without follow-up washing yielded an increase weight of 0.00537 grams to the millipore pad. This weight will be used for control and comparison purposes when filtering the other coolant samples associated with Filter No. 30.

Analysis of Filtrate:

Al - 0.5 ppm by wt.
K - 0.88% by wt.
K_2HPO_4, based on PO_4 - 2.0% by wt.

Coolant Sample No. 2 (107 cc) - Sampled from cooling loop system immediately after servicing system.

pH - 9.2

Turbidity - Similar to sample No. 1.

Filtration residue weight of 100 cc - No appreciable differences than sample No. 1

Analysis of Filtrate:

Al - 0.5ppm by wt.
K - 0.89% by wt.
K_2HPO_4, base on PO_4 2.0% wt.

Analyses of coolant samples and residues associated with ATM C&D cooling loop test run while using filter number 30.

Coolant Sample No. 3 (131 cc) - Sampled from system. Represents five hours of EREP cold plate circulation.

pH - 9.2

Turbidity - Similar to sample No. 1

Filtration residue weight of 100 cc - No appreciable differences than sample No. 1.

Analysis of Filtrate:

Al - 1.4 ppm by wt.
K - 0.86% by wt.
K_2HPO_4, based on PO_4 - 2.0% by wt.

Coolant Sample No. 4 (60 cc) - Sampled from system just prior to removal of filter assembly. Represents sixteen hours of EREP cold plate circulation.

pH - 9.2

Tubidity - Similar to sample No. 1.

Filtration residue weight of 60 cc - weight of millipore pad increased by 0.00391 gm. This is only slightly more than a proportional weight of sample No. 1 (approx. 0.0032 gm. equal to 60 cc of No. 2).

Analysis of Filtrate:

Al - 1.5 ppm by wt.
K - 0.87% by wt.
K_2HPO_4, based on PO_4 - 2.0% by wt.

Coolant Sample No. 5 (35 cc) - Removed from filter assembly after assembly was received by MDAC at St. Louis.

pH - 9.2

Turbidity - Quite turbid based on method used for measurement (suspended solids were visible).

Filtration residue weight of the 35 cc - The 35 cc yielded an increase weight of 0.00694 gm to the millipore pad. If a proportional weight of 35 cc of Sample No. 1 (0.0017 gm) was substracted from sample No. 5, it could be said that sample No. 5 yielded 0.0052 gms. of true residue.

Analysis of Filtrate:

Al - 2.3 ppm by wt.
K - 0.85% by wt.
K_2HPO_4, based on PO_4 - 2.0% by wt.

Analysis of residue from Sample No. 5.

The Scanning Electron Microscope showed the presence of Al, K, and P with K being present in greater concentration than Al and P.

Filter Assembly No. 30

Filter Element:

The element yielded (shaking, brushing, etc, but no rough scraping) 0.00055 grams of residue. The residue which consisted of white particles was collected on a blue millipore pad and weighed. An analysis of the residue using the Scanning Electron Microscope showed the presence of Al, K, P with K being present in a greater concentration than Al and P.

APPENDIX IV CONTINUED

Filter Housing:

The housing yielded 0.0032 grams of white particles which were removed similar to those particles taken from the element.

Elemental Analysis of the housing residue:

Al - 12.7% by wt.
K - 18.0% by wt.
PO_4 - 40.8% by wt. (based on P)

APPROVAL

ATM C&D PANEL/EREP COOLING SYSTEM CONTAMINATION PROBLEM

By
James G. Williamson

The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

This document has also been reviewed and approved for technical accuracy.

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