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GENERAL ELECTRIC
COMPANY

RICHLAND, WASHINGTON

MANAGED ATOMIC PRODUCTS OPERATION

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BY *GW Ordas* DATE *6/17/81*

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CONSIDERATIONS FOR ADDITIONAL 321 BUILDING MERCURY
DISSOLVING STUDIES

INTRODUCTION

Studies in the 321 Building dissolver during December, 1953 and January, 1954, were successful in developing a laboratory-proved mercury-catalyzed dissolving flowsheet into a suitable plant procedure. However, this flowsheet was not adapted for Redox plant operation because of uncertainty about the possible presence of hydrogen above the lower explosive limit in the off-gases. Subsequent laboratory work has resulted in a better understanding of the hydrogen evolution, and has resulted in developing low hydrogen evolution flowsheets. When one of these flowsheets is selected for further work, it will be tested in the 321 Building dissolver with non-irradiated slugs to provide information for scaling-up the single-slug laboratory data to a plant-scale operation.

OBJECTIVE

It is the purpose of this memorandum to outline the factors considered to be pertinent to the 321 Building investigation, to be used as a guide in making preparations for the runs to be performed.

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DISCUSSION

The run plans of the previous series may be used as a guide in preparing run plans for this series. Except for increased interest in the hydrogen content of the off-gases, the run objectives are similar and similar data should be obtained.

Two possible flowsheets may be used. Both of these are shown in the attached appendix. Most of the following discussion generally covers both; specific items pertaining to only one flowsheet are so indicated.

A. Initial Conditions.

Physical properties of solutions are not to be studied, so normal vessel cleanliness is permissible in this series of runs. (Special precautions need not be taken to remove caustic coating removal residues as was required in the last series of runs.) It is desirable that the heel material be the result of several successive dissolvings so that the slugs shall be in various states of dissolution with a surface-to-volume ratio found in plant dissolver heels.

B. Run Conditions.

The reaction should be controlled by heating and cooling to maintain a constant pot vacuum with a rapid reaction rate. Ample control should be available to stop the reaction by turning off the steam and applying cooling water, but a solution of cupric nitrate will effectively poison the catalytic action if this is required. Drowning the reaction by addition of water may be used, in contrast to the previous run series, but most of the effect will be due to cooling rather than dilution.

The air introduced into the down-draft condenser, the sparge air, and that air which gets in by leakage should be constant throughout the runs and the series. Air sparge should be used as the equipment permits, and especially during the period in which the vessel is being cooled down after jacket dissolving. (The sparge knocks loose the scale from the slugs and exposes the U metal for more rapid subsequent dissolving.)

An important objective of this series of runs is to determine the minimum time cycle for this dissolution. This can be done only if runs are long enough to permit complete jacket removal; solution analysis can locate the end of jacket removal in an extended run but not in a run which is terminated too early. Previous experience indicates that reaction in the 321 Bldg. equipment rates may be several fold lower than those found in the laboratory. The runs must be long in duration to insure the success of this series.

C. Sampling.

Obtaining data on off-gas composition is a prime objective of this series of runs. Duration of the peaks in the hydrogen evolution in the laboratory was as short as 10 minutes, but averaged from 30 to 60 minutes for single-slug dissolvings; plant peaks may be broader for multiple-

slug dissolving. About 30 sampling bulbs are available from Process Chemistry. The ability of Analytical (100-B mass spectrometer) personnel to rapidly return the used bulbs, the facilities for cleaning the bulbs, as well as all of the foregoing considerations will determine the gas sampling schedule.

The total time to be consumed in the series of runs will be largely dependent on the speed with which analytical results are obtained on solution samples. Assigning priorities and carefully controlling the submission of samples will do much to speed up this series of runs. No adverse effects will be encountered if sodium ion is used for a "tracer" in this series as in previous dissolver work.

D. End of Run Operations.

No solution dilution is required to prevent freezing or reduce the viscosity of the coating solutions produced by this type of flowsheet. Little uranium will be dissolved, hence the heel will not be appreciably affected. It might be preferred to remove the slugs manually after each jacket removal run rather than to dissolve them; in any event the dissolver contents should be inspected after each removal run to see if the coats were completely removed.

E. Specific Flowsheet Considerations.

1. Flowsheet A. - After the first run with this flowsheet, the ANN for the jacket removal solution for subsequent changes should probably be made up by acidifying the jacket removal solution from the preceding run, to duplicate planned plant operation. Analysis of such solutions would be required to insure proper run conditions.

In the acid-deficient coating solution produced by this flowsheet, the mercury present is insoluble, and will collect as the metal at the bottom of the dissolver. Determining the fate of this mercury is an objective of this series of runs; the path of the mercury also must be known to insure controlled mercury concentrations in the solutions used in the runs. Heating a dilute nitric acid solution in the dissolver following the coating removal step (obtaining volume and mercury analysis on the wash) should be sufficient to remove any pools of mercury metal from the tank bottom.

2. Flowsheet B. - The success of this type of run lies in the rate of acid addition. If acid is added too rapidly, excessive hydrogen is formed, while if the acid is added too slowly the reaction will take too long. As indicated previously, the reaction rates in the 321 Building dissolver may be considerably lower than in the laboratory set-up; hence, laboratory-scale rates of acid addition may be too high for this series of runs. In any event, a series made with this flowsheet will probably be devoted to determining the optimum rate of acid addition commensurate with hydrogen evolution and rate of dissolving. Such a program might be dependent on gas and solution sample analysis results, but it may be that pot vacuum or tower differential pressure readings may allow some latitude during runs, because hydrogen evolution is directly related to off-gas evolution rate.

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APPENDIX

PROPOSED FLOWSHEETS FOR MERCURY-CATALYZED
ACID DISSOLVING OF SLUG JACKETS

Flowsheet A

The jacket is dissolved, with the aid of 5% mercury catalyst, in a solution of aluminum nitrate, resulting in a final solution of basic aluminum nitric.

1. Charge jacketed slugs to a dissolver containing a heel of bare slugs (100% heel, or heel weight equal to charge weight).
2. Charge jacket removal solution, 1.5 M ANN, to dissolver (1600 ml. used to de-jacket one 4" slug 58 grams of Al).
3. Add catalyst to the dissolver. (A solution of 1 M mercuric nitrate in 6 M HNO_3 . Weight of $\text{Hg}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}$ equal to 5% of total aluminum in charge.)
4. Heat to boiling. Maintain at boiling until end of run.
5. End of the run will be indicated when specific gravity of successive solution samples becomes constant.

Flowsheet B

The jacket is dissolved, with the aid of 5% mercury catalyst in a medium of water to which nitric acid is slowly added.

1. Charge jacketed slug to a dissolver containing a 100% heel of bare slugs.
2. Add initial water (800 ml. used for 4" slug).
3. Add mercury catalyst (5%).
4. Heat to boiling.
5. Start adding 60% HNO_3 at a rate that will complete the acid addition in 90 minutes (400 ml. of acid for one 4" slug).
6. Maintain boiling conditions until end of run.
7. When successive solution samples show constant specific gravity, the run may be terminated.

END

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