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PILOT-PLANT STUDIES OF MERCURY-CATALYZED DISSOLVING OF ALUMINUM-JACKETED FUEL ELEMENTS

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For The Atomic Energy Commission

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PILOT-PLANT STUDIES OF MERCURY-CATALYZED
DISSOLVING OF ALUMINUM-JACKETED FUEL ELEMENTS

I. INTRODUCTION

In the present Hanford process, irradiated fuel slugs are dissolved in a two-step procedure: first, the aluminum jackets and bonding material are dissolved in sodium hydroxide - sodium nitrate solution, then the uranium and its products are dissolved in nitric acid.

With the aid of a mercury catalyst, aluminum may be dissolved in dilute nitric acid. A report by Bradford, Curtis, and Harmon (1) describes experience gained at other sites as well as bench-scale work done by the Process Chemistry group of the Chemical Development Unit in developing a technique for simultaneous dissolving of aluminum and uranium by nitric acid with a mercury catalyst. The bench-scale work indicated that catalytic dissolving is feasible and potentially offers the following advantages over the present two-step method:

Elimination of the Ammonium Nitrate Problem. When the jacket removal with sodium hydroxide - sodium nitrate solution (which step releases NH_3 gas) is proceeding in one plant dissolver simultaneously with uranium dissolution (which step releases oxides of nitrogen) in another dissolver, ammonium nitrate is formed in the plant main ventilation stack which discharges to the atmosphere. As a result, crystals of ammonium nitrate, severely contaminated with fission products, have on a few occasions been spread over a wide area surrounding the Redox Plant. This potential radioactivity hazard might be reduced by eliminating the caustic dissolution step.

Reduction of Dissolver Time Cycle. The present Redox dissolver cycle, consisting of the coating-removal step and two uranium-dissolution cuts, requires 32 hours. It is expected that it will be possible to complete a two-cut catalytic dissolving cycle in 24 hours.

Reduction in Aluminum Consumption. At present, the aluminum from the slug jackets is discarded as waste. In catalytic dissolving, however, the jacket aluminum is retained in the dissolver solution, and the amount of aluminum nitrate which is used subsequently as a salting agent for solvent extraction is thus reduced.

Improvement in Solvent Extraction. When the irradiated uranium fuel elements are dissolved by the present two-step procedure employing first, caustic to remove the aluminum jackets, followed by acid to dissolve the uranium metal, gelatinous solids are produced and suspended in the uranium dissolver solution. These solids, principally silicon

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and tin compounds from the slug jacket bonding layer, are incompletely removed by the Redox Plant feed centrifugation step and contribute to emulsification (and hence impaired capacity and performance) in the solvent-extraction columns. By contrast, acid dissolution of the aluminum jackets results in granular, easily centrifuged silica and is expected to minimize emulsification in the extraction columns.

Elimination of Coating Waste Disposal. Catalytic dissolving also opens the way for flowsheet changes in the Redox Plant, which would result in a reduction in the ultimate volume of Redox waste stored in underground tanks, assuming that the present requirement for underground storage of the caustic coating waste is not relaxed.

II. OBJECTIVES

The objective of the experimental studies reported herein was to demonstrate the laboratory-developed mercury-catalyzed dissolving procedure on a pilot-plant scale by conducting a series of test dissolutions of aluminum-jacketed, unirradiated uranium slugs in the 150-gallon (about 1/14 plant scale on a volume basis) downdraft type semiworks dissolver in 321 Building. It was desired to establish: 1. the effect of the larger-scale operation on time cycle, 2. dissolution rates of aluminum and uranium, 3. controllability of the reactions, and 4. the concentration of mercury catalyst required.

III. SUMMARY AND CONCLUSIONS

It has been demonstrated on a pilot-plant scale that two-cut mercury-catalyzed dissolving of aluminum-jacketed uranium fuel elements is feasible and that the dissolution process may be readily controlled. However, results of these semiworks dissolving runs, supplemented by later laboratory studies, indicate a potential explosion hazard due to hydrogen and oxygen concentrations in the dissolver off-gas. The mercury-catalyzed dissolving procedure is not considered ready for a Redox Plant trial until further laboratory and semiworks studies have succeeded in eliminating this potential hazard or proving that it does not exist.

To obtain complete dejacketing plus rapid uranium dissolution, it was necessary to use in the first cut a concentration of the mercuric nitrate catalyst, $\text{Hg}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}$, equal to 5 percent of the weight of the aluminum [approximately 0.0025 M $\text{Hg}(\text{NO}_3)_2$ in the 2 M uranyl nitrate dissolver solution]. Complete aluminum removal could not be achieved, under the test conditions, with catalyst concentrations of 2 percent or less.

The shortest time cycle was achieved when nearly all the aluminum was dissolved in the first cut. The second cut was then made with 60 percent nitric acid. In this manner, advantage was taken of the fact that uranium dissolves faster in concentrated acid, but aluminum dissolves more rapidly in dilute acid.

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The total required dissolving time for a two-cut charge in the pilot-plant dissolver was about 18 hours.

An acid efficiency* of about 80 percent for the first cut and 65 percent for the second cut was achieved in a catalytic dissolving cycle employing a downdraft condenser under optimum conditions (100 percent bare-uranium heel, 5 percent catalyst in the first cut; 60 percent acid in the second cut). (auth)

IV. EQUIPMENT

A. Downdraft Dissolver. The 321-Building dissolver with downdraft condenser is illustrated in Figure 1. The purpose of the downdraft condenser is to recover nitrogen oxides by absorbing them in the condensate. The dissolver diameter is 2.5 ft. The downdraft condenser is a 6-inch diameter pipe, 18-feet long, with a water-tube bundle consisting of seven 1/2 inch, schedule - 40 pipes. The lower 13-ft. portion of the condenser is packed with 1/2-inch Raschig rings.

A coil in the dissolver and a jacket around the tank may be used for circulating either water or steam. In this program, the coil was used only for heating and the jacket was used for cooling after completion of a run.

Low-pressure compressed air was metered into the top of the dissolver tank, permitting controlled air addition for oxidation of NO to NO₂, only the latter being absorbed in the downdraft condenser.

* 100 percent efficiency = 3 moles of HNO₃ consumed per mole of Al dissolved + 2 moles of HNO₃ consumed per mole of U dissolved. This represents ideal downdraft dissolving conditions in which all nitrogen oxides are recovered and oxidized by air. The overall reaction for uranium dissolving being: $U + 2HNO_3 + 1.5 O_2 \rightarrow UO_2(NO_3)_2 + H_2O$.

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B. Instrumentation. The dissolver is equipped with instrumentation for the following variables:

Temperatures:

Dissolver pot
Vapor line
Reflux
Off-gas
Condenser water inlet
Condenser water outlet

Pressures:

Pot coil steam
Dissolver pot
Off-gas stack

Flow rates:

Air addition
Off-gas
Reflux
Condenser water

Specific gravity of solution

Dissolver weight factor

Heat duty (from measurement of steam condensate)

C. Sampling Devices. Samples of dissolver solution were withdrawn through a vented outlet near the bottom of the dissolver. Off-gas samples were collected by connecting a gas sample bottle between a water aspirator and a sampler on the off-gas stack.

V. OPERATION

Metal was charged to the dissolver by hand through the charging chute. The bare uranium heel in all the runs consisted primarily of partially dissolved uranium slugs, but when the available amount of partially dissolved slugs was insufficient, new 8-inch uranium slugs were used to complete the heel.

The charge consisted of 8-inch aluminum-jacketed reject slugs (Al-Si-bonded) which contained an average of 7.8 lb. of uranium and 0.24 lb. of aluminum. The weights of uranium and aluminum are not known accurately since no effort was made to select only those slugs which were of the correct weight.

Nitric acid for dissolving was weighed in through chemical addition tanks. The first cut of each run was started with 45 percent nitric acid, enough of which was used to cover the slugs completely. Sixty percent

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nitric acid was added gradually after the aluminum-dissolving reaction had started. The lengths of time for acid addition and the relative amounts of 45 and 60 percent acid were varied from run to run.

In all runs but one, the acid for the second cut was added at the beginning of the cut. Second cuts were made with either 45 or 60 percent nitric acid.

Mercury catalyst, in the form of a 1.0 M $\text{Hg}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}$ solution, was added to the initial acid charge in the addition tank. Catalyst was used in the first cut of every run and in the second cuts of two runs.

Air was metered into the top of the dissolver at a rate of 4.5 cu.ft./min. (STP).

A steam jet in the off-gas line was used to maintain a vacuum of 1 to 3 inches of water in the dissolver pot. The vacuum in the off-gas line was between 3 and 7 inches of water.

The dissolver contents were not sparged during the first two runs, but in all subsequent runs, a 5-minute air sparge was used every 30 minutes.

The dissolver heating coil was connected to the 321 Building high pressure steam supply (approximately 120 lb. per sq.in. gage) through a pressure-reducing valve. The steam was maintained at the highest pressure which could be used without causing the condenser column to flood.

Samples of dissolver solution were taken every 30 minutes for the first two hours of each cut and hourly thereafter. Gas samples were taken at irregular intervals.

The end point of a cut was established by a decrease in a rate of change of specific gravity and vapor temperature. It was generally attempted to continue the run to a point of nearly complete acid consumption. After completion of a cut, the dissolver solution was diluted with water to approximately 2 M UNH and jetted to a storage tank. Following each cut, the dissolver lid was opened and the remaining metal was inspected.

VI. DISCUSSION AND RESULTS

This program of six runs established that a catalytic dissolving technique for aluminum-jacketed uranium slugs is feasible on a large scale. However, a greater concentration of catalyst was required than had been anticipated on the basis of laboratory studies.

A. Effect of Catalyst Concentration. The amount of aluminum dissolved during the first cut increased with the catalyst concentration. In Run 1, for which the amount of $\text{Hg}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}$ was 1 percent by weight of the aluminum, 80 percent of the aluminum was dissolved in the first cut.

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In Runs 2 and 3 in which 2 percent catalyst was used the percentages of the aluminum dissolved in the first cuts were 88 and 90, respectively. In Runs 5 and 6, with 5 percent catalyst, the percentages of aluminum dissolution were 93 and 96, respectively, in the first cut.

In Run 4, 1 percent of catalyst (based on total aluminum in charge) was added in both the first and second cuts, but a total of only 71 percent of the aluminum was dissolved in both cuts. Considerable flooding of the condenser column occurred during this run which made it necessary to reduce the steam pressure. It is believed that the poor performance in Run 4 was a result of foaming and flooding caused by the larger batch size and decreased freeboard.

Figure 2 shows the progress of aluminum dissolution for the six runs.

B. Effect of Uranium Heel. The dissolution rate of uranium in nitric acid of a given concentration is directly proportional to the surface area of the metal; a conclusion which is borne out by the results of this program.

In Run 1, the uranium heel was 50 percent. (Heel is expressed in percent of charged uranium. Thus, a 100 percent heel would equal 50 percent of the total uranium in the dissolver at the start of a run.) In 9 hours of operation, only 40 percent of the charged uranium was dissolved. In Runs 2 and 6 with 100 percent heel, 60 percent of the uranium charge was dissolved in 7 to 7.5 hours. In Run 3 with 200 percent heel, 67 percent of the uranium charge was dissolved in 6 hours.

Figure 3 shows the progress of uranium dissolution for the six runs and illustrates the effect of heel size.

C. Effect of Nitric Acid Concentration. An initial charge of 45 percent nitric acid plus a continuously added amount of 60 percent acid was used for the first cut of each run. Except for Run 3, equal volumes of 45 percent and 60 percent acid were used. The total amount of acid in the first cut was 2.8 moles of nitric acid per mole of uranium charged. In Run 3, a larger portion of the acid was added as 45 percent nitric acid in order to cover the metal completely, but the acid-to-uranium ratio was held at 2.8 moles per mole.

The aim of continuous acid addition was to take advantage of the fact that aluminum dissolution is most rapid in fairly dilute acid while uranium reacts more rapidly in concentrated nitric acid. In the absence of other nitrates, aluminum dissolution reaches a peak rate at a nitric acid concentration of 23 percent.

While the continuous acid addition periods for the six runs ranged from 35 to 75 minutes, it is not possible to isolate the effect of addition rate from the effects of other variables. In general, it appears desirable to start continuous acid addition as soon as the aluminum

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dissolution rate reaches its peak (as indicated by a maximum in the off-gas flow) and to add the acid at such a rate as to maintain a constant nitric acid concentration in the dissolver. The second cuts of Runs 1 and 2 were made with 45 percent acid; second cuts of runs 3, 5, and 6 were made with 60 percent acid. In each of these runs, all the acid was added at the beginning of the cut and amounted to 2.3 moles of nitric acid per mole of uranium (as charged).

The effect of going from 45 percent to 60 percent acid for the second cut may be illustrated by a comparison of Runs 2 and 6. In Run 2, 45 percent nitric acid was used. During 10.5 hours of dissolving, only 36 percent of the uranium charge was dissolved and the final acid concentration was 2.3 moles of HNO_3 per mole of UNH. In Run 6, 60 percent acid was used. During 10 hours of dissolving, 72 percent of the uranium charge was dissolved and the final acid concentration was -0.1 mole of HNO_3 per mole of UNH (i.e., this much deficient in HNO_3 with respect to stoichiometric neutrality).

The change of nitric acid concentration during dissolving is plotted for all six runs in Figure 4.

D. Effect of Air Sparging. The dissolver contents were not sparged at any time during Runs 1 and 2. During all subsequent runs, the dissolver solution was sparged with air for five minutes every half hour.

Air sparging was found to be effective in removing the bonding layer material from de jacketed fuel elements, since at the conclusion of unsparged runs, it was found that many of the slugs were coated with reddish-brown bonding layer material, but at the end of sparged runs, the surface of de jacketed slugs consisted of bare uranium metal. By removing the bonding material and exposing the bare metal, air sparging is effective in increasing the uranium dissolution rate.

E. Effect of Freeboard. The amount of freeboard (free space between the surface of the solution and the top of the tank) must be fairly large in order to permit dissolving to proceed at a maximum rate, since both aluminum dissolution and uranium dissolution are accompanied by foaming. As a result, an increase in the volume of metal and solution in the dissolver may require a decrease in the heat input to the dissolver in order to slow the reaction rate sufficiently to prevent overflowing the tank and flooding the condenser.

Since the dimensions and proportions of the 321 Building dissolver are quite different from those of a Redox Plant dissolver, it was felt that an attempt to determine optimum freeboard in these pilot-plant tests would be unprofitable. The 321-Building dissolver has a total capacity of 150 gallons (including charging chute). The cylindrical section of the pot (see Figure 1) holds about 70 gallons. The volume of solution plus metal for the runs in the program ranged approximately from 27 to 48 gallons, corresponding to freeboards of 38 to 30 inches. The 2000-gallon Redox dissolvers are filled to a maximum of 1150 gallons,

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(about 2 hours before the end of a cut) leaving a minimum freeboard of 33 inches.

In a qualitative way, the adverse effect of reduced freeboard may be gathered from a comparison of Runs 1 and 4. In Run 4, the amount of metal was 73 percent greater and the volume of solution 30 percent greater than in Run 1. Only 50 percent of the aluminum was dissolved in the first cut of Run 4 as compared with 80 percent in Run 1. Figure 8 is a photograph of the slugs in the dissolver after the first cut of Run 4. It can be seen that while some are completely dejacketed others are hardly touched.

F. Off-Gas Composition. Since some hydrogen is liberated in the reaction between nitric acid and aluminum, there is concern about the potential explosion hazard created by hydrogen in the stack gases. Therefore off-gas samples were taken during several runs and analyzed for hydrogen.

The lower explosive limit for hydrogen in air is 4.1 percent by volume (2). Table II shows that only one of the dissolver off-gas samples (the first sample for Run No. 6) had a hydrogen concentration close to this limit. However, in the light of subsequent laboratory studies, it now appears likely that in the other runs the point of maximum hydrogen evolution was missed on the sampling schedule. This sample taken at "zero" time in the first cut of Run 6, was collected at the time of maximum aluminum reaction, as indicated by high off-gas rate. The analysis of the gas sample shows that the amount of air added was barely enough for oxidation of the nitric oxide, since there was only 1.9 percent oxygen in the off-gas. The differential pressure across the condenser at the time of sampling was 1.2 inches of water, confirming that the air leakage rate was fairly low.

The mercury-catalyzed dissolving process is not considered ready to be recommended for a trial in the Redox Plant until this potential explosion hazard from hydrogen has been further studied, and either eliminated or proved not hazardous.

G. Ammonium Concentration. The maximum concentration of ammonium ion in the dissolver solution at the end of a cut was 4×10^{-4} mole per mole of UNH. Ammonium ion concentrations for all runs are listed in Table III. Mass spectrometer analyses of the off-gas did not show the presence of any ammonia.

H. Determination of End Point of Reaction. As the nitric acid in the dissolver is depleted, a decrease occurs in the rate at which the vapor temperature and the solution specific gravity change. It has been suggested that one of these two variables would offer a good index for determining the reaction end point.

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The specific gravity of the dissolver solution and vapor temperature are shown plotted against time in Figures 5 and 6. It was found that the vapor temperature reading did not give a very good index of reaction end point in the pilot-plant study, due to the location of the temperature-sensing element which was such that a true equilibrium vapor temperature was not recorded. For example, it was found that a change in steam pressure to the coils produced a change in the vapor temperature.

The rate of increase in specific gravity decreases as the reaction end point is approached, and in order to establish what rate of change of specific gravity could be used for the end point determination, it would be necessary to have the data for several identical runs. Such data were not obtained in the pilot plant.

For actual plant operation, either vapor temperature or specific gravity would probably be a satisfactory index of completeness of reaction. The variables, such as steam pressure acid concentration, and percent heel, which affected vapor temperature and specific gravity in the pilot-plant operation would presumably be constants in the plant. It will be necessary to develop an end point index in a full-scale test.

I. Corrosion. Corrosion coupons of stainless steel types 304L, 309 SCb and 347 were suspended in the dissolver during the last five of the six catalytic dissolving runs. The total exposure time of 95 hours was insufficient to obtain meaningful data as to the relative merits of the three alloys. No intergranular attack occurred on any of the coupons. Corrosion rates in inches per month ranged from 0.0003 to 0.0006 in the vapor phase and from 0.0001 to 0.0002 in the liquid phase.

VII. FUTURE WORK

Full-scale Redox Plant trials of catalytic dissolving cannot be recommended until the possibility of potentially explosive hydrogen concentrations in the off-gas has been eliminated. A laboratory program attacking this problem is now in progress.

Possibilities to be explored include methods for removing the hydrogen from the off-gas and techniques for smoothing out and lowering the peak evolution in the aluminum dissolving reaction, thus maintaining the hydrogen at a non-hazardous concentration. When satisfactory conditions are demonstrated in the laboratory for eliminating this potential hazard, it is planned to confirm the optimum mercury-catalyzed dissolving conditions in the 321-Building semiworks-scale dissolver before recommending a trial in the Redox Plant.

REFERENCES

- (1) HW-30838: Application of the Mercury-Catalyzed Aluminum-Jacket Dissolving Technique to the Redox Process, by J. L. Bradford, M. H. Curtis and M. K. Harmon, December 11, 1953.
- (2) N. Irving Sax, Handbook of Dangerous Materials New York: Reinhold Publishing Co., 1951, p. 199.

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DETAILS OF 321 BUILDING CATALYTIC DISSOLVER RUNS
(See Figure 1 for equipment details)

NOTES:

(2) $\text{Hg}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}$ as weight per cent of aluminum in charge.

$$(3) \quad 100 \text{ per cent efficiency} = 3 \frac{\text{moles HNO}_3 \text{ consumed}}{\text{mole Al dissolved}} + 2 \frac{\text{moles HNO}_3 \text{ consumed}}{\text{mole U dissolved}}$$

TABLE II

ANALYSES OF OFF-GAS SAMPLES TAKEN DURING 321 BUILDING CATALYTIC
DISSOLVER STUDIES

(See Table I for Run details and Figure I for equipment details.)

Run	Cut	Hours from Start of Cut	Volume Per Cent					
			$m/e=44^{(3)}$	A	O ₂	N ₂	H ₂	$m/e=30^{(4)}$
2	1	1(1)	0.12	1.02	10.38	88.47	0.01	
		3(2)	0.3	1.0	12.5	83.4	0.01	2.8
		5(2)	0.3	1.0	11.3	85.6	0.01	1.8
3	1	3(1)	0.08	1.13	5.30	93.49	0.01	
3	2	3.5(1)	0.11	1.04	11.87	86.98	0.01	
4	1	0(1)	1.93	0.92	15.10	80.33	1.39	0.33
		0(2)	5.01	0.90	14.64	77.18	1.07	1.2
		1.5(1)	0.33	1.09	8.27	90.31	0.01	
5	1	3(2)	0.7	1.0	9.2	88.2	0.1	0.7
5	2	2.5(2)	0.7	1.1	3.2	93.4	0.1	1.5
6	1	0(2)	8.6	0.9	1.9	82.5	4.0	1.7
		2(1)	0.1	1.1	2.9	95.6	0.2	
		5(1)	0.1	1.1	10.8	87.4	0.2	0.2

- (1) Sample analyzed after liquid-nitrogen freeze-out, i.e., sample is immersed in liquid nitrogen which reduces temp. to -196°C.
- (2) Sample analyzed without liquid nitrogen freeze-out.
- (3) An m/e , i.e., molecular mass divided by the charge, equal to 44 could be CO₂ or N₂O.
- (4) An m/e equal to 30 could be NO or N₂O₂.

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TABLE III

AMMONIUM ION CONCENTRATION SOLUTION DURING 321 BUILDING
CATALYTIC DISSOLVER STUDIES

(See Table I for Run details and Figure I for equipment details.)

<u>Run</u>	<u>Cut</u>	<u>Moles NH_4^+ per Mole UNH at End of Cut</u>
1	1	9×10^{-5}
	2	3×10^{-4}
2	1	2×10^{-4}
	2	2×10^{-4}
3	1	7×10^{-5}
	2	3×10^{-4}
4	1	8×10^{-5}
	2	1×10^{-4}
5	1	1×10^{-4}
	2	2×10^{-4}
6	1	1×10^{-4}
	2	4×10^{-4}

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FIGURE 2
PROGRESS OF ALUMINUM DISSOLUTION DURING 321-BUILDING
CATALYTIC DISSOLVER STUDIES

(See Table I for Run Details and Figure 1 for Equipment Details)

FIGURE 3
PROGRESS OF URANIUM DISSOLUTION DURING
321-BUILDING CATALYTIC DISSOLVER STUDIES
(See Table I for Run Details and Figure 1 for
Equipment Details)

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FIGURE 4
NITRIC ACID CONCENTRATION DURING 321-BUILDING CATALYTIC
DISSOLVER STUDIES
(See Table I for Run Details and
Figure 1 for Equipment Details)

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FIGURE 5
CHANGE IN SPECIFIC GRAVITY OF DISSOLVER SOLUTION
DURING 321-BUILDING CATALYTIC DISSOLVING STUDIES
(See Table 1 for Run Details and Figure 1 for Equipment Details)

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FIGURE 6
DISSOLVER VAPOR TEMPERATURE DURING 321-BUILDING
CATALYTIC DISSOLVING STUDIES
(See Table I for Run Details and Figure 1 for Equipment Details)

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FIGURE 7
PROGRESS OF 321-BUILDING CATALYTIC DISSOLVER
RUN NO. 6

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Photograph Secret HP-1084

**FIGURE 8: APPEARANCE OF METAL AFTER COMPLETION OF FIRST
CUT IN 321 BUILDING CATALYTIC DISSOLVER RUN NO. 4**
(Wires are hangers for corrosion samples)

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