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DATE: March 20, 1956

TO: C. B. Graham

FROM: P. P. Holz

SUBJECT: HRT Heat Exchangers - Cleaning Runs Performed at Y-12

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SUMMARY

The HRT blanket (No. 8531) and fuel heat exchangers (No. 8532) were subjected to thorough oxygenated water flushing runs to eliminate small amounts of carbon deposits and impurities from the tube sides which had accidentally entered during thermal cycling operations at the Foster Wheeler plant. The oxygenated water flushings proved effective on both heat exchangers to assure HRT cleanliness qualifications.

Utilizing the flushing loop setup, additional tests were run on the fuel heat exchanger. Uranyl sulphate solution (approx. 10 gm/l) was circulated to explore a minute leak, which was first noted during the final helium leak test of the primary side at Foster Wheeler, proved effective in reducing the leak from 0.35 - 0.50 cc/day to 0.006 cc/day. It is assumed that the leak, which was probably located in the tube joint region, was essentially sealed by corrosion products. Further, condensate sampling of the shell side during the "soup" run, and analyzing the condensate for uranium, verify this conclusion.

Other tests conducted in connection with the flushing loop setup included "Pressure Drop Calibrations," "Heat Loss Data Tests," and a "Helium Leak Test." Specific summary reports are listed in the bibliography.

BACKGROUND HISTORY INDICATING NECESSITY FOR HEAT EXCHANGER CLEANING RUNS PERFORMED AT Y-12

Visual inspection of the heat exchangers at Foster Wheeler Corporation, following their Dowtherm "A" loop thermal cycle runs (approx. 100°C to 300°C), indicated fouling of the primary sides of both exchangers. This formation, which was black in appearance, consisted of approximately 25 per cent iron oxide and 75 per cent carbon. Even though utmost care had been taken to degrease and clean all component parts, to permit only heliarc welding, etc., by-products of the Dowtherm cycling operation deposited impurities into the tube sides of the exchangers. The final diagnosis rendered indicated that the Dowtherm partially decomposed during the thermal cycling as localized temperatures within the gas heating furnace exceeded 300°C. Both the furnace and the loop were made of carbon steel; hence, loop flow carried the Dowtherm deposits and some iron oxide throughout the system, including the heat exchanger tubes.

To remove these deposits from the tube sides of the exchangers at the Foster Wheeler plant, many tests of various flushing mixes were tried. The fuel exchanger was subjected to acid cleaning employing trichromic acid, followed by caustic, and subsequently, by nitric acid in respective runs at about 200°F. During the last half hour of this cleaning run it was noted that the plating hose and rubber pump couplings, etc., started to decompose, caused by the hot acid. The dissolved out decomposition products then deposited within the tube side of the exchanger. Subsequent cleaning with hot xylene decomposed an estimated to be ninety per cent of the previous sticky, gluey, rubbery black deposit. In effect, at this point, the fuel exchanger tube side contained greater carbon scaling than prior to the acid cleaning attempt. With the heat exchanger head removed, each individual tube was next rotary brushed while being flushed with detergent solutions. Demineralized water flushes followed felt pellets blown through the tubes under approximately 100 psi air pressure. Repeat operations continued until the outcoming pellets indicated very little discoloration. To de-soap completely,

additional 135⁰F demineralized water flushing continued until a pH paper check indicated like incoming and outgoing pH readings. A final degreasing operation then concluded the Foster Wheeler cleaning runs; all the water was drained out from the tube side which was then purged with nitrogen, and thereafter, completely filled with acetone. After an hour's hold time, the acetone was removed and the exchanger subjected to a final nitrogen purge.

In essence, the blanket exchanger cleaning was similar to, but not as extensive as, that of the above described fuel exchanger. As the hot acid cleaning was not attempted on the blanket exchanger, this exchanger resulted in being the cleaner of the two. A de-soaping operation involving felt pellets, demineralized water and acetone as described above completed Foster Wheeler's cleaning attempt on this exchanger.

On both exchangers, with the heads removed, all accessible stainless parts were washed, wire brushed, and acetone cleaned.

During the above course of cleaning operation, it became obvious that further proper cleaning should be performed in a stainless steel, high temperature, high pressure loop. Foster Wheeler had no such loop available. To save time and money, it was therefore decided, prior to acceptance of the dirty units from Foster Wheeler, to subject both heat exchangers to a cleaning operation in a loop which would be set up in the REED Division at Y-12. It was planned to try oxygenated water on the blanket exchanger, the first one to be received, with uranyl sulphate flushings to follow only, if necessary⁶. The fuel exchanger, however, was scheduled for both oxygenated water and "soup" flushings in order to also explore the probable high pressure leakage noted during earlier helium leak testing at the vendor's plant^{1,5}.

SETUP

The 400-B Westinghouse pump loop was modified to accommodate the heat exchangers. A suitable steel beam cradle was fabricated to support the exchangers and at the same time properly distribute the loadings on the second floor building beams. New 2" schedule 80, type 347 stainless piping served as transitory

connections between the existing loop and the exchangers. This piping was designed to accommodate the total stresses of the new loop, and to permit the installation of an additional 15 kilowatt heater capacity. In general, the existing loop instrumentation required a minimum of alteration, although a second dp cell was added to record pressure drops through the exchanger. Both dp cells (loop flow and heat exchanger pressure drop) were also provided with recording meters and calibrated gages. The steam side of the heat exchangers received supply from the existing 250 lb. building main. A steam inlet station included a regulating valve, needle valve combination utilized in the initial slow heating of the shell, and a line sized globe throttling valve bypass alternate used in the final heating to obtain maximum available steam line pressure with its corresponding higher temperature. Steam was injected to the exchanger through the 2" - 900 lb. top flange. The condensate discharge connected to a 1" heat exchanger condensate outlet and included a trapping station ahead of the vented and cooled flash tank to which the building drains were then connected. A safety relief valve was also employed. It connected to a 4" - 900 lb. heat exchanger flange, and through a vent line to the atmosphere outside of the building. All piping was insulated with double layers of 1" magnesia. Aluminum foil insulated the exchanger heads. A felt blanket was used as cover over the top and sides of the shell to assist in the initial heat up, and to reduce heat losses. See Figure A for setup.

PROCEDURE OF FLUSHING OPERATIONS

The blanket exchanger flushing run commenced the morning of January 18, 1956, and continued for approximately 50 hours. Initially, following a 2000 lb. hydrostatic pressure test of the loop, all the air was driven out from the shell side by entering low pressure steam and releasing it through the relief valve bypass vent line after short holdup periods. Thereafter, all loop heaters were turned on (approx. 28.5 KW) and the steam pressure (flow) to the shell adjusted so as to raise the primary and secondary side temperatures of the heat exchanger uniformly up to the maximum steam pressure available, 225 lb. (200°C). The electrical loop heaters were then further utilized to raise the loop temperature to 250°C, and the

pressurizer temperature to 255°C. The loop was charged with about 21 gallons of distilled water of high purity, and 75 psi. of oxygen. The starting water level of the system was to the bottom of the horizontal pressurizer of 7.3 gal. (cold water) capacity. At temperature conditions, the loop pressure developed was slightly in excess of 1000 lbs., and loop flow about 300 gpm.

Graph A, HRT Blanket Heat Exchanger, Water Flushing Data records the average curves obtained by plotting oxygen, carbon dioxide, chromium VI, chloride (-), pH and conductivity versus time for the duration of the blanket exchanger run. This flushing run was discontinued after about 50 hours of continuous operation as the prescribed test specifications⁶ were met. Carbon dioxide values rose rapidly during the initial three hours of the run, and continued a gradual rise over the next twenty-four hours. At this half point of the run, values levelled off and then actually tapered down gradually. pH readings all remained well above 3.5 (4.75 avg. at the conclusion of the run), while chloride (-) levelled off at about 2.5 ppm and Cr VI approached 6.5 ppm.

The fuel heat exchanger flushing was conducted in practically the same manner as the previously described blanket exchanger run. This run was started the afternoon of February 3, 1956, and continued for about 65 hours. After 40 hours, using a pulsafeeder pump setup, 3350 cc of cold distilled water was added to the system while simultaneously dumping 1550 cc. This water injection - dump operation was performed for two reasons: (1) To increase the parts per million concentration of oxygen in the system, accomplished by adding more than dumping, and by raising the overall loop pressure from 1000 lbs. to 1040 lbs., while maintaining loop and pressurizer operational temperatures of 250°C and 255°C, respectively, (2) To test out the injection and dump system preparatory to "soup" injection.

While proceeding with the flushing run, several checks were also taken of zero readings of the instrumentation used in connection with the Pressure Drop Calculations³. The pump was also run in reverse to obtain additional data points.

Chart E illustrates these calibrations.

Graph B, HRT Fuel Heat Exchanger, Water Flushing Data, represents the average curves obtained by plotting results from chemical analysis data of loop samples taken. While the slopes of the curves follow those obtained from the blanket exchanger run, it is obvious that greater concentrations of impurities were present in the high pressure side of this fuel exchanger. As much as 140 ppm CO_2 was noted compared to 50 ppm of the blanket unit. Cr VI levelled out near 32 ppm as compared to 6.5 ppm, Cl (-) levelled out at 2 ppm, compared to a similar levelling out at 2.5 ppm, and pH at 5, compared to 4.75. The tapering off of the curves, however, conclusively indicates that the exchanger cleaning was effective. A small increase of about 1 per cent in loop flow rate by the end of the 250°C loop temperature water flushing run (298 to 301 gpm) further substantiates this conclusion.

The pulsafeeder setup was once more utilized to inject "soup" into the flushing loop, while the dump system was employed to discharge from the system and permit room for the "soup" addition. A concentrated "soup" batch was prepared with 24 liters of "soup" containing 1.910 liters UO_2SO_4 and 115 cc H_2SO_4 . This mix resulted in approximately 10 gm/l uranyl soup with 50 per cent excess H_2SO_4 acid. Three hours were required for this injection. The loop temperature of 250°C and a flow of approximately 300 gpm were maintained during this period. The "soup" flushing circulation then continued for about 140 hours.

Graph C, HRT Fuel Heat Exchanger, "Soup" Flushing Data illustrates averaged plotted values of uranium, sulphate, chloride, oxygen, chrome, carbon dioxide, pH, nickel and iron analysis versus flushing run time. The following curves appear self explanatory: pH with a slight, but uniform rise from 1.66 to 1.77, U, and SO_4 with gradual, and uniform decreases of from 8.25 to 8.15 ppm and 5.1 to 4.9 ppm respectively. Also, the rise in Ni from 10 ppm to a levelled off approximately 150 ppm and the drop in Fe to less than 1 ppm following a rapid initial rise from 2 to 9 ppm, as well as the uniform average of 1.75 ppm of Cr VI preceded by an immediate fast rise all follow the expected trend. However, slight

irregularities in the CO_2 values near the mid point of the run, and a similar occurrence in the Cl (-) behavior near the three quarter run time can not be explained readily, unless these irregularities may be fully attributed to limitations of the accuracy in the chemical laboratory analysis. The average curves for CO_2 and Cl (-) are therefore shown as straight lines, with CO_2 uniform at 134 ppm and Cl (-) decreasing from about 4-1/2 to 2-1/2 ppm.

Chart D, Results of Condensate Sampling of the HRT Fuel Exchanger during the "soup" run does not follow any set pattern. A condensate shell side sample taken shortly after the completion of the "soup" injection, and one taken 24 hours thereafter both indicated the presence of uranium under the colormetric thio cyanate method analysis. A fluometric laboratory check of the initial (2 ppm U) sample, however did not confirm this presence, although a similar fluometric check of the second sample verified uranium presence (9 ppm colormetric analysis 7 ppm fluometric check analysis). Subsequent shell side condensate sampling did not reveal further significant uranium presence by either analytical methods.

To possibly recheck for minute uranium presence in the fuel exchangers shell side, following the helium leak test, low pressure steam was re-entered into the shell. It was hoped that such a steam rinse of the shell side might effectively pick up possible uranium remains from the tube sheet. Chemical checks of the condensation of this rinse steam, however, proved inconclusive. No uranium notations were had from colormetric analysis, however, one fluometric check noted a 1.6 ppm uranium presence in a sample taken from the very last pint of the condensation. Still, another lab's fluometric check would not confirm these results, reporting < 1 ppm on the same sample.

The inconsistencies in the various chemical analysis reports for the same samples indicate an inability to detect very minute amounts of uranium uniformly and accurately. Still, the verified presence of uranium in the shell side at the outset of the "soup" run, and a possible check on its presence from the tube sheet rinse point towards a possibility that a very minute crack or opening may have been present between the tube and shell side which eventually nearly plugged completely from corrosion products.

Condensate samples were also taken from the heat exchanger channel drain near the tube sheet, with no uranium detectable from any samples.

CONCLUSIONS AND RECOMMENDATIONS

Chemical analysis results indicate that the oxygenated distilled water flushings conducted at 250°C and about 1000 psi total pressure were sufficiently effective to permit both the blanket and the fuel heat exchanger to pass HRT cleanliness specifications.

It is probably that corrosion products from the "soup" flushing served to almost completely seal a small leak between the fuel heat exchanger tube and shell sides. There exists, however, a possibility that thermal cycling during HRT operation may tend to open this now minute leak of 0.006 cc/day². To safeguard, it is therefore recommended to continue to sample fuel exchanger's shell side condensate periodically during the initial HRT test operations and analyze the samples for possible uranium content. It might also be well to consider using a radioactive fissionable tracer in the exchanger's high pressure system to obtain more conclusive and exacting results from shell condensate analyses for in leakage from the primary side. An additional helium leak detection test is also recommended to be conducted just prior to the exchanger being placed into critical operation.

P. P. Holz

PPH: job

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PHOTO 25518

INSULATED HEAD

BLANKET HEAT EXCHANGER

FLASH TANK

400-cpm PUMP

Heat Exchanger Cleaning Installation.

HRT BLANKET HEAT EXCHANGER

LOOP T₂ 250°F

CHART "E"

LOOP & HEAT EXCHANGER CALIBRATIONS

H.R.T. FUEL HEAT EXCHANGER

LOOP TEMP.	PUMP DIRECTION	ΔP LOOP ORIFICE		ΔP HT. X. ORIFICE	
		FT. OF WATER	G.P.M.	FT. OF WATER	P.S.I.
15°C.	NORMAL	26.8	280	35.4	15.4
15°C.	REVERSE	4.3	112	7.46	3.24
250°C.	NORMAL	24.4 - 24.6	298 - 301	20.0	8.69

LOOP FLOW: $3\frac{1}{2}$ " N.P.S. FLANGE TAP ORIFICE
2.007" ϕ OPENING, SHARP EDGE

HT. X. DROP: HI SIDE CONN: PERMANENT TAP, ~ 27 " FROM
HT. X. CHANNEL

LOW SIDE CONN: HT. X. CHANNEL DRAIN

WESTINGHOUSE 400 A PUMP
OXYGENATED WATER, ~ 1000 ppm O_2

DPH 3/10/56

380 013

GRAPH "B"

CHEMICAL ANALYSIS DATA

WATER FLUSHING RUN 2/3/56 - 2/6/56

H.R.T. FUEL HEAT EXCHANGER

LOOP T = 250°C

380 014

GRAPH "C" CHEMICAL ANALYSIS DATA - URANIUM FLUSHING RUN 2/6/56 - 2/12/56

PPH 3/1/56

380 015

CHART "D"

ORNL-LR-Dwg. -12897

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H.R.T. FUEL HEAT EXCHANGER - SHELL CONDENSATE SAMPLE ANALYSES

DATE	TIME	COLORMETRIC URANIUM ppm	FLUOMETRIC URANIUM ppm	FLUOM. URANIUM CHECKS ppm	SO ₄ ppm	SO ₄ CHECKS ppm	Fe ppm	Fe CHECKS ppm	PH	PH CHECKS	CONDUCT. $\times 10^6$ mhos/cm	COND. $\times 10^6$ CHECKS mhos/cm
2-7-56	1300	2	<.2	-	3	-	<1	-	7.0	-	40	-
2-8-56	1230	9	7	-	<2	-	<1	-	7.1	-	45	-
2-9-56	0930	<2	.2	-	4	-	2	-	6.5	-	37	-
2-9-56	1400	<2	<.2	<1	<2	<1	1	1	6.2	6.2	10	-
2-10-56	0845	<2	.3-.4	<1	4	<1	153/53	12/49	6.1	5.9	6.7	8.0
2-10-56	1515	<2	.2	<1	4	<1	99	25	6.2	6.4	26	20
2-11-56	1030	<2	<1	<1	<2	<1	136	71	6.0	6.0	18	13
2-12-56	1000	<2	<1	<1	<2	<1	36	85	6.2	6.1	14	12
2-12-56	1530	<2	<1	<1	<2	<1	21	46	6.3	6.4	29	24
2-13-56	0900	<2	<.2	<1	-	-	32	25	8.2	-	43	-
2-13-56	1130	<2	<.1	<1	<2	<1	-	-	8.4	6.6	-	-
2-17-56	1000	<2	1.6	<1	<2	<1	21	-	7.7	6.5	30	-

380 016