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DATE: November 5, 1956
SUBJECT: Suggested Procedure for Operation
of the Dissolver System
TO: S. E. Beall
FROM: H. O. Weeren

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To: S. E. Beall
From: H. O. Weeren
Date: November 5, 1956
Subject: Suggested Procedure for Operation of the Dissolver System

The following procedure is a modification of and supplement to the operating procedure outlined in CF 56-3-22, "Operating Procedure for the ERT Chemical Plant". The operation of the dissolver system is given in some detail. Comment and questions are welcomed.

The operation of the high pressure system will be unchanged from that given in CF-56-3-22. The difference in procedure will occur after the charge has been dropped from the underflow pot to the dissolver and after the underflow pot has been rinsed (Step IV). From this stage the procedure is as follows:

I. Refluxing the Dissolver

At the present time it seems likely that the high pressure system should be put back on stream as quickly as possible. If such should be the case, the dissolver contents can be refluxed until such time as manpower is available and the dissolving operation can be started

1. Maintain CW to H-1 at 4 gpm.
2. Maintain CW to H-14 and H-16 at 1 gpm.
3. Maintain 250 lb steam on superheater H-7.
4. Close valve HCV-1112-2.
5. Open valve HCV-T-3-2.
6. Drain CW from condenser E-20.
7. If heat in addition to the heat of radioactivity is necessary, regulate the steam pressure to the dissolver jacket to provide a boil-up rate of 3 to 5 lbs of D₂O per hour. This rate can be checked by observing the steam flow integrator (FEC-1) for a reasonable length of time.
8. Check dissolver temperature and pressure hourly.
9. If the pressure in the dissolver vapor line increases during refluxing, this is an indication of plugging in the entrainment separator. Cooling water should then be turned on the S-2 reflux coil until the pressure returns to normal.

II. Boildown of the Dissolver

This is the first step of the dissolving operation.

1. Close HCV T-3-2.
2. Observe the rise in level of T-2 and the temperature of the dissolver. When almost all of the D₂O has been boiled off this temperature will rise suddenly. Open HCV T-3-2 and allow the contents of T-3 to flow into the dissolver, thereby keeping the solids from being baked.

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III. Isolation of Dissolver from D₂O System

This procedure is proposed to keep contamination of the D₂O inventory to a minimum. The D₂O system is sealed off from the dissolver by valve and freeze plug combinations.

1. Shut off the steam (if any) being sent to the dissolver jacket.
2. Close HCV D-3-6 and HCV D-3-4.
3. Thaw freeze plugs FP-17 and FP-18. This will charge a small quantity of D₂O to the space around FP-1 and FP-2.
4. Freeze plugs FP-1, FP-2, FP-17, and FP-18.
5. Turn CW on the dissolver jacket. Observe the lowest reading on PE D-3. A reading that stays significantly below the reading of PE C-1 indicates a good seal between the D₂O and the H₂O systems.
6. Thaw FP-16. Allow to remain unfrozen 15 minutes.
7. Freeze FP-16. This refills T-24.

IV. Charging Dissolving Solution

The dissolving solution is charged by gravity from outside the cell.

1. Thaw plugs FP-13 and FP-14.
2. Charge dissolving acid to T-26. 81 gram mols of H₂SO₄ will be needed.
3. Valves HCV T-21-2, HCV T-22-2, and the valves in the dissolver sampler should be closed.
4. Open HCV D-3-8 and HCV T-26-2.
5. Valve HCV D-3-2 is opened. T-26 is vented to the dissolver system pressure. The valve below T-26 at the cell wall is opened and the acid is charged to the dissolver.
6. The valve below T-26 and the vent valve are shut off and a small quantity of H₂O is charged to T-26.
7. The valves are opened and the water is allowed to flow into the dissolver. This water serves to wash the concentrated acid off the valves and piping and reduce corrosion. The valve at the cell wall is closed. HCV T-26-2 is closed.

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V. Dissolving

The solids are dissolved by refluxing them with a solution of 10.8 M H_2SO_4 , diluting to 4 M, refluxing, concentrating back to 10.8 M, and repeating the procedure.

1. Shut off the flow of CW to the dissolver jacket. Send CW to the jackets of H-20, H-21, and H-22. Close HCV D-3-2.
2. Regulate the steam pressure to the dissolver jacket to start the solution boiling. Note the boiling point and the system pressure. Reduce the flow of CW to H-20 until enough water distills over to bring the solution boiling point to the correct value for 10.8 M acid. Resume the flow of CW to H-20.
3. Reflux for 6 hours. Shut off the steam to the dissolver jacket and allow to cool.
4. Open HCV D-3-2 and HCV T-26-2 and add sufficient water from T-26 to dilute the acid concentration to 4 M. Close the valves.
5. Reflux for 4 hours.
6. Reduce the flow of CW to H-20 and distill over water until the acid concentration reaches 10.8 M as indicated by a correlation of acid concentration with boiling point and system pressure. Resume the flow of CW to H-20.
7. Reflux for 6 hours. Shut off the steam to the dissolver jacket and allow to cool.
8. Repeat step 4.
9. Reflux for 1 hour.

VI. Sampling

A HRT low pressure sampler is used to obtain the sample. Pressurization is used to get the sample to the sampler. The solution that has passed through the sampler is held in T-23 until a satisfactory sample has been obtained, then dropped back to the dissolver.

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1. Close HCV D-3-8. Shut the cooling water off H-20 and H-21. Turn cooling water on H-24. Open D-3-2 and the sampler valve. See that HCV T-23-2 is closed. Reduce steam flow to D-3 to the minimum practical value.
2. The pressure build-up in D-3 will force the liquid through the dip line, through the sampler and into T-23. The solution is cooled to reduce corrosion as it passes H-22 and H-24.
3. A sample is taken. Steam is cut off the jacket of D-3; HCV D-3-8 is opened. CW is sent to the jacket of H-20.
4. HCV T-23-2 is opened, allowing the solution in T-23 to return to the dissolver. HCV T-23-2 is closed.

VII. Transfer to the Decay Tanks

After sampling is complete, the solution is transferred to either of the two decay tanks by pressurization. The dissolver and sampler are then rinsed with water and the rinse solution is transferred to the decay tank.

1. Open HCV T-21-2 (or HCV T-22-2). Close HCV D-3-8. Shut the CW off H-20. Send steam to D-3 slowly.
2. The pressure build-up in D-3 will force the liquid through the dip line into T-21 (or T-22). A sudden decrease in the pressure in D-3 will indicate that transfer is complete. Close HCV T-21-2 (or HCV T-22-2). Shut the steam off the jacket of D-3.
3. Open HCV D-3-8. Charge 5 gallons of water from T-26 to D-3. Close HCV D-3-8.
4. Heat the solution in the dissolver and pressurize through the sampler circuit into T-23. Shut the steam off the jacket of D-3 and allow the pressure to fall to system pressure as measured by PR 8-3. Open HCV D-3-8. Turn CW on H-20.
5. Open HCV T-23-2 and allow the solution to return to the dissolver. Close HCV T-23-2.
6. Reflux the solution in the dissolver for 30 minutes.
7. Open HCV T-21-2 (or HCV T-22-2). Close HCV D-3-8. Turn CW off H-20.
8. Pressurize the solution from the dissolver to T-21 (or T-22).

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VIII. Drying the Dissolver

The dissolver is dried by heating with 250 lb steam, rinsing with a small amount of D_2O , and reheating.

1. Turn steam on H-20, H-21, and H-22. Turn full steam pressure on D-3. Heat until the temperature stops rising.
2. Close HCV D-3-2. Allow the dissolver to cool until the temperature reaches 215°F.
3. Charge a small quantity (500 cc) of D_2O into T-26. Open HCV D-3-2 and HCV T-26-2 and add this D_2O to the dissolver. Close HCV T-26-2.
4. Repeat step 1.
5. Close HCV D-3-2 and HCV T-21-2 (HCV T-22-2).
6. Thaw FP-15. Charge a small quantity of D_2O from T-26 to the freeze plugs. Freeze FP-13, FP-14, and FP-15.
7. Shut the steam off the jacket of D-3, H-20, H-21, and H-22. The pressure in D-3 should stay well below the pressure of PR S-3; this indicates a good seal between the H_2O and D_2O systems.
8. Thaw FP-1 and FP-2. Open HCV D-3-4 and HCV D-3-6.

IX. Decay Tank Operation

The solution is allowed to accumulate in the decay tank. The tank is kept cool to reduce corrosion. A natural convection loop is operated to recombine the hydrogen produced. Periodically the solution in the decay tank is concentrated to make room for fresh solution.

1. Turn steam on H-25 and R-1, CW on H-26. Flow should be adjusted to keep the temperature leaving H-26 at the desired value.
2. Turn CW on T-21. Adjust the flow to keep the solution temperature below 140°F.
3. Level indication in T-21 will be by a bubbler system. When the level gets too high in the tank the CW to T-21 is shut off and steam is sent to H-26 to keep this cooler from acting as a reflux condenser. The solution in T-21 is boiled (steam heat is added if necessary) and vented to S-3.
4. CW is sent to H-2 at 4 gpm and H-17 at 1 gpm.
5. Maintain 250 lb steam on superheater H-8.
6. The condensate is collected in T-25. Periodically the contents of this tank are jetted to the waste evaporator. The valve in the east valve pit must be opened before this jetting is done.

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7. The extent of the boil-down is followed by a periodic check of the level in the decay tank. When the level has fallen enough to indicate that sufficient water has been boiled off to increase the acid concentration to 4 M, boiling is stopped.

X. Transfer to the Carrier

Solution is accumulated in one decay tank for 13 weeks and allowed to decay for another 13 weeks while the other decay tank is being filled. At the end of the decay period the solution is dropped in batches to a transfer tank and pressurized outside the cell to a carrier. To empty T-22:

1. Attach the carrier to the flanged connections in the loading pit. Open the valves on the carrier and lines, open HCV T-27-2.
2. Thaw FP-19. Check the level in T-22 to confirm the filling of T-27.
3. Freeze FP-19, close HCV T-27-2, turn steam on T-27, turn cooling water on H-23.
4. The pressure build-up in T-27 will force the liquid through the dip line into the carrier. The carrier is vented into the process equipment off-gas system. The solution is cooled as it passes H-23.
5. A sudden drop in the pressure indicated on PR-27 will indicate the end of the transfer operation.
6. Shut cooling water off H-23. Open HCV T-27-2.

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