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TECHNICAL MEMORANDUM

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TITLE: EVALUATION OF METHODS TO REMOVE DECOMPOSED MIX-4P3E
FROM 9Cr-1Mo STEEL

ABSTRACT

Migration of mix-4P3E, the SNAP-8 lube/coolant fluid, into the mercury system of the PCS through the space seals, may result in deconditioning of the boiler. If deconditioning occurs, the decomposed residue of the L/C fluid should be removed from the boiler tube surface prior to continuation of tests. This report covers evaluation of selected candidate procedures for removal of thermally decomposed mix-4P3E.

Oxidation of the residue by 1300°F air exposure, followed by pickling, successfully removed surface adherent residue. The procedure consisted of twice performing the following multi-step cycle: (1) air exposure at 1300°F for ½ hour; (2) three-step clean with Turco Products, Inc., fluids Turco 4931, then Turco 4338C and finally Turco 4931.

A yellowish surface discoloration of the specimen resulted from cleaning. Further evaluation is required to determine if this surface condition represents an unacceptable mercury non-wetting condition.

APPROVED:

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INTRODUCTION

The SNAP-8 PCS utilizes a polyphenyl ether (mix-4P3E) fluid for lubricating the bearings of various rotating components and cooling electrical components. The interface components between the Hg and L/C loops, the turbine alternator assembly and the Hg pump, contain separate seals to space for each loop. These space seals, and the intervening space vacuum, should minimize the cross-diffusion of the lubricant into the mercury loop. Reference (1) indicates that a small amount of lubricant will enter the mercury loop even under ideal conditions. Reference (2) describes an investigation wherein mix-4P3E lubricant was found in the RPL-2 mercury loop during a series of test runs. Any lubricant which does enter the mercury loop through the Hg PMA or TAA interface space seal system may be carried to the boiler. This presents a potential problem of boiler deconditioning because of a heat-transfer inhibiting oil film on the boiler tube wall and thermal degradation of the lubricant. A concomitant result of thermal degradation is the deposition of decomposition products on the boiler tube wall.

Hg capsule wettability tests at LeRC indicate that a film of organic fluid as thin as 500 Angstroms may prevent mercury wetting. A program was initiated to determine a suitable method for removing decomposed mix-4P3E from the wall of a 9Cr-1Mo mercury-containment tube if such contamination occurs during a SNAP-8 system test.

VYCOR CAPSULE TESTS

Mix-4P3E was decomposed in Vycor glass capsules to evaluate the capability of various standard SNAP-8 cleaning solutions in removing the residue from the capsule wall.

Duplicate Vycor capsules were prepared from 7 mm diameter tubing by hermetically sealing approximately 1/4 cubic centimeter of mix-4P3E fluid in the tubes. The tubes were heated at approximately 1150°F for 18 hours. After recooling to 75°F, the capsules were broken and surfaces containing the decomposed residue, as well as separated residue samples, were subjected to the following cleaning solutions:

TABLE I
CLEANING OF VYCOR CAPSULES AND SURFACE RESIDUE

<u>Fluid</u>	<u>Sample</u>	<u>Remarks</u>
Type I MIL-C-14460	2 Vycor Sections	After approximately 1 minute exposure to the fluid at approximately 200-210°F, the residue broke into small particles and tube cleaned.
Type I MIL-C-14460	Charred Residue	No visual dissolution after 2 hours.
Oakite-90	2 Vycor Sections	Approximately 20% of residue removed after 15 minutes in solution at approximately 200-210°F.
Oakite-90	Charred Residue	No visual dissolution after 2 hours.
Trichloroethylene	Charred Residue	No visual dissolution after 2 hours.
Warm Alcoholic 0.1 Norm. KOH	Charred Residue	No visual dissolution after 2 hours.

Results of these tests indicated the residue might be removed from a surface by exposure to a caustic solution of Type I per MIL-C-14460.

PRELIMINARY 9Cr-1Mo TAB SPECIMEN TESTS

To further evaluate the MIL-C-14460 under conditions more closely approximating that of a contaminated boiler tube, tests were run using 9Cr-1Mo steel flat specimens approximately 1" x 1 1/2" x 1/8". These specimens were heated with a small quantity of mix-4P3E in a hermetically-sealed tube for 18 hours at 1150°F. The sealed tube was similar in configuration to Figure 1.

The specimens were first cleaned using various solutions with the following results.

TABLE II

CLEANING OF 9Cr-1Mo STEEL TUBE SPECIMENS

<u>Solution</u>	<u>Conditions</u>	<u>Remarks</u>
Type I MIL-C-14460	Temperature 180-200°F	No visual change after 2 hrs exposure.
Methylene chloride followed by Type I MIL-C-14460	Methylene chloride dip at room temperature; caustic at 180-200°F.	Methylene chloride changed color slightly; caustic did not change additionally after 2 hrs exposure.
6% HNO ₃ (by wt) followed by Type I MIL-C-14460	Same specimen as above after original exposure	15 minutes in acid did not change the appearance, nor did one additional hour in caustic solution.
Turco Jetisoil-NP	Room temperature	No visual change after 2 hrs exposure.
Turco 4931 + Turco 4338C + Turco 4931	Multi-step procedure discussed below.	Procedure removed approximately 90-95% of residue from surface of specimen.

The Turco Products Company solutions were able to remove most of the residue from the 9Cr-1Mo specimens. Further improvement in the amount of residue removed was produced by subsequently heating the specimens to approximately 1200°F in an air atmosphere. This procedure was evaluated as a result of a discussion with Dr. C. L. Mahoney of Shell Development Company, Emeryville, California. Shell utilizes three different techniques to eliminate mix-4P3E degradation products from their process equipment: a) heating in air to a dull red heat; b) "moderate" concentration of hydrofluoric acid; and c) a high concentration (20% hydrogen peroxide) in ammonium hydroxide (approximately 30% NH₃). Only the heating in air is amenable to SNAP-8 test system operation; thus, it was tried. The scale resulting from this hot air exposure was readily removed by acid cleaning, the procedure in Reference (3). The full procedure was as follows:

- Step 1 Immerse specimen in Turco 4931
 - a. Concentration: 12 oz/gal
 - b. Temperature: 170-180°F
 - c. Time: Approximately 1 hour
- Step 2 Water rinse
- Step 3 Immerse specimen in Turco 4338C
 - a. Concentration: 2 lbs/gal
 - b. Temperature: 190-200°F
 - c. Time: Approximately 1 hour

- Step 4 Water rinse
- Step 5 Immerse specimen in Turco 4931
- a. Concentration: 12 oz/gal
- b. Temperature: 170-180°F
- c. Time: Approximately 30 minutes
- Step 6 Water rinse
- Step 7 Expose specimen to air atmosphere at 1200°F
 for ½ hour
- Step 8 Clean per AGC 10226, Type II.

The acid utilized in the above procedure produces pitting of 1010 low alloy steel; therefore, it cannot be considered universally applicable for cleaning boilers or systems. Any components which contain this material, for example, a 1010 steel turbulator in the boiler, must be cleaned by an alternate method. The investigation was continued to find one.

FINAL 9Cr-1Mo TAB SPECIMEN TESTS

A final series of tests was conducted to establish the optimum cleaning procedure for removing mix-4P3E residue from 9Cr-1Mo steel surfaces. Table III lists the procedures evaluated and specimen distribution.

Pieces of 9Cr-1Mo steel, 9" long x 1" wide were first pickled using the procedure described as Type II in Reference 3. Two specimens were placed in each of three 9Cr-1Mo steel capsules open at one end, see Figure 1. Approximately 5 cc of mix-4P3E was added, by dropper, to the capsule. The liquid was dropped onto the surfaces of the specimens. An end-closure cap containing a small diameter through tube was welded on. A vacuum of 10^{-2} to 10^{-3} mm Hg was produced in the capsule using the end-cap tube. The small diameter tube was then pinched closed and Heliarc welded to seal the capsule.

The three capsules were heated for 24 hours at 1150°F. After cool-down, the specimens, each of which now contained a surface contaminated with decomposed mix-4P3E residue, were removed from the capsules and were sheared into tab specimens 1½ inches long. Specimens unexposed to the mix-4P3E were also prepared.

The specimens were cleaned as described in Table III. Those which were exposed to an elevated temperature, 1100 to 1300°F, air-atmosphere as part of the cleaning procedure were placed on a coarse, stainless steel screen in the oven to permit free circulation of air on all sides. The cleaning solution described as "Turco Products, Inc. Recommendation" is identical to that used during the previously mentioned Preliminary 9Cr-1Mo Tub Specimen Tests. The other cleaning solutions and associated procedures are described in Appendix 1. As is noted in Table III, both single and double-cycle cleaning procedures were used.

DISCUSSION OF RESULTS

Figure 2 shows the specimens at the various stages of cleaning. It was observed that the Turco technique was capable of restoring the specimens close to their original appearance if the residue was first burned off by elevated temperature air exposure. Without this exposure, approximately 5 to 10% of the decomposed mix-4P3E residue remains. The double-cycle cleaning procedure (each cycle consists of heating to 1300°F and solution cleaning) resulted in somewhat better cleaning than the single-cycle treatment at the lower temperatures. A yellowish surface discoloration was noted on all specimens cleaned using the Turco solutions.

NaK at 1100 or 1300°F did a moderately good job of cleanup if the decomposed mix-4P3E residue was first subjected to elevated temperature air exposure. In no instance was the result as good as that achieved with the Turco solution. There was some dark discoloration remaining on the specimens. Some scale was loosened and could be peeled off. The double-cycle exposure to 1300°F air resulted in a lustrous external layer that could be peeled off. Underneath the lustrous layer the specimen was bluish-black. The use of NaK to clean decomposed mix-4P3E from the internal tube surfaces of the boiler should not be considered, except as a last resort. This is because of very special handling requirements due to its reactivity and the problems associated with the removal of the residual NaK from the system after exposure. The exposure to the solutions referred to in AGC-10319/6 resulted in very poor removal of the contamination.

CONCLUSIONS

1. The optimum cleaning technique of those evaluated for removing decomposed mix-4P3E from 9Cr-1Mo steel surfaces consisted of heating the specimen in air at 1300°F for one-half hour, followed by immersion in Turco 4931 then Turco 4338C solutions and reimmersion in Turco 4931.
2. Although NaK at 1300°F did a good job in removing residual decomposition products of the mix-4P3E, it would be very expensive to utilize because of the special handling and thus can only be recommended as a last resort.
3. The fluids of Specification AGC-10319/6 appear to have very little effect in removing the decomposed mix-4P3E as deposited, or the oxidized product resulting from elevated temperature exposure.
4. The Turco solutions leave a surface discoloration film after cleaning. The effect of this film on mercury wetting of the tube wall is unknown.

RECOMMENDATIONS

1. The procedure involving heating in 1300°F air, followed by Turco-cleaning, should be used to remove decomposed mix-4P3E from SNAP-8 components. A technique to suit the specific component to be cleaned must be established prior to cleaning.
2. NaK should not be considered for cleaning because of potential handling and cleaning problems.
3. Investigate the effect on wettability by mercury of the discoloration film left by the Turco solutions.
4. Perform additional tests to further optimize the cleaning technique using the Turco solutions aiming toward reduction of the process steps. Pickling prior to the elevated temperature oxidation and solution cleaning afterwards may eliminate one of the elevated temperature steps.

REFERENCES

1. Memorandum to E. Eber from J. N. Hodgson, 350:64-701, dated 5 November 1964, "Vacuum Requirements for SNAP-8 Seals-to-Space."
2. Memorandum to B. S. Pilling from H. Dercw, 4842-65-0052, dated 15 March 1965, "Analysis of Surface Residue from RPL-2 Dump Tank."
3. AGC Specification, AGC-10226, "Steel, Cleaning and Dehydration, Procedures for," Type 1I - Removal of Scale from 9Cr-1Mo Steel.

TABLE III

SPECIMEN DISTRIBUTION FOR EVALUATION OF TECHNIQUES
FOR REMOVAL OF DECOMPOSED MIX-4P3E

	Not Burned Off	1 hr @ 1100°F	½ hr @ 1200°F	½ hr @ 1300°F	½ hr @ 1300°F; clean ½ hr @ 1300°F; clean
A. Pickle per AGC-10226 Type II, then expose to mix-4P3E in an evacuated 304 SS container for 24 hrs at 1150°F to produce decomposed residue. Clean as follows:					
1. Control (no cleaning)	1	1	1	1	
2. AGC-10319/6	1	1	1	1	1
3. Turco Products, Inc. recommendation*	1	1	1	1	1
4. NaK: at 1000°F	1	1	1	1	1
at 1300°F	1	1	1	1	1
B. Pickle per AGC-10226 Type II only (unexposed to decomposed mix-4P3E) Clean as follows:					
1. Control (no cleaning)	1	1	1	1	
2. AGC-10319/6		1	1	1	1
3. Turco Products, Inc. recommendation*		1	1	1	1
4. NaK at 1300°F		1	1	1	1

* Turco 4931 (alkaline permanganate followed by Turco 4338C (bisulfate base solution), followed by Turco 4931.

METAL CAPSULE FOR DECOMPOSITION STUDIES OF MIX-4P3E

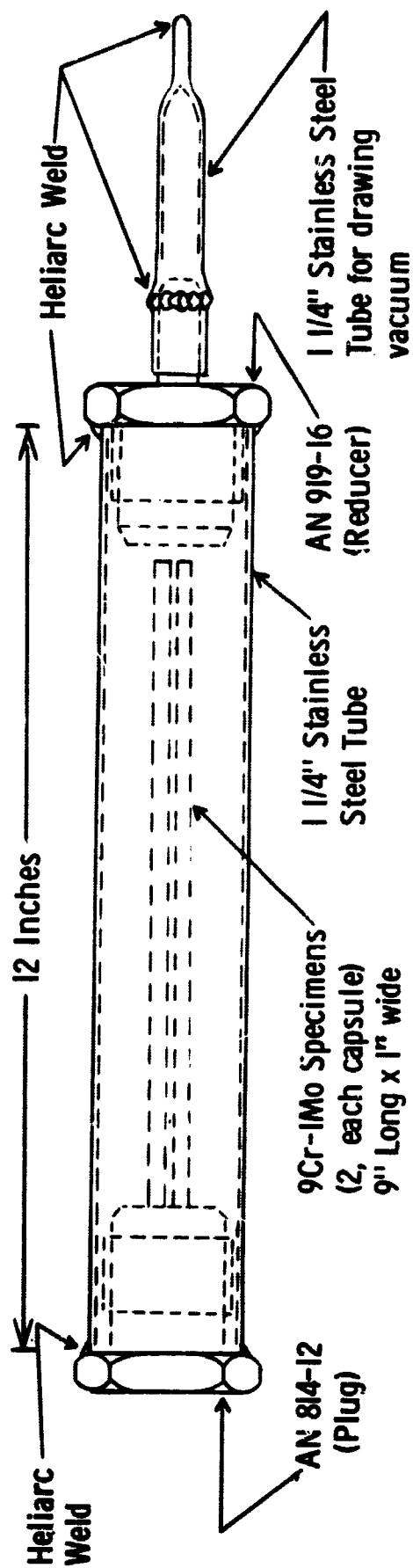


Figure 1

Not

A. Pickle per AGC 10226, Type V,
then expose to mix-4P3E in an
evacuated, sealed 304 SS con-
tainer for 24 hrs at 1150°F
to produce residue. Then clean
with specified methods.

1. Control -
no cleaning
2. AGC 10319/6
3. Turco Products,
Inc. recommenda-
tion*
4. NaK: 1000°F
5. NaK: 1300°F

B. Pickle per AGC 10226, Type II,
only (unexposed to decomposed
mix-4P3E). Then clean with
specified methods.

1. Control -
no cleaning
2. AGC-10319/6
3. Turco Products,
Inc. recommenda-
tion*
4. NaK: 1300°F

- * Turco 4931 (alkaline permanganate) followed by Turco 4331
(bisulfate base solution) followed by Turco 4931.

FIGURE 2

EN DISTRIBUTION FOR EVALUATION OF TECHNIQUES FOR
L OF DECOMPOSED MIX-4P3E POLYPHENYL ETHER

Burned			
Burned Off	One Hour at 1100°F	One-Half Hour at 1200°F	One-Half Hour at 1300°F
			
			
			
			
			
			
			
			
			

Off

ur ½ Hour at 1300°F; ½ Hour at 1300°F;
Clean; ½ Hour at Clean; ½ Hour at
1300°F; Clean 1300°F; Clean

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