1250435

[0064444H]

Department of Energy
Richland Field Office
P.O. Box 550
Richland, Washington 99352

File

93-ERB-087

FEB 16 1993

RECEIVED
FEB 25 1993

Mr. David B. Jansen, P.E.
Hanford Project Manager
State of Washington
Department of Ecology
P.O. Box 47600
Olympia, Washington 98504-7600

Dear Mr. Jansen:

HEXONE REMEDIATION

This letter is in response to the State of Washington Department of Ecology (Ecology) request for the review of documents pertaining to the decontamination of several hexone remediation equipment items (Ecology ltr. to J. D. Bauer, U.S. Department of Energy, Richland Field Office [RL], and R. E. Lerch, Westinghouse Hanford Company [WHC], from G. T. Tebb "Remediation Equipment Decontamination, M-20-27," 9207414, dtd. October 12, 1992). As you will recall, WHC rinsed part of the distillate transfer system, specifically the two air-cooled condensers, the 30 gallon distillate transfer tank and pump, and the pipeline to the four receiver tank cars, as well as the four tank cars themselves. The letter requested the following information:

1. *Any spill reports associated with the hexone remediation process and equipment.*

   The hexone remediation process and equipment met stringent requirements for radionuclide and hazardous material containment through equipment design and operating practices. The facility did not experience hazardous waste spills, and consequently no spill reports are associated with the project.

2. *Original operating logs of the hexone remediation process and equipment decontamination.*

   The original operating logs of the hexone remediation process are available for inspection in Room A-101 of the 2750-E Building in the 200 East Area. Copies are provided as Enclosure 1. Operating logs were not used for the equipment decontamination. The work is recorded in the following documents:

   - Hexone Distillation System Rinse Summary (Enclosure 2)
   - Manifest for Trailer Loads #7 and #8 to Diversified Scientific Services Incorporated's incinerator; includes the rinsate (Enclosure 3)
   - Organic Vapor Monitoring Data for the Tank Cars (Enclosure 4)
   - Total (radio) Activity Analysis for the Rinsate from Condenser #1 (Enclosure 5)

RECEIVED
OCT 30 2018
EDMC

228

- The Hexone Storage and Treatment Closure Plan (DOE/RL Z-92-40) submitted to Ecology on November 30, 1992
- Job Safety Analysis for Loading Tank Trailer from Tank Cars (Enclosure 6)
- Job Safety Analysis for Rinsing Equipment Into the Tank Trailer (Enclosure 7)

We consider the two condensers, the product tank, the distillate pump and associated small length of piping to be free of hazardous materials by virtue of the large volume of flush solution used, exceeding the triple rinse criteria of 0.0018 gal/sq in./rinse, as approved by Ecology's letter (Ecology ltr. to S. H. Wisness from T. L. Nord "Equipment Decontamination Procedures," 9103435, dtd. July 24, 1991, [Enclosure 8]) by a factor of at least 10. We also consider the rinsed tank cars to be free of hazardous materials by virtue of the organic vapor measurements that all indicate no organics above the 0.1 parts per million (PPM) detection limit and verify absence of the solvents (hexone is highly volatile and the concentration of hexone in air, when liquid is present at room temperature in a closed system, is 19,700 PPM).

3. *Equipment Decontamination Waste Analysis Records.*

Decontamination effectiveness for the tank cars is recorded in the organic vapor detector measurements {included per Item (2) above, Enclosure 4}. Decontamination of the condensers/product tank/pump/piping system was carried out through the triple-rinse volume criteria, which does not require sampling. The #1 condenser rinsate was sampled for total (radio) activity. Results were below the detection level of <50 pCi/mL (compared to the average distillate concentration of more than 5,000 pCi/mL). {Included per Item (2) above, Enclosure 5}.

4. *Quality Assurance (QA) Documents Associated with the Waste Analysis Records.*

An instrument calibration log copy for October 7, 1992, the day of tank car vapor sampling, is enclosed as the only applicable QA document (Enclosure 4).

5. *Please Provide an Explanation of the Intended Future Deposition on Function of the Rail Cars Used in the Distillation Process.*

The four rinsed distillate tank cars are considered free of radioactive and hazardous materials and are in excellent mechanical condition. The tank cars are currently in use as spacer (idler) cars by the Hanford Railroad. Upon your concurrence with the cars' non-hazardous status, it is intended to start using the cars in the spring of 1993 for onsite haulage of non-hazardous materials delivered to the Richland interchange yard in commercial tank cars. Eventually the cars will likely be excessed or salvaged for scrap metal.

RECEIVED

Upon Ecology's review and concurrence with the rinsing and verification measures described above, WHC plans to classify the following equipment as non-hazardous:

- The four U.S. Department of Transportation 103-W tank cars that stored distillate
- The two condensers, 30-gallon product tank, pump and connecting piping
- The dismantled distillate transfer pipe
- The dismantled secondary containment piping

It is planned to reuse or salvage the two condensers, product tank, and pump. The rinsed distillate transfer pipe and secondary containment piping has been cut up and packaged into 55-gallon drums and is to be disposed of in the low-level waste landfill, as this piping was located in a radioactive surface contamination area and cannot be internally surveyed for unconditional radioactive release.

If you have any questions or require additional information, please contact Mr. Robert G. McLeod, RL, at (509) 372-0096, or Mr. Fred A. Ruck III, WHC, at (509) 376-9876 .

Sincerely,

James D. Bauer, Program Manager
Office of Environmental Assurance,
Permits, and Policy
DOE Richland Field Office

ERD:RGM

R. E. Lerch, Deputy Director
Restoration and Remediation
Westinghouse Hanford Company

Enclosures: As stated

cc: P. T. Day, EPA (w/o encls)
D. L. Duncan, EPA (w/o encls)
W. F. Heine, WHC (w/o encls)
R. E. Lerch, WHC (w/o encls)
T. M. Michelena, Ecology (w/o encls)
D. C. Nylander, Ecology w/o encls)
G. T. Tebb, Ecology (w/encls)

ENCLOSURE 1

Hexone Remediation Process Operating Logs

CONTROLLED COPY

JUN 27 1990

RETURN TO CLASSIFIED DOCUMENT CONTROL

NOTEBOOK NUMBER
WHC-N-408-1

DATE OF ISSUE
6-27-90

SERIES AND COPY
#1

TITLE (INDICATE SUBJECT CONTENT)
HEXONE REMEDIATION DEMONSTRATION PROGRAM

AUTHOR (S) - DEPARTMENT AND SECTION
R. J. CASH

PERIOD COVERED (INCLUSIVE - MONTH, DAY, YEAR)
FROM
TO

IF CONTINUED FROM ANOTHER NOTEBOOK, GIVE NOTEBOOK NUMBER

IF CONTINUED IN ANOTHER NOTEBOOK GIVE NOTEBOOK NUMBER

ROUTE TO	P. R. NO.	LOCATION	ROUTE DATE	SIGNATURE & DATE
R. J. CASH	90294			

APPROVED FOR PUBLIC RELEASE

CW 12-23-92

Date Received	INFORMATION RELEASE REQUEST	Reference:
12-23-92		WHC-CM-3-4

Complete for all Types of Release

Purpose

- ☐ Speech or Presentation
  - ☐ Full Paper
  - ☐ Summary
  - ☐ Abstract
  - ☐ Visual Aid
  - (Check only one suffix)
- ☐ Speakers Bureau
- ☐ Poster Session
- ☐ Videotape
- ☐ Reference
- ☐ Technical Report
- ☐ Thesis or Dissertation
- ☐ Manual
- ☐ Brochure/Flier
- ☐ Software/Database
- ☐ Controlled Document
- ☐ Other _____

ID Number (Include revision, volume, etc.): WHC-N-408-1

List attachments.

Date Release Required: 12-29-92

Title: Notebook Hexone #1 Hexone Remediation Demonstration Proj.

Unclassified Category: UC-

Impact Level:

New or novel (patentable) subject matter? ☐ No ☐ Yes
If "Yes", has disclosure been submitted by WHC or other company?
☒ No ☐ Yes Disclosure No(s). _____

Information received from others in confidence, such as proprietary data, trade secrets, and/or inventions?
☒ No ☐ Yes (Identify)

Copyrights? ☒ No ☐ Yes
If "Yes", has written permission been granted?
☐ No ☐ Yes (Attach Permission)

Trademarks?
☒ No ☐ Yes (Identify)

Complete for Speech or Presentation

Title of Conference or Meeting: N.A.

Group or Society Sponsoring:

Date(s) of Conference or Meeting:

City/State:

Will proceedings be published? ☐ Yes ☐ No
Will material be handed out? ☐ Yes ☐ No

Title of Journal:

CHECKLIST FOR SIGNATORIES

Review Required per WHC-CM-3-4	Yes	No	Reviewer - Signature Indicates Approval: Name (printed)	Signature	Date
Classification/Unclassified Controlled Nuclear Information	☐	☑			
Patent-General Counsel	☒	☐	B.D. Williamson	B.D. Williamson	12/21/92
Legal-General Counsel	☒	☐	B.D. Williamson	B.D. Williamson	12/21/92
Applied Technology/Export Controlled Information or International Program	☐	☑			
WHC Program/Project	☒	☐	G.T. BERLIN	G.T. Berlin	12-17-92
Communications	☐	☑			
RL Program/Project	☒	☑ CW	N/A		
Publications Services	☐	☑			
Other Program/Project	☐	☐			

Information conforms to all applicable requirements. The above information is certified to be correct.

	Yes	No
References Available to Intended Audience	☐	☐ N.A.
Transmit to DOE-HQ/Office of Scientific and Technical Information	☐	☒

Author/Requester (Printed/Signature): O.R. RASMUSSEN O.R. Rasmussen

Date: 12-17-92

Intended Audience

☐ Internal ☐ Sponsor ☒ External (WDOE)

Responsible Manager (Printed/Signature): G.T. BERLIN G.T. Berlin

Date: 12/17/92

INFORMATION RELEASE ADMINISTRATION APPROVAL STAMP

Stamp is required before release. Release is contingent upon resolution of mandatory comments.

APPROVED FOR RELEASE
OFFICIAL DOCUMENT
CW
Date 12-23-92

Date Cancelled

Date Disapproved

BD-7600-062 (08/9
Part 1

- Nitrogen and CO_2 addition to tank cars completed and O_2 check made on friday July 6, 1990 - all four cars were less than 2% oxygen

R K Cowan 7/9/90.

System completed 7/13/90

R G Cowan

APPROVED FOR PUBLIC RELEASE

CW 12/23/92

JULY 16, 1990 Surveyed oxygen concentration in exhaust duct sample port downstream of HEPA Filter. Oxygen concentration was 4-6 percent, indicated underground tanks are inert.
OR Rasmussen / [illegible] (PPE Tech)

Seal Loops in Underground Tank Pans have been filled with oil
OR Rasmussen

Tank cars have been filled with CO_2 (oxygen concentration 4-5 %. Tank cars are inert.
OR Rasmussen

Started flow 141 to distillation System - Temp of Hexane 69°
Time . 11:15 RG Cowan

the data logger locked up at the 11:45 log - restarted OK - this could be a problem
RG Cowan.

Increased Oil temp to 225°F at 13:10 RG Cowan

Increased oil temp to 235°F at 13:30 RG Cowan

Turned down heat untill 0 #1 press then reheated to get confirmed fill of Trap on overflow #1 vessel.
RG Cowan

Reset temp to 230°F looks good!
RG Cowan

74 RW

1327 hrs 7-16-90 Increased Oil temp. to 235° F. RGC

Turned off Product Transfer pump to build up enough level to take sample of material 1430 RGC

Still getting high temp on # overflow line (PO4) so tried another turn down and set back to be sure overflow trap is full

1445 RG Cowan

Turned on transfer pump and pumped from 44.7 gal → 9.1 gal = 35.6 gallons to the tank car

5:20p Second tank car transfer 32.7 → 9.3 = 21.4 gal
Total transfers 35.6 + 21.4 = 57 gallons

5:30p Took first product sample hexone 1

16:56 TOOK SAMPLE HEXONE 1 TO 222-S LAB

1700 Problem with #1 Vessel overflow temp — the seal loop is lost when the vessel cycles at too high a vessel pressure and vapor goes back down the line — Need to hold down rate until this can be resolved holding pressure 20-25 with low flow to #1 vessel ΔP at ~ 3-5 psi indicated
RG Cowan

1750 third transfer of product to tank car ~ 22 gal. RGC

1825 fourth transfer of product to Rail car — 22 gal RGC

ERW
ESW

7-16-90 — Run B first day (day 197)

We are continuing to chase the B.P. Temp on #1 vessel going up on oil temp as the Hexone boiling point goes up.
Now boiling at 220°F with oil at 247
RGC - 1940

Computer locked up on log print out at 1950 and could not be rebooted from program disk - went to back up disk - Will make new backup and replace all used disks but this could become a real problem / RGC

Set the N_2 cover pressure down to .25 - .30 was up to .4+, this is not holding real steady
Had some frost on the N_2 Bottle RGC/

HEXONE SAMPLE #1 RESULTS: 3,020 pCi/ml. BSP 23:35

AVES 23:30 7/16/90 → 8:00 7/17/90 RED/RICH/PAV & NEE

- PRODUCT TANK PUMP DID NOT KICK-IN. PRODUCT TANK FILLED TO 43.5 GALLONS AND WAS OVERFLOWING BACK TO THE UNDERGROUND TANKS. PROBLEM WAS THE ELECTRICAL RELAY IN THE 2ND PANEL BOX MUST HAVE BEEN STICKING. A FEW SWIFT PUNCHES TO THE BOX UNSTUCK THE RELAY AND THE PUMP KICKED-IN. (SEE BULLS-EYE ON SIDE OF PANEL). RSP

RGC

- DICK CHANGED OUT ALL 3 DATA DISKS ON DATA LOGGER AT SHIFT CHANGE. TO ASSURE WE HAD CLEAN DATA DISKS. HOWEVER, WHEN THE DATA LOGGER LOOKS FOR THE PREVIOUS DATA DISK, IT ISN'T THERE. SO WE'RE PROBABLY NOT RECORDING ON THE DISCS. PRINT-OUTS ARE NOW EVERY 20 MINUTES INSTEAD OF 10 MINUTES. (WHICH IS ADEQUATE). SINCE THIS ALSO SEEMS TO HAVE ELIMINATED THE COMPUTER LOCK-UP PROBLEMS, WE WILL LEAVE THINGS THE WAY THEY ARE. (IF IT AIN'T BROKE ... DONT FIX IT) THIS SHOULD CLEAR WHENEVER COMPUTER IS RE-BOOTED.
ALSO: THE ~~PRINTOUT~~ COMPUTER DOES NOT RETAIN THE PREVIOUS READING FOR PRINTOUT ~~ON THE PRINTOUT~~ BSP

RGC

RSP

— ON THE DATA SHEETS: THE NITROGEN HEADER PRESSURE IS READ ON THE NORTHEAST CORNER OF THE DISTILLATION MODULE ABOUT 8 ft high. (on the nitrogen header). PREVIOUS READINGS WERE ON THE DEMAND REGULATOR OF THE PRESSURE OF THE STEP DOWN REGULATOR. RSP.

RGL 2:15 Relay to transfer pump stuck again — 41 gallons. Sharp blow to "target" (bulkhead) remedied. RGL.

4:09 Relay stuck again RGL.

04:20 141 original volume: 19241
141 current 18355
886 gallons in RR. TANK
17 HOUR
= 0.9 gpm. AVG.

for GRANDYALD 18596
- 18355
241 gallon ∴ 4hr 50min = 0.8 gpm
low because of transfer pump being down.

06:00 for data sheet reading for tank car 3684
141 Start 19241
141 current 18321
920 gallon. this value used on data sheet since the value on data logger is erratic.

05:28 Had to thump product pump relay again. Should have electrician look at it on day shift. RSP

- DID NOT TAKE A SAMPLE. SEEMS LIKE ONE or two samples per day SHOULD PROVIDE ADEQUATE RECORD (9AM & 9PM?)

05:58 Adjusted cover gas pressure from .35 to .22 inches of water. Also noticed a slight smell of Hexane this is the reason for adjusting the pressure RGL

7-17-90 Tuesday

- Note: Currently the product tank will alarm high before it pumps out (Dick will reset this alarm point next time the system is down) RG

7/17/90 - WO 0930AM We sampled The product Tank Hexone 2 + Hexone 3. We archived Hexone 2 and sent Hexone 3 To The Lab. Hexone 3 was almost Totally organic while 2 had alot of water. RG

AM Note: We have adjusted To Magnehelic a couple of Times as it keeps dripping significantly below the 0.2 setting. RG

WOJ 11:30 - We sampled the product tank - Hexone 4 and Hexone Hexone 4 was archived and Hexone 5 was submitted for analysis The Lab Number for Hexone 5 is E 1772. RG

WOJ 14:15 - We Took four hexone Samples. RGC

WOJ 14:30 - Olaf raised oil Temp setting 1 deg to 264°. RGC

1530 Swing Shift RGC JWB DDP

Present processing rate is RG Cowan

10.8	34:31
14.3	44:12
3.5 gal	4:35

1600 adj. N_2 up to read ~ .22 on magnehelic J.J. Pain

Sample Labeled Hexone #6 AT SITE. Not recorded in log book.

Hexone #7 - Lab number E 1774

Discussed SITE LOG SHEET page 8

Increased Hot oil to 265°F (1° up)

1710 Cut back to 264 (high pressure #1 Vessel)

1720 Checked rate is .78 gpm - RG Cowan

7-17-90

1800 Changed the high level set point on the alarm for Product level to 35 gal. This change will be lost when we restart. RGC

1920 Set temp back up to 265 and cut N_2 purge pressure back to .22 "H_2O RGC

2030 Small oil leak on the 3" hot oil valve - Need to check when we are Down. No repair to hot valves at

2040 set temp to 266°F ~~[illegible]~~

2100 adj N_2 Purge Down to .22 on magnehelic ~~[illegible]~~

Samples # 10 & 10 S taken see table on pg 11 ~~[illegible]~~

2125 Took Samples 2, 4, 6, 10, 10S to 222-S LAB. LAB WORKERS WANTED TO KNOW why we were bringing samples to lab without a proper RSA form (Request for special analysis) this needs to be straightened out on day shift.

2200 The Data logger program stopped - Why fault I was trying to find out why we only got a log every 20 min not 10 - Disk was OK. It has been running OK and is back to every 10 minutes but lower drive seems to be giving trouble off now RGC

2200 Got the results from samples #5 + 8 RGC

#5 is 1530 pCi/ml
#6 is 1520 pCi/ml

2300 adj N_2 Purge Down to .22 from .28 on magnehelix ~~[illegible]~~

KGrat QRU

GRAVEYARD 23:30 7/17/90 - 05:00 7/18/90

0025 - Switch N_2 tanks Dewar No 2 is being used Ellen Magnahelic is reading .2 in. of H_2O
* NEED TO HAVE OTHER DEWAR CHANGED OUT ON DAYS

0037 Rail tank Car check was performed by Grave Yards only PIC.

MIDNITE: The data logger printouts have mysteriously reverted back to every 20 minutes, and are not showing the previous readings. I think the computer misses Cowan.

03:08 initial 141: 19,241
current 141: 16,973
2268 gallon pumped
- 500 approx system hold up
1,768 gallon in rail car

$\frac{2268 \text{ gall}}{40 \text{ hr}} = 0.95$ gpm

05:00 THE COMPUTER LOCKED UP AT 04.15. ALL ALARM LIGHTS WERE ON AND THE DATA LOGGER QUIT SCANNING. WE WENT THRU A RESTART (SHUT OFF POWER & FOLLOW INSTRUCTIONS) AND RECEIVED AN ERROR MESSAGE WHEN THE DATA DISC WAS BEING READ (UPPER RIGHT DRIVE). AFTER SEVERAL FAILED RESTARTS WE WOKE MR COWAN UP (04:30) (SORRY DICK) DECIDED THAT THE ERROR WAS IN THE DATA DISC AND CHANGED IT OUT WITH ONE FROM THE LOWER DRIVES (LOWER DRIVE UNIT IS OFF), AND REBOOTED. EVERYTHING IS NOW WORKING.

IF PROBLEM RE-OCCURS PULL THE DATA DISC FROM UPPER RIGHT HAND DR AND REPLACE IT WITH A ~~THE~~ NEW DISC FROM THE BOX (DISC IS LABELED "NEW BOX IS ON UPPER PRINTER. THEN SHUT THE POWER OFF AND RESTART

IF THIS FAILS MORE DISCS WILL HAVE TO BE FORMATTED

THE CAUSE OF THE COMPUTER LOCK-UP WAS PROBABLY OVERHEATING, THE AIR CONDITIONER WAS OFF AT THE TIME. ALSO, TO AVOID WAKING DICK UP AGAIN, FAILURES ARE ONLY ALLOWED AT 9AM OR 9PM.

THANKS AGAIN DICK, HOPE WE DONT HAVE TO WAKE YOU AGAIN. RSP

JOHN & DEIRELL: THE DESK IS CLEAN

LJS (3-2435 DOT) called with
RESULTS of Sample E 1774
→ 1.53×10^{-7} pico curies/ml. Bob.

7:15 PUMP OUT TOTAL TO DATE: 2,535 GALLON
PUMPOUT THIS SHIFT : 473 GALLON

7:25 SHIFT TOTAL PUMPOUT 483 GALLON
(ANY CHALLENGES FROM OTHER SHIFTS?)

Day Shift - 0730 7/18/90

Jillbrook Trembath 9:30 We went to draw off two Analytical samples. We encountered problems with the electrical relay for the Transfer pump. We called an electrician, but got the relay to work by jarring the panel box. We did take down the information on the relay when the electrician come. We called Bob Cash in 300 Area to see if spares are available in case of failure

ACROMAG Inc. Relays DorS? not E
Transfer pump is Model 372-E-1
Feed Pump is Model 874-S-1

9:40 We were able to get the Samples taken; Submitted lab sample E1776, and kept the other as a Archive sample.

10:25 • Contacted Dave Dodd on Tritium samples. Lab ran sample thru silica gel to remove any water present. This did not change tritium results in hexane.
• Contacted J. Jesky (3-4935) at 100K lab. The two samples tested by liquid scintillation indicated 1,025 and 1,027 pCi/ml respectively.
Total alpha was 0 ± 0.5 pCi/ml and 0 ± 0.3 pCi/ml
Total beta was 0 ± 1.6 pCi/ml and 0 ± 1.7 pCi/ml

NOTE: These SAMPLES WERE SWITCHED TO DETERMINE EFFECT OF TRITIUM ESP 7/19/90

10:40 - We raised the Temperature Setting on the Oil by 1 degree from 266 to 267. The vessel pressure was 29.7.

> 10:33 - The Data Logger locked up - We'll restart with the last Disk

10 charge ii
xJAD ii

Sample log Results

Lab Sample No	Hexone No.	Date	Results
N/11 (E1769)	Hexone 1	7/16/90	3019.92 pCi/ml ~~3000 pCi/ml~~ 6000 pCi/gmd
N/A (E1770)	Hexone 3	7/17/90	Actual 4827.39 pCi/ml planed in 4500 pCi/ml
E1772	Hexone 5	7/17/90	1530 pCi/ml
	Hexone 6	7/17/90	1775 pCi/ml
E1773	Hexone 8	7/17/90	1520 pCi/ml
E1774	Hexone 10	7/18/90	1530 pCi/ml.
at 100KE	Hexone 7	7/18/90	~~1025~~ pCi/ml
at 100KE	Hexone 9	7/18/90	~~1027~~ pCi/ml
E1772	Hexone 5	7/18/90 - 10:45AM	1.53E3 piCi/ml
E1773	Hexone 8	7/18/90 "	1.52E3 piCi/ml
E1774	Hexone 10	7/18/90 "	1.53E3 piCi/ml
E1787	Hexone 13	7/19/90	1.50 x 10³ piCi/ml
PNL	Hexone 15	7/18/90 (PNL)	6,238 pCi/ml Tα = less than 0.3 pCi/m Tβ = less than 1.3 pCi/m
E1776	Hexone 12	7/19/90	1527 piCi/ml - Total Aci
E1777	Hexone 2	7/19/90	1325 piCi/ml Total Activ
E1778	Hexone 4	7/19/90	1540 piCi/ml Total Activ
E1779	Hexone 6	7/19/90	1529 piCi/ml Total Activ
E1780	Hexone 105	7/19/90	1508 piCi/ml Total Activ
E1781	'89 #196	7/19/90	< 5 piCi/ml Total Activ

Sample log

Sample No		Time	Date	Type of Sample	
Hexone #1		~~7/16/90~~ ~1600	7/16/90	Total Activity	
Hexone #2		0930	7/17/90	~~Total Activity~~ (not submitted	
		20% water, ~80% organic			
Hexone #3		0930	7/17/90	Total Activity	
		All Organic except 1 drop water			
Hexone #4		1130	7/17/90	Archive Sample	
Hexone #5 Lab Sample (E1772)		1130	7/17/90	Total Activity	
Hexone #6	Lab No	1415	7/17/90	Archive Sample	
Hexone #7		1415	7/17/90	Total Act.	100K Area Lab
Hexone #8	E1773	1415	7/17/90	" "	222-S
Hexone #9		1415	7/17/90	" "	100K Area Lab
Hexone #10	E1774	~~2102~~ 2102	7/17/90	" "	
~~Hexone #~~ 10S	Special	~~2102~~ 2102	7/17/90	~~Archive Sample~~ Special	
Hexone #11		9:30	7/18/90	Archive	
Hexone #12	E1776	9:30	7/18/90	Total Activity	
Hexone #13	E1787	14 45	7/18/90	Archive	
Hexone #14		14:45	7/18/90	Total Activity	-2225 ~~-300 Area~~
Hexone #15		14:45	7/18/90	Total Activity - 300 Area	
Hexone #16		8:10	7/19/90	Archive	
Hexone #17	E1788	8:10	7/19/90	222-S	

Sample Log Results

Lab Sample No	Hexone No.	Analysis Date	Results
E 1782	'90/#016	7/19/90	< 5 pCi/ml Total-Activity
E 1787	Hexone 13	7/19/90	1500 pCi/ml Total Activity
E 1788	Hexone 17	7/23/90	1763. pCi/gm
E 1790	Hexone 19	7/23/90	1540. pCi/gm
E 1794	Hexone 21	7/23/90	1550 pCi/gm
E 1813	HEXONE 23	7/30/90	4.97 E3 pci/ml. H_3 4,970 pci/ml (so dat cum uac
E 1966 FEED	Hexone 28 (Feed Sample)	8/17/90	T_B-liq 7.88 μCi/l $A_{T_{liq}}$ 2.08 x10⁻, GEA 8.63x10⁻¹ μCi/l Act_{Total} 8.9x10³ pC
E 1909 FEED	Hexone 25 (Feed Sample)	8/17/90	T_B 5.65 μCi/L A_T < 1.4x10⁻⁴ μCi, GEA < 4.42x10⁻³ μCi/L Act_{total} 7.55x10² pC
E 1967 PRODUCT	Hexone 30 (Product Sample)	8/17/90	T_B 6.7x10⁻⁴ μCi/L A_T < 1.4x10⁻⁴ μCi/L GEA < 4.37x10⁻³ μCi/L Act_{total} 1.91x10³ p Tritium 3,482 pCi/ml
E 1910 PRODUCT	Hexone 26 (Product Sample)	8/17/90	T_B < 4.38x10⁻⁴ μCi/L A_T < 1.6x10⁻⁴ μC GEA < 1.19 x10⁻³ μCi/L Act_{total} = 1.12x10³ pC Tritium 4,344 pCi/ml
E 2029	Hexone 32	8/23/90	Tritium 4727 pCi/ml, Total Act. 1227 p
E 2030	Hexone 33	8/23/90	Total Act. < 50 pC

FOR SAMPLE STATUS CALL DICK VOGEL 3-3683

Sample Log

Sample No	Lab No	Date	Time	Type of Sample
Hexone 18		7/20/90	08:30	Archive
Hexone 19	E 1790	7/20/90	08:30	Total Activity
Hexone 20		7/20/90	11:15 AM	Archive
Hexone 21	E 1794	7/20/90	11:15	Tritium & Total Acti
— Samples were placed in a different type glass bottle sample —				
Hexone 22		7-27-90	0915	Archive
Hexone 23	E 1813	7-27-90	0915	Total Activity Tritium
Hexone 24	E 1816	7-27-90	1315	Dave Odd Exchange Test
Taking @ Feed Pump — Hexone 25	E 1909	8-8-90	0915	GEA, Total Alpha, Beta, Acti
Hexone 25A	INLET	~~8-8~~ 8-8-90	0915	Archive
Hexone 25B		8-8-90	0915	Archive
Taking @ Thru-Feed pump — Hexone 26	E1910	8-8-90	0915	GEA, Total Alpha, Beta, Activity
Hexone 26A	PRODUCT	8-8-90	0915	Archive
142-S Feed Samples — Hexone 27		8-14-90	1000	Archive
Hexone 28	E 1966	8-14-90	1000	GEA, Total Alpha, Beta, Act
142-S Product Samples — Hexone 29		8-14-90	1000	Archive
Hexone 30	E 1967	8-14-90	1000	Total Activity
Hexone 31	Archive	8-22-90	13:30	
Hexone 32	E 2029	8-22-90	13:30	Total Activity, Tritium
Hexone 33 (Rain Water)	E 2030	8-22-90	13:30	Total Activity

Log. -

Dayshift - 7/18/90

11:18 - Data logger is running; Time interval is 10 min.

12:30 - We are having problems with both the Feed pump and Transfer pumps. We keep having to switch the power on and off to break the electrical lock or whatever.

3:15 - Prepared a shipment to Malin Weiler. It is a limited Quantity shipment. Put sticker inside on ice cream carton, put in some absorbent, and then got release sticker for outer can. We took sample with us to 300 Area.

Swing Shift 1530 - 2400 RGC, JWB, DDP

1810 Had to start transfer pump twice by Rapping Relay panel at target site. DDP

1815 Continuing to work on computer / data logger. locked up and would not restart on lower drive. Got it going again but plan to alter the program to avoid possible problems.

1900 adj N_2 Purge rate Down from .28 to .22 on mag. DDP

2100 adj N_2 Purge rate Down from .28 to .22 on mag. DDP

2230 Found contaminated mouse droppings in fat coat laundry bag RPT. Removed ~ 1,000 CPM DDP

2245 Checked out program and made a small change to reduce the number of disk transfers per log. Have one more change to make when its down but not going to fix what's running. If it stops always check the printer it may just be jammed.

Rlwd. JRW RGC

7/18/90 Wednesday

2245 The transfer pump continues to stick — a sharp rap on the panel always starts it and sometimes it starts by its self. We need to buy ~~or~~ find a spare unit ASAP & will see what I can find Thursday or friday in the 300 area.

If we can not get ~~it~~ the transfer to start, ~~[illegible]~~ RGC have the electrian jumper out the high level relay contacts and just switch it on when the level comes up to high alarm.

DPW RGC RGC

Graveyard Shift 7/19/90 THURSDAY MORN.

01:00 - CONDUCTED WALK DOWN OF TANK CARS; STILL NO SIGN OF ANY LEAKAGE.
- LOWERED N_2 DEMAND REGULATOR FROM 0.25 TO 0.22.
- THOUGHTS ON SAMPLING.

RGC

WE SHOULD OBTAIN SOME FEED SAMPLES FROM 141. SINCE WE DON'T WANT TO OPEN THE 4" TANK RISER (AND LOOSE INERT ATMOSPHERE), WE'LL NEED TO OBTAIN SAMPLE AT PUMP. ONE POSSIBILITY IS TO SHUT OFF FEED PUMP AND OPEN STRAINER TOP, MAY BE ABLE TO DIP SAMPLE FROM THE STRAINER HOUSING. THE OTHER WAY WOULD BE TO SHUT OFF FEED PUMP, AND DISCONN THE PUMP OUTLET TO VESSEL #1, VALVE DOWN PUMP, THEN TURN ON PUMP AND FILL SAMPLE BOTTLE. THE OPTION WOULD BEST BE DONE WHEN SYSTEM IS COOL. ANOTHER POSSIBILITY WOULD BE TO OPEN PLUG ON PUMP RECIRCULATION TEE-VALVE; SHUT DOWN PUMP, TURN T-VALVE AND BLOW SAMPLE OUT USING THE VESSEL PRESSURE. (NOT SURE THIS WAY WOULD WORK).

PAV

ANY ONE OF THESE WOULD REQUIRE FITTER & A JSA.

L43 CALLED — E 1787 → 1.50×10^{1} pici/ml.

07:00 - HAVE BEEN ADJUSTING N_2 DEMAND REGULATOR DOWNWARDS DURING THE NIGHTS (I.E. PRESSURE CLIMBS). AFTER SUNRISE THE PRESSURE DROPS AND PRESSURE REGULATOR IS ADJUSTED UPWARDS. KEEP AN EYE ON IT, ESPECIALLY EARLY IN THE MORNING (THIS IS WHEN IT COULD DROP TOO LOW).

RGC

[illegible]

RGL NEEDLE JUST PRESSES BACK ON. WE LEFT COVER GLASS ON LOOSELY SO THAT IT WOULD BE EASY TO FIX NEXT TIME.

RGL OVERALL, A QUITE SHIFT., EVERYTHING RUNNING WELL. MUST BE A WELL ENGINEERED SYSTEM! WE PROBABLY DON'T NEED ALL THREE PEOPLE ON GRAVEYARD. TONIGHT WAS LAST NIGHT/SHIFT FOR FULL TIME HPT SUPPORT

NO HEXONE VAPORS NOTICED ON SHIFT

07:20 DKL; THO ONES FOR YOU.
THE DATA LOGGER WAS RUNNING WELL EXCEPT ALL OF THE ALARM LIGHTS WERE ON, EXCEPT FOR #1 (WHICH WAS ALARMING, AND SHOULD HAVE BEEN ON). I
RGL THE COMPUTER WAS NOT LOCKED UP, AS IT WAS STILL SCANNING AND PRINTING. I REBOOTED WITH NEW "NEW" DISC, AND PART WAY THRU THE REBOOT THE COMPUTER LOCKED UP. (THE SCREEN SAID TO WAIT, THE DISC DRIVES HAD BEEN TRANSFERRING DATA, AND STOPPED, DATA LOGGER WAS SCANNING) REBOOTED ONCE MORE AND EVERYTHING WAS FINE. DISREGARD ABOVE NOTE ABOUT EVERYTHING RUNNING WELL.

ALSO NOTE THAT WE NEED TO RECEIVE NEW N_2 DEWAR TODAY OR WE'LL HAVE TO SHUT DOWN @ ½ BOTTLE, ON SWING.

Day Shift - 7/19/90 - WD Greenhalgh, Olaf Rasmussen, & Geo. McDonald

RGL 08:10 Took 2 product samples Hexone 16 and 17. Hexone #16 was archived and Hexone 17 (Lab # E1788) was submitted to 222-5 for Analysis.

RGL 08:40 Turned oil Temp up 1° from 266 to 267. The demister pressure was 27.

RGL 09:00 The data logger got hung up (locked up), but a touch of the paper advance unlocked it. Apparently it was strictly a paper lockup.

RGL Note We keep using the special pump ~~tool~~ to get the Transfer pump to work,

RGC 13:40 - The Transfer pump keeps cycling off. We Taped up some shading material for the panel and turned the oil heater Temp. down 1 degree from 268 to 267. Vessel press 34.6.

RGC 14:00 - Computer locked up (not paper lockup). We had to change out the disk and reboot. One disk did not work right so used a different one.

RGC 14:20 - Data logger OK + operating ..

RGC 15:00 - Computer locked up again and disk was changed.

15:10 - Malin Weiler @ PNL reported Total Alpha =<0.3 pCi/m
OMc Total Beta =<1.3 pCi/m
Tritium = 6,238 pC

- Rich Weiss reported total activity on original 276-5-141 composite as ~700 pCi/ml !

RGC - Talked to NSSI, mixed waste handler in Houston, TX. Said they use 10 CFR 20 Appendix B for release which calls for Tritium < 3,000 pCi/ml

DDP Swing Shift 1530 - 2400 RGC, JWB, DDP

I increased Set point on oil heater to 268 (+1°)

Restarted the Computer after finding a problem with the printer - set now to print log on alarm printer if lower one fails. RG Cowan

1930 Computer is now doing OK but prints a lot of logs on alarm printer - Printer was problem - Note: procedure for data Logger only allow 25 to start

RMM 2/92

7/19/90 Thursday

1930 Autotransfer only worked twice Normally have to hit it to start PO for new unit written.
RGC

2200 Very steady operation - May need to to hourly or better inspections to keep alert

2230 Fitter never showed to recharge eye wash stations And connect full N_2 Dewar. needs to be resch on Days to complete Job.

2300 Total Hepone out of 141 now over 5000 gal. we have processed over 20 distillation vessels full of solvent and no significant change in heat transfer.

GRAVEYARD THURS NIGHT → FRI MORN 20 JULY 90

03:12 RAISED THE HOT OIL TEMP A DEGREE AT ≈ 01:00, AND HAVE BEEN ABLE TO MAINTAIN A VESSEL PRESSURE OF 31-32 INCHES. THE HOT OIL TEMP HAS BEEN SLOWLY INCREASING (SET POINT NOW 269, WAS 260 A FEW DAYS AGO). THE ΔT ACROSS THE HOT OIL IN, OUT REMAINS CONSTANT AT 9°. THE SOLUTION TEMP IN THE VESSEL HAS LIKEWISE INCREASED A FEW DEGREES.

WHAT THIS INDICATES TO ME IS THAT WE ARE NOT FOULING THE OUTSIDE OF THE HOT OIL TUBES (THE ΔT IS UNCHANGED). THEY AREN'T FOULING BECAUSE THEY'RE IMMERSED IN A BOILING SOLVENT. THE BY-PRODUCTS (THAT CAUSE FOULING) ARE MOST LIKELY STAYING IN SOLUTION AND BEING CONCENTRATED. THIS WOULD INCREASE THE BOILING POINT OF THE SOLUTION

(JUST SOME THOUGHTS / HALUCINATIONS FROM A SLEEPLESS GRAVEYARD ENGINEER)

06:30 FURTHER THOUGHTS & HALUCINATIONS ON THE SAME SUBJECT

BEFORE RAISING THE TEMP (AS MENTIONED ABOVE) AT 268° WE HAD PUMPED 116 gallon IN 2 HR. (58.0 GPH). TO DETERMINE HOW MUCH FASTER WE WERE PROCESSING AT A HIGHER TEMP 269° WE MEASURED 83 gallon in 1.7 hr (49.7 GPH). TO VERIFY THIS UNLIKELY TREND, WE DROPPED THE TEMP BACK TO 268° AND PUMPED

101 gal in 2 Hr (50.5 GPH). CONCLUSION: PUSHING THE TEMPERATURE UP TO THE MAX OPERATING PRESSURES DOES NOT SIGNIFICANTLY CHANGE THE PRODUCTION RATE. IN FACT THE INCREASED PRESSURE IN THE VAPOR SPACE MAY ONLY RAISE THE SOLUTION BOILING POINT AND DECREASE PRODUCTION RATE. COMMENTS?

TO SWING SHIFT ON FRIDAY; PLEASE EMPTY THE TRASH CANS BEFORE CLOSING UP. NO USE IN ATTRACTING MICE OVER THE WEEKEND. THANKS.

TO DAY SHIFT: WHAT HAVE THE INDUSTRIAL HYGIENE SURVEYS BEEN FINDING ON THEIR DAILY INSPECTIONS?

7:35AM Day Shift - 7/20/90 - D. Greenhalgh, Olaf R. & Geo McDonald.

08:30 Took 2 hexone product samples - Hexone 18 & 19, Hexone 19 (E1790) was Taken To 222-S for Analysis.

11:15 Took 2 hexone product samples - Hexone 20 and 21. Hexone 21 (E 1794) was taken To 222-S for Analysis. These samples were placed in a different (non certified) lab sample bottle rather than The ICI certified bottles.

13:30 Geo Wrapped reflective paper around the Magnihelic system to keep it cooler.

13:40 Ordered 16 ea Flammable Liquid Placards for Railroad Cars - Transportation pk 6-2768.

DPL
JDL RGC

Swing Shift 1530-1200 RGC, DWB, DOP.

1630 Set up N_2 purge to .23 inches, dropped again as it cooled? RG Cowan

~~The~~ Raised temp 1° but had a high pressure - cut back, running at steady 0.96 gpm by pump to

1 M

7/20/90 Friday
Swing – cont

1800 The valve on the hot oil from the Hydrotherm is still leaking a little at the stem packing. Wiped up the oil and placed a paper wipe under the valve to determine the leak rate. RG Cowan

1930 Still no ~~[illegible]~~ detectable hydrocarbon vapor around the site; working as designed.
RGC

2138 Check on drop out in 141 Tank level VS the processing rate from pump tank fill rates

T-141 @	20:57	=	12969
@	9:07		13636
	11:50		-667 gal

ave. rate = 667 gal / 710 mins = 0.939 gpm

ave of 20 fill rates on two shifts = 0.949 gpm

These are within 1% which is the limit of accuracy for 141 level. Anything better than ~10% is luck.
RGC

2200 Reduced OIL HTR SET POINT TO 68°F
SHUT OFF FEED PUMP
2215 Shutoff OIL HEATER
2220 SHUTOFF TRANSFER PUMP POWER
SHUT OFF CONDENSER FANS
SHUT OFF RECIRC (OIL) PUMP
MAIN POWER LEFT ON FOR SUMP PUMP
2300 FINAL WALK AROUND OF AREA NO PROBLEMS NOTED.

ERW

2345 Changed Program on data logger to allow skipping log printouts. Log is running and just hit return and reset skip as you feel best - I suggest all until steady state

7/21/90 Saturday

9pm Checked nitrogen dewar #1 - read ~ 0.2 (20%).
System pressure 0.2" water.
OPR Left #1 dewar valved in. Some ice on #1 dewar.
Tightened valve.
#2 dewar venting audibly, but no ice.

7-22-90 Obtain Hexane gal and total

7-22-90 Checked nitrogen. - System pressure 0.12" water.
OPR #1 dewar reading 45 psi. Should still have ~10% liquid volume left.
6:30pm Valved in #2 dewar, but system pressure went way up 0.45-1.0".
Sunday Tried to adjust down without success.* Strong nitrogen flow at demand regulator.
Valved #2 out, and valved #1 back in. Set pressure to 0.16".
RGG Will let PAV adjust system pressure with #2 dewar before end of c
Even if #1 runs out before graveyard shift comes on (unlikely) the system will still be inert, since it just had a good shot of N_2 and good pressure
* could not get system pressure below ~0.45"
ER

7/23/90 GRAVEYARD PAV/RED/RICHARD

- Started feed pump per procedure and no ΔP. Apparently we lost our prime. Could not get HPT from the labs, so went to AW and tracked down the west area Tank Farm "Rover". (If you ever need to track him down, phone is 3-2731 or Car-46 on everyone else's radios but ours) (call tank farm office in East 3-1600)

- Followed procedure for priming pump, pressurized inlet started pump up, then lost suction and shut pump down. On second try we pressurized the line (pressure goes high, should be controlled by gate valve at rotometer) and. and started pump. (Step (V. 11.f)) as pump was running it lost suction and started making loud hammering sound. We shut off pump.

After shuting off both inlet & outlet of pump were pressurized (regardless of valve #9 setting). Motor now hums but we get no suction on the pump inlet, only positive press (probably still from pressurized. Also the same pos. pressure on the outlet side. Sounds like pump impeller is displaced and wedged so that it blocks outlet.

RGC
Read the log pos.

Also, Nitrogen purge was lost between 00:40 and 00:51. We noticed on the 04:20 report that the product tank volume had gone to near zero immediatly checked N_2 pressures all (manifold & tank 142) were at zero Then valved in the full dewar (that was supposed to have been on all weekend). So N_2 purge was only lost for 30 to 40 minutes Should be no problem, Especially since we were not operating due to pump

~~Water priming raised tank 141~~ After restoring N_2 purge Tank 141 and 142 were reading 138 & 128 gallons higher (respectively) than at the 23:53 report from Friday swing. Product tank also reading slightly higher at 30.6 compared to 29.2. Because of this cannot determine how much water was added to 141 during priming, expect that volume would only be 10 to 20 gallons.

THE TWO RULES OF CHEMICAL PROCESSING.
1) "IF IT AIN'T BROKE, DON'T FIX IT"
2) "IF ITS RUNNING GOOD, DON'T SHUT HER DOWN"

-23-90
3:15 PM
Cosh

MAINTENANCE SUPPORT WORKING ON PUMP CHANGEOUT

NAME	PR#	FUNCTION	TIME IN	TIME OUT
Gary Altmiller	66619	NPO	1300	1505
D. Roneli	69729	MAINT.	1300	15:53
Thomas Hyde	64760	Main.	1300	15:00
William [illegible]	67920	NPO	1300	1505

SMR Replaced feed pump (pump & magnetic shaft) with parts from spare pump.
Found screen full of "sand" – rust & scale. Pump had rust & scale sand also. Gear teeth were broken off. Debris plugged pump discharge.

[illegible] Pump started c-down at 7:20 p.m. Plan to run thru Fri. & then shut down

on days tomorrow to re-inspect and/or clean screen. Need to add additional screening capacity.
Theory: backflush into tank may suspend debris in tank, which pump then picks. May consider deleting backflushes into tank when priming the pump.

Truck picked up #2 dewar this pm

7-23-90 Swing RJC DDP JWB

16:00 Continuing heatup of oil system
16:56 XFER PUMP DIDN'T COME ON - BANGED RELAY CABINE - OK Now TRANSFERING

1800 Trying to stabilize boiling rate. Some wet still in #1 and more in #2 vessel. Setpoint now 247°F.

1945 Water pretty much out of both vessels BP #1 241°F BP Vessel #2 234°F setpoint - Hot Oil 264°F

2120 Reached equil on hexane. BP #1 245°F BP #2 242°F. Transfer pump relay acting up more. Had to pound on cabinet to or it

2115 Going up 2° more in setpoint - now 272°F

We're getting different numbers now than last week - But it's cooler outside 77°F W 20 mph wind from WNW. Last week ~~7-2~~ 7-19 at 2118 outside temp was 89°F - probably little wind.

23:00 RSP THE THERMOCOUPLE ON THE HOT OIL INLET TO VESSEL #1 IS INTERMITTANTLY OPEN, AND GIVING LOW/HI TEMP ALARMS. HAVE A TEMPORAR TC IN PLACE BUT WE CANNOT PUSH IT PAST THE FLEX FLANGE AND KEEP IT NEXT TO THE PIPE, SO IT READS LOWER. FOR A TRUE ΔT ACROSS THE COILS USE THE HOT OIL INLET TO VESSEL #2. IT SHOULD BE IDENTICAL

7/23 – 24 /90 GRAVEYARD

ON SUNDAY NIGHT WHEN WE WENT TO START UP, SINCE THERE HADN'T BEEN ANY TRANSFERS FOR 2 DAYS A GOOD STABLE VALUE FOR THE TANK CAR WAS OBTAINED 6,970 gallons in the Tank car as of Monday morn

OPERATING SPECS HAVE A MAXIMUM TANK CAR VOLUME OF 9,800 gallon AT 0.96 GPM; THIS WOULD GIVE US ≈48 HRS OF OPERATING BEFORE WE HAVE TO CHANGE TANK CARS (WEDNESDAY DAY SHIFT PROBABLY)

~~THE 6,970 gallon in the Tank car. compares well with the 7,450 gallon shown as 141 pump down. This gives us a 480 gallon holdup, 29 gallons were in the product tank, so there would be 451 gallons in the vessels (225 gall/vessel) & I had calculated a vessel hold-up of 250 gallon earlier so everything seems to be reasonable.~~

not true. see note below

WHEN THE PROCESS IS SHUT DOWN FOR ANY TIME, PLEASE NOTE THE TANK CAR VOLUME FROM THE DATA LOGGER.

REV. 1) The 6970 gallon in the tank car does not compare well with the 6450 gallon shown as 141 pump down (ref. 7/22/90; 23:50). If there were 500 gallon holdup, then either a) there should only be 6,000 gallon in the tank car or b) we've added 1020 gallon of water to ~~the~~ 141 tank when priming

ANYTIME WE ADD WATER TO THE SYSTEM FOR PRIMING/FLUSH/ETC. RECORD THE START/END READINGS FOR THE FLOW TOTALIZER. IN THE LO SO THAT WE CAN DETERMINE A MASS (VOLUME) BALANCE.

06:00 BIG THUNDERSTORM CAME THRU @05:25 WITH THE FOLLOWING RESULTS

1) AS LIGHTNING WAS STRIKING NEARBY, AN ARC SPARKED FROM THE COMPUTER, ALL ALARMS ON, COMPUTER DOWN. WE PUT IN CLEAN "NEW" DISC AND REBOOTED.
2) UTILITIES NEVER GROUNDED TRAILER. ELECTRICAL, WILL CALL BOB EGGE IMMEDIATELY THIS MORNING.
3) ONCE COMPUTER REBOOTED LIQUID LEVELS IN 141 & 142 WERE READING 243 & 294 gallons higher (respectively). This is similar to MONDAY WHEN WE CHANGED OUT N_2 DEWAR AND THE TANK LEVELS BOTH INCREASED BY 150 gallon. JUST TO REMIND US THESE VALUES ARE NOT ABSOLUTE.
4) THE DP ON #2 DEMISTER DOUBLED TO 3.5 (from 1.7)
5) WE OPENED DRAIN LINE VALVE FROM DISTILLATION CATCH PAN TO PREVENT FEED PUMP SHUT-DOWN. (RED BRAND TORRENTIAL RAINS & RAGING CURRENTS) WE'LL NEED TO SHIP THE HALIDE AFTER THIS

RAINS HAVE PASSED (TO PROVIDE LEAKAGE CONTROL)

6) TRANSFER PUMP IS GOING CRITICAL. WE COULDN'T GET THE RELAY TO CLICK IN. HARD RAPS MADE ~~ALL THE~~ THE FEED PUMP LIGHT BLINK ALSO THE OIL HEATER LIGHT (ON THE OTHER PANEL) WOULD ALSO FLICKER. ABOUT ONE MINUTE OF LIGHT RAPPING GOT THE RELAY TO CLICK IN. DO NOT HIT THE PANEL HARD! I THINK WE ARE ENDANGERING OTHER EQUIPMENT.
IF DAY SHIFT CAN GET AN ELECTRICIAN TO JUMPER AROUND THE RELAY UNTIL NEW PART ARRIVES, THEN WE CAN MANUALLY TURN PUMP ON-OFF.

7) WE WERE RUNNING ALONG PEACEFULLY AT 273° and VESSEL PRESSURE of 30" BEFORE STORM, AFTER STORM VESSEL #1 PRESSURE KEPT CLIMBING AND WE HAD TO DROP TEMP TO 270° TO CONTROL TRIED TO RAISE IT AND PRESSURES ROSE AGAIN. WE'RE KEEPING IT AT 270° FOR NOW.

PRETTY DARN EXCITING STORM!!

06:30 ALL THE ELECTRICAL BOX BASHING HAS TAKEN ITS TOLL. WHILE RAPPING THE BOX IN HOPES OF TRANSFERRING PRODUCT WE LOST THE FEED PUMP AND HOT OIL SYSTEM. FIGURING THERE MUST BE LOOSE WIRES AWRY IN THE PANEL, WE PROCEEDED TO SHUTDOWN. WE WILL GET AN ELECTRICIAN ON DAY SHIFT TO BYPASS THE TRANSFER PUMP RELAY AND TROUBLE SHOOT THE OTHER (FEED/HEATER) CIRCUITS. WHO SAYS COLD FUSION IS EASY.

Day Shift - 7/24/90 - [illegible] Greenlaugh, Geo. McDonald, Bob Parkin

09:30 - The electrician checked out the system and found we had lost the 3rd phase - Probably due to the storm.

10:55 - Computer locked up and it was rebooted with a new disk.

13:45 Getting ready to open Strainer Body - to check for debris PF + NPO + HPT. Need to get a supply of green Solvex gloves - Hexane compatible. ~~Tank~~ PF + HPT + blend NPO as yesterday. Also working on recal of DP cells. Apparently resistors got zapped by lightening this morning.

Gal
PM

7/24/90

1420
~~1415~~
JSR

Delivered Liq N_2 Dewar. Frost spot ~3" x
near bottom. STORES felt it has a cracked;

300
ORM

Wilbur called with the stores # for Solvex Gloves: 37-3002-190 for size
-210 for size

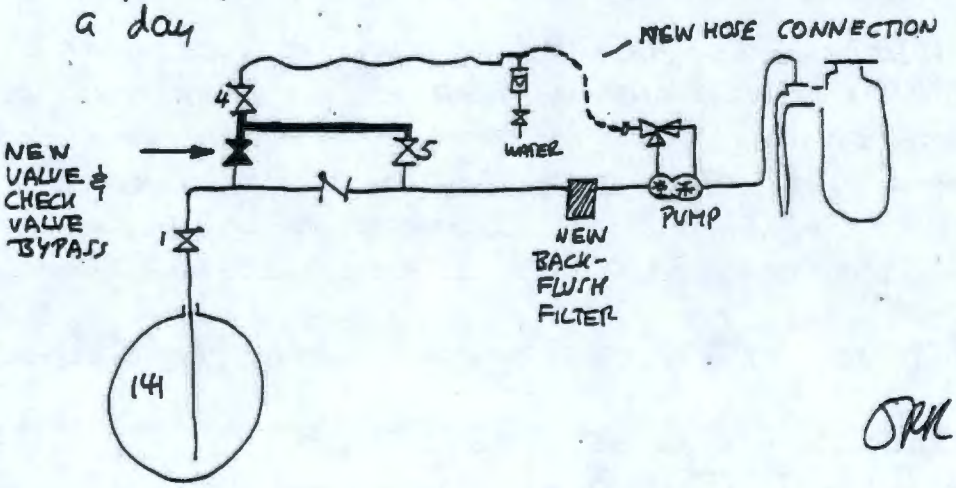
(not ordered)

Issued JSA for filter cleaning (see JSA book)
Issued: J-1 and J-4 forms for filter cleaning
" for routine filter and maintenance support
" for electrical & instrument support

Checked pump intake screen. It was about ~1/4 full of rust particles.
Cleaned and replaced screen. Re-filled pipe with water.

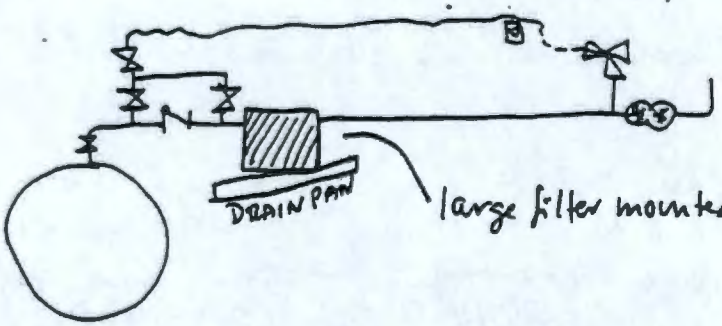
Power problems still in resolution (no A-phase).

Major effort to clean filter – need a filter we can back flush once
a day



ORM

or alternate larger filter:



large filter mounted over drain pan on 141-5

7/24/90

627-2720 Bill Heine Phone to upday - RG Cowan
Electrical Problem with 'A phase is most likely in 276-S Building and an entry to the building is needed to check wiring and contactor.

The computer system must have taken an invisible shot last night, 5 of 6 resistor used to convert 4-20 mA to 1-5 Volts are bad

The loss of "A" phase was not at the same time as the lighting - but may have resulted from water leaking into 276-S, this happened once before and damaged the electrical panel

DAY SHIFT WED 25 JULY 90 PAV/GEORGE

MISC. NOTES. ON TRITIUM

FROM RICH WEISS: when we ask for total activity the 222-S lab equipment is not calibrated for tritium and doesnot see the softer betas, therefore our readings are probably about half of the total activity due to tritium (i.e. double our readings) This would explain the difference between 222-S and other labs. He will check into getting us a separate tritium analysis w/our samples. He also believes that the hexane is "activated" not just the water.

From Harry Babad: The alpha hydrogen next to the carbonyl on the hexone molecule could be exchanging with a deuterium to "activate" the hexone. We could determine this by extracting the hexone with a dilute solution of nitric acid and see if we get the hydrogen ion to exchange with the deuterium on the hexone.

NOTES ON SAMPLES.

We're getting a bad rep with the lab folks for not following protocol (i.e. no RSA's at first, rumors of transporting samples in personal vehicles Make sure our samples are properly cleared by HPT, and that lab personnel have proper documentation to track our samples (they get hundreds) and

knowing what they are. (Rev, I think samples should be taken on graveyards now)

12:00 Progress this morning;

1) the trailer Electrical is now grounded, (grounding rod driven in next to util pole - 8 ft long.)

2) All 8 of the resistors on the back of the data logger were replaced. and all pressure dependent readings are now back to pre-storm values. (We have processed 7,038 gallon to date from tank 141 and have 7,706 gallon in the tank car; unusual mass balance)

3) Entered 276-5 and found Electrical breaker OK., they opened the breaker and will Meg the lines in and out of 276 this afternoon we will have to re-enter to energize the breaker.

note: what we need for entering 276-5 is the following
- Asbestos work permit (Mary Armatis supplied)
- Certified Asbestos worker (Mary scheduled / Jim Hodgson is supervisor)
- PAPR (from Hodgson or Personal Protection - Martin O'neil - 34885
- asbestos suit, gloves, boots. (Hodgsons people supply)

Also ~~some~~ respiratory protection had some feedback on how we handled the pump change out. When we open lines that we know could still have heroine in them (like opening pump lines and draining heroine into a catch pan) the face cartridges may not be adequate protection. PLEASE call Martin O'neil 3-4885 and he will come down and sample (if necessary) and determine if face cartridges are adequate or if supplied air is needed. Has here to help us, lets us him.

14:30 Olaf called from Crater Lake. Its Beautiful but cold there. Stated that he thinks we should stay shut down until we get a new pump filter system in place, and not risk destroying another pump As a reminder he stated that once we start 142 the contamination levels will be a lot higher and that doing pump work will be more restricted. He'll call again tommorrow.

7/26/90 Thursday

day shift

1130 Electrical checks out OK and interlocks were calibrated. New relay installed on tank level for pump out - this should eliminate "manual start" RG Cowan

1140 Starting up - !! reduced log skip to 0 RG Cowan

Pump (Feed) started and primed OK without water addition

1250 Rick Weise (3-3869) called: Total activity results are expressed as pCi/ml since the Hexane samples are lighter than water.

RL

14:30 IF HPT SUPPORT IS NEEDED ON BACK SHIFTS CALL
HPT OFFICE (EAST AREA) 3-2526
OR TANK FARM SHIFT OFFICE 3-2689
AND ASK FOR THE "ROVER" HPT. HE'LL BE IN CAR 46

WEST AREA
3-2731 or 2557
DEAN

1600 Tried to heat up too fast and it seemed like the vessel ~~to~~ rolled over the pressure went to 50+ inches and blew the seal loop. Turned off heaters let pressure ~~come~~ down to 0 then turn on the heaters and increased set point to ~10° above the boiling temp and came back up slowly.
RG Cowan

2030 Slowly increase hot oil setpoint. Set point now at 270°F. Pressure at ~26 inches waiting for system to stabilize & optimize.

RL

7/26/96 Thursday

2145. Set the hot oil to 272°F, this should be about steady state - running very well

2215 Autopumpout works every time now 40 → 13 gal on each transfer ~ 3 gal/hr

2RW

Grave Yard - Wilbur Greenhalgh, Red Wood, + Dick Henderson

2330 Vessel pressure is about 33 so Turned Oil heater setting down 1 deg. from 274 to 273 deg.

2355 Vessel pressure is still 33 to 34 so Turned Temperature setting down 1 more deg 273 to 272.

July 27, 1990.

0220 Vessel pressure 28 to 29, Turned Temperature setting up 1 deg to 273 from 272.

0235 Vessel pressure 33, Reduced Temperature settin 1 deg To 272 from 273.

0350 Magnehelic reading 0.3 reduced setting to 0.24.

0700 Magnehelic reading 0.19, raised it to 0.22.

0720 Vessel pressure 28, Turned Temp setting to 273 from 272.

DAY SHIFT - JUST GEORGE & PAU. FRI 27 JULY 90
09:00 PROCESS RUNNING SMOOTH

GEORGE WENT OUT TO STICK THE RR TANK CAR WHILE WE HAD HPT COVERAGE. THERE IS NO AVAILABLE PORT FOR MEASUREMENT. USING THE MANUAL THUMP METHOD (NOT TO BE CONFUSED WITH THE TRANSFER PUMP START UP METHOD) THE LIQUID LEVEL IS APPROX 70 INCHES IN AN 88 INCH dia TANK. I'LL WORK UP THIS VOLUME LATER.

uly 27, 1990

JULIE RING CALLED TO SAY THAT HER BOSS (CINDY BORNEMAN) IS CONCERNED ABOUT THE AFFECT THAT THE HIGHER TRITIUM HAS ON OUR AIR NOTIFICATION LETTER TO THE ~~STATE~~ DEPT OF ECOLOGY JULIE RAN AN AIR-DOS MODEL YESTERDAY AFTERNOON AND USING THE NEW CONCENTRATIONS (5,000 pCi/ml. → 0.36 curies ^{14}C total) WE ARE STILL BELOW THE LIMITS, BUT ARE BORDERLINE. THIER CONCERN IS WITH 14C LEVELS, IF THEY ARE ALSO SIGNIFICANTLY DIFFERENT THEN THE REGULATORY STATUS CHANGES SIGNIFICANTLY. THEY MAY RECOMMEND SHUT DOWN.

WE FOUND A MASK IN THE OUTSIDE SUPPLY CABINET THAT WAS PARTICULATE ONLY. IF YOU'RE USING A MASK MAKE SURE IT IS A GMA-H OR GMC-H THAT IS GOOD FOR BOTH PARTICULATE & ORGANIC VAPORS. IT'S EASY TO SPOT, SINCE THE CARTRIDGE IS A LOT THICKER.

D&D IS OUT FIXING THE ASBESTOS IN 211-S AREA, I CALLED BOB EGGE TO HAVE HIM REQUEST THAT THEY FIX THE SMALL AMOUNT OF ASBESTOS IN 276-S AND GET THE BUILDING RELEASED. IF EGGE REQUESTS THIS WORK, IT WOULD HAVE A BETTER CHANCE OF BEING DONE, SINCE IT WOULD APPEAR TO BE TO BENEFIT D&D NOT HEXONE. RJL

CASH IN AT 12:05 7-27-90 RJL

1210 RJL Error 125 on line 80 1201 computer locked up.

Rebooted and back running on computer @ 12:23 Hrs

1315 RJL Took large bottle sample # HEXONE 24 (E1816) GTMC took sample to Rich Weiss for Dave Dodd's exchange test. Sample size ~1 pt to 1½ qt

Swing Shift 1530 to 2400 RGC, DDP, JNB

1555 Readjusted N_2 purge to 0.20 on magnehelix

2015 Material Balance for Hexone distillation to I flew out the transfer line by using max N_2 flow on purge meter for ~10 minutes

ESP.

July 27, Friday 1990

Material balance, cont.

If all the liquid in the overhead line is blown down to the tank car the level in the tank car should be accurate

Starting in 141	19,300 gal
Water added est.	+ 100 gal
Start total	19,400 gal
amount in #1 & 2 Vessels	- 400 gal
amount in 141	-10,647 gal
Net out	8,353 gal

Rail car reading	8,195 gal
Δ	-158 gal

This seems to be an excellent balance as the amount of water and the hexane in the distillation system ~~on~~ are estimate and could be off by this much.

R L Cowan

≈100 adj N_2 Purge from .28 down to .20 on magnahelic

2235 STARTED SYSTEM SHUTDOWN - REDUCED NTR CONTROL SETTING - Shut off H+R., feed pump

2305 Completed Shutdown - Recirc pump off. Condenser fans off.

~~B~~2330 System down - line to rail car blown out a little more than last time - now balance is not as good

19300 - 10,488 - 400 + 8090 = +322 gal increase

GM RSP

DAY SHIFT MONDAY 30 JULY 90

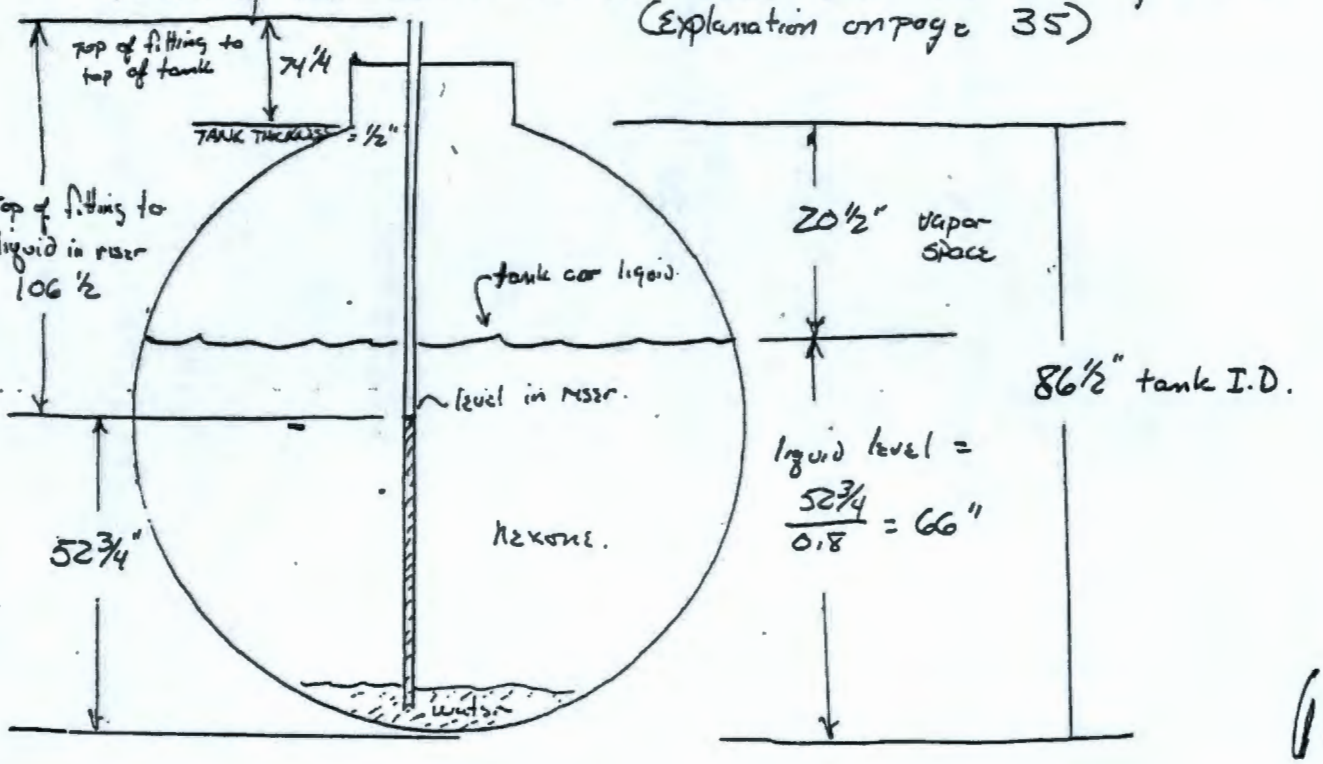
0940 The Liquid Level in Tank Car 3802 is 1¼ inches.

1000 Startup Run D Third Week - 141
Feed pump primed without adding water but took longer than I would like.
RG Cowan

The modifications to the feed piping and the ~~char~~ clean out of the filter were delayed due to problems with the ECN! Will try to reschedual for Tuesday. (or Wednesday)

Feed pump lost its prime shortly after startup. We primed the feed line with enough water to pressurize pump inlet, (used 0.2 gallon water) turned on pump with NO RECIRCULATION, as pump stabilized we adjusted the recirculation flow to minimize suction head on pump inlet.

Concern with RR. tank car liquid level: we got a pipefitter to open the cap on the "tee" fitting on the fill line and placed a measuring rod down the fill tube. with the following results.
(Explanation on page 35)



GM

7-30-90 RJC

RAILCAR. CALIBRATION CURVE. PER. RG COWAN.

0	.5	8	1.0	21	1.5	
60	2.5	84	3.0	110	3.5	1
168	4.5	200	5.0	234	5.5	2
307	6.5	345	7.0	385	7.5	4
469	8.5	513	9.0	557	9.5	6
650	10.5	698	11.0	747	11.5	7
848	12.5	900	13.0	953	13.5	10
1061	14.5	1116	15.0	1172	15.5	12
1286	16.5	1344	17.0	1403	17.5	14
1522	18.5	1583	19.0	1644	19.5	17
1769	20.5	1832	21.0	1895	21.5	19
2024	22.5	2089	23.0	2154	23.5	22
2287	24.5	2353	25.0	2421	25.5	24
2556	26.5	2625	27.0	2693	27.5	27
2832	28.5	2902	29.0	2972	29.5	30
3113	30.5	3184	31.0	3255	31.5	33
3398	32.5	3470	33.0	3542	33.5	36
3687	34.5	3760	35.0	3832	35.5	39
3979	36.5	4052	37.0	4126	37.5	41
4273	38.5	4347	39.0	4421	39.5	44
4569	40.5	4643	41.0	4717	41.5	47
4866	42.5	4940	43.0	5015	43.5	50
5163	44.5	5238	45.0	5312	45.5	53
5461	46.5	5535	47.0	5609	47.5	56
5757	48.5	5831	49.0	5905	49.5	59
6052	50.5	6125	51.0	6198	51.5	62
6344	52.5	6417	53.0	6490	53.5	65
6634	54.5	6706	55.0	6778	55.5	68
6920	56.5	6991	57.0	7062	57.5	71
7202	58.5	7272	59.0	7342	59.5	74
7479	60.5	7548	61.0	7616	61.5	76
7751	62.5	7817	63.0	7884	63.5	79
8015	64.5	8080	65.0	8145	65.5	82
8272	66.5	8335	67.0	8398	67.5	84
8521	68.5	8581	69.0	8641	69.5	87
8760	70.5	8818	71.0	8875	71.5	89
8988	72.5	9043	73.0	9097	73.5	91
9204	74.5	9255	75.0	9306	75.5	935
9405	76.5	9454	77.0	9501	77.5	954
9591	78.5	9635	79.0	9678	79.5	971
9759	80.5	9797	81.0	9834	81.5	987
9904	82.5	9936	83.0	9966	83.5	999
10020	84.5	10044	85.0	10065	85.5	1008

7-30-90 RSL

When we measured with a measuring rod, we still had 33 3/4" to the top of the tank, or only 6,490 gallons in the car way too low. After many rominations it was realized that all the water would be at the bottom of the tank car and that the fill tube would be filled with water not hexane. Therefore a density correction (82 3/4" ÷ 0.8 = 66") was needed. The new ht. corresponds to 8272 gallons. (reasonable). Hammering on the side of the tank gave an approximate ht of 72 inches (8988 gallon) plus or minus a few inches at best.

Bottom line: Specification limit is 9800 gallon / rail car.
Operating procedure is 9,500 " " "

IT IS A LOT EASIER TO ADD MORE TO A TANK CAR THAN IT IS TO PUMP IT OUT, AND TO RESPOND TO A SPECIFICATION VIOLATION.

As of 16:00 we could process about 1,000 more gallons.
Tank 141 had 10,333 gallons at that time.
we need to change to new tank car by 9,333 gallon:

to change cars.
1) shut off transfer pump
2) value product tank to recirculate.
3) Blow down fill line by increasing rail car N_2 @ rotometer for a few minutes — at least 10 minutes. RSL
4) shut off N_2 to rail car.
5) open new rail car vent/fill lines
6) open close old rail car " " "
7) restart N_2, revalve product tank to pump out, turn on transfer pump

On back shifts note my previous entries on obtaining HPT support.

Also Tank Car 3685 fill line inlet valve has been leaking hexane. Pipefitter re-tightened packing bolts Leak is minimal (≈ 1/8 cup or so). The valve will be hard to turn when we need to. RSL good!

7-30-90 Swing Shift 7-30-90 CASH, BIGLIN, PAINE

1600 Still heating up. Oil Temp set at 264°F
Operations normal.

17:40
7-30-90 Setpoint on Oil to 272°F

1830 increase oil htr setpoint to 273°F High wind may be causing cooling effect.

1900 Wind dropping off some - nastly front will see if pressure increases and if we have to drop setpoint ΔP = 27.5 psi

1930 ΔP = 27.3 psi holding good; still @ 273
Wind about the same

2030 set oil htr setpoint to 274°F
2210 set oil htr setpoint to 275°F
Wind reduced to a slight breeze. Temp 86

2213 At 1600 Hrs had 1000 gallons to go to get Tank Car 3684 to 9500 gallons.
19,300 − 10075 = 9225 − 400 = 8825 + ~100 gal H_2O

@1600 - 141 TK volume was 10333
2224 @2223 " " Volume was 10065
268 gallon in 6 HR 2

268 gal in 383 min = 0.7 gal/min

10065
− 9333
732 gal ÷ 0.97 gpm = 755 min = 12 Hr, 35 m

22:23 + 12 Hr 35 m = ~10:58 Hrs — Change Tank car 3684 to 3687

Don't forget to put on placards & MSDS:

2324 10014 gal in − 141 10065 − 10014 = 51 gal
= 0.85 gpm

Grave yard - (Wilbee Dickee Redee) 2330 hrs 7-30-90
Kissed Swing-shifter good night and things are running smooth.

7-31-90

0230 AM - It appeared by Tank 141 volume measurements that the feed pump was slowing down. However, after a couple of measurements, a computation of the Flowrate was 67 gal/80 min = 0.84 gpm which is pretty close to the distillation rate of about 0.9 g

0500 Vessel pressure 28 inches, so increased Temp setting to 276 from 275 (1 deg).

0545 Vessel pressure 29 inches, increased Temp. setting 1 degree to 277 from 276.

0708 Dropped temp. setting back to 276°. Vessel Pres. Keeps hitting the upper limit of 33 psi. Red

Day shift RGC - GTMc - RSP

8:00 Down to 9500 gal in Tank 141 Reduced set point to 200°F and turned off the heater - cooling to clean the screen on the pump suction. Need to have temp ~200°F before water flush.

Reduced log printout to every 20 min.

1300 Started clean out of pump strainer Vessel temp 201°F

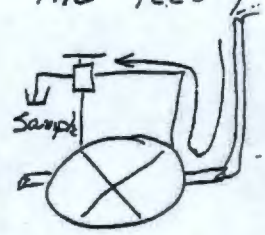
1430 Changed Rail Cars, Tighten valves on top of cars, some hexone has gotten through and dissolved the grease.
TK CAR HO-104 - 3684 FILLED, NOW FILLING HO-104 3687

1500 heat back on at 200°F

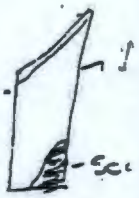
1510 When we cleaned out the pump inlet filter; we first purged the feed line with water. We worked the feed pump recirculation valve so that we flushed the recirculation line and the pump. Total water use was 15.5 gallon. (at 1 to 2 gpm). When we opened/drained the system we found about 2/3 water, 1/3 hexone.
LESSON LEARNED: [illegible]

7-31-90

2) While the feed pump was down, before we cleaned the filter I clec
Red & my idea of obtaining a feed sample from the feed p
recirculation valve. The idea is that with
the pump off that there is enough head on
the pump outlet line to get a sample from
the spare port on the recirculation valve.
When we took the cap off and opened the
valve to straight thru we were able to
get 1/2 to 1 pint of liquid (water/hexane mix). So we will ge
the fitters to add a section of tubing and a ~~pipe~~ swage
cap on the valve and we have a quick, easy, co
sample point for our feed.



3) The filter had hardly anything in it, mostly it was
~~f~~ rust and scale about the size of coffee grounds/tea leaves.
I filled up the spare filter with sand to about the
same level and set it on the main desk.
Bottom line: we can easily run a week without
clean out, but probably shouldn't go longer.



Also: There is no o-ring under the filter basket lip. The o-ring
came with the spare basket is for the lid to the contai
not for the strainer. (we took spare and checked)

4) hexane has dissolved the grease in the fill line valves on a
rail cars, mostly noticeable on 3085. but also to a barely noticeable
amount on the other cars. Don (pipefitter) tightened up the val
which will make them more difficult to turn whenever we
to change rail cars next times. Do not expect it to be an ea
job. If valve does not turn, get pipefitter and loosen packing no
and then hit tapered end of valve to toosen.

Swing Shift 7/31/90 1530 - 2400 RJC, DDP,

1545 continuing to raise oil temp to start distillin

1615 HX oil temp setpoint raised to 238°F
1630 Raised oil temp setpoint to 241°F
1642 Burp is tank shut off oil heater; reduced setp

WKS

7-31-90 Pressure before bump got to 50 psi; apparently a bunch of water flashed over causing ΔP to rise. High pressure lasted from 16:39 to 16:42 based on printout) readout of alarms.

1737 Raised Oil Temp to 240 °F from 238 °F
1805 Raised oil Setpoint from 240°F to 242°F
1815 Raised oil Setpoint from 242°F to 245°F
1825 Raised oil Setpoint from 245°F to 250°F
1835 Raised Oil Setpoint from 250°F to 255°F
1845 Raised oil Setpoint from 255°F to 260°F
1855 Raised oil Setpoint from 260°F to 264°F.
1909 Raised oil Setpoint from 264 to 267°F
1927 " " " " 267°F to 270°F
1945 " " " " 270°F to 272°F
2003 " " " " 272°F to 273°F

2113 Distillation rate is ~10 gal/10 min - A little higher than previously. Wind came up ~6:00pm and still blowing 15-20 mph. from NW. 86°F outside

2134 -141 TK LEVEL 9241 GAL WAS 9978 GAL @ 0000 HR ON 7-31-90 = 737 GAL. START 9451 @ 13:24 210 GA DISTILLED 80 FAR.

2241 Good distillation rate right at 10.1 to 10.2, 9.10 ~~gpm~~ gall per 10 min. 52 gal change in 60 min in Tank - 141 (2234 Hrs) [illegible]

2330 - Graveyard Shift - W. Greenhalgh, Red Wood, & Dick Henderson 7/31/90

0200 - Raised Temperature setting 1 deg.

0300 - Vessel pressure 25, raised Temp. setting 1 deg. To 275.
0305 - Hexane tanks press was .27 in water adjusted to .24 Red
0415 - Dropped temp. to 274, vessel press Kept bouncing to 34+ Red

0450 [illegible] - 0579 = 242 [illegible] / [illegible] = 66.75 [illegible] / [illegible] [illegible]

8-1-9 Graveyard (Cont'd)

There is a hexane waste inspection log out on the chain about the drum of mixed waste. A weekly inspection is required once the drum is full so I have already initiated it so we don't get tattled on by all of the environmental and safety people.

RJP (DDP, DIDN'T) FCC

DAYSHIFT WED 1AUG90 PAV / COWAN / GEORGE MD

The last Reading for rail car 3684 was 9,261 gallon. This is from the 14:03 log on 31JULY90. We need to 'stick' the tank for definite value.

RED: The new pump is in spare parts room. It is not an 895-MD just an 895. Does this just mean that the mag drive is not attached? or do we need an 895-MD? PAV.

1030 raised temp to 276°F getting a slow drop off in heat transfer - OR WERE RAISING THE BOILING POINT OF THE DISTILLATION VESSEL SOLUTIONS. C.E. the VESSEL temps are also increasing

Barring any major difficulties we should be finishing up tank 141 on thursday or friday of next week (Aug 9 or 10)

JWB DDP

1725 Raised oil temp setpoint to 278°F

Swing Shift 8/1/90 RJC, DDP, JWB

1941 Raised Set point to 279°
0004 to 2004 Hrs 1224 gallons distilled over — ÷ 20 ÷ 60 = 1.02 gpm.

2314 System operating very well. Distilling at 1 gpm all evening.

GM

8/1/90 - Graveyard Shift W. O'Greenhalgh, Red Wood, & Dick Henderson

Everything is running smooth at the Top end of shift.

0020 - Vessel pressure 33+, Turned Temp. Setting down 1 deg. to 278.

0415 Vessel pres. 28.8 - Raised setting to 278.

0438 Changed nitrogen Dewars, also adjusted Magnehelic from .30 to 0.23. The fittings around the Dewar were leaking (improperly fitted). The leaks were eliminated by the Tightning and leak testing of the several fittings.

(Please Arrange To GET Dewar Filled!)

RGC
RS

8/2/90 Days Geo, Pav, Cowan

Everything is running smoothly

0830 Disconnected N_2 Dewar - (Don) and recharged Eye wash stations.

1245 Still have a very small oil leak from Valve H9 on the hot oil system - may have lost a few oz. of oil this week. This should be check the next time we are down, & will have the fitter look at it monday. R L Cowan

1300 I increased oil temp to 280°F because I just can not stand going nothing. This is the only interaction with the unit this shift

1500 576 gal this shift?

98

8-2-90 SWING SHIFT RJC, DDP, JWB

Running smoothly!
141 Volume 9122 gal 0004 8-1-90
" " 7694 0003 8-2-90
1428 gallons in 24 Hrs = 0.99 gpm

141 Vol 8084 8-1-90 16:04
6694 8-2-90 16:04 Hr
1390 in 24 Hrs = 0.97 gpm

1825 Raise oil Heater setpoint to 281°F
1847 Raise " " " " 282°F
2044 " " " " " 283°F

2210 Running very smoothly. Pressure 30.X with X varying from 0 to 8

192 gallons in last 4 Hrs or 0.8 gpm - slowed down a little - last night - same time period (1800 to 2 Hrs) rate was 206 gallons based upon decrease in tank volumes

8-2-90 Graveyard Shift - WO Greenlgh, Red Wood, + Dick Henderson

The system is running good.

0355 Computer Crashed - put new disc in & rebooted.

0538 Raised temp. to 284°F Red

FRIDAY DAYSHIFT (3 AUG 90)

9.40 RUNNING SMOOTHLY AT THIS POINT - WELL ENGINEERED SYSTEM!

11.45 ISSUED ECN 106785 which incorporates the feedline check valve bypass and the new feed line filter. WE are scheduled to install the feed line bypass on Monday morning (Aug. 6) and the [illegible] is in the "back

of the logbook.
Ose

8-3-90 Swing Shift Biglin, Paine, (Pauline, Greenhalgh)

16:35 Received N2 Dewar. Better late than never.
Graveyard: I doubt there is enough in the current dewar to assure that adequate flow till Monday. Should change out to the new Dewar for the weekend before leaving. On Monday we'll use up this Dewar.

Disregard above note; We will continue on with this dewar so that we will have a full dewar to start with on Monday. Red at the end of graveyard or when you get home call Dick (Cowan) and let him know how much is left in the dewar. He will come out sometime Sunday to make sure we're still ok. or change out the dewars then.

Note For Day shift on Monday – Please have the operator make-up an inventory of the waste in the waste drum. We will need to have that inventory listing when we dispose of the Drum — Thanks
Hilton

20:45 Vessel Pressure 28 to 29, So raised Temp setting 1 deg to 285.
Ose

8-4-90 Graveyard – W. Greenhalgh, K. Wood, R. Wendorf

0140 Raised temp. to 286° Red
0240 Raised temp. to 287° Red
0500 Dropped temp. to 71° which is ambient
0525 Shut off feed pump.
0531 Shut off Hydrotherm heater
0557 Shut off oil pump & Condensers.
0600 Set cover gas press. at 2.0 in H2O. No. 1 N2 Dewar is reading ~ 40%. Opened Valve 3" going drain pan under dist. Units to sump pump.

0630 Systems are all shutdown & data logger switched to

Monday
8/6/90 Day shift RGC, JTMc, RSP

N_2 Still OK tank pressure .12 increased to .2

1130 completed addition of bypass on check valve in feed line. Drained pump stripper, cleaned, returned all hexane to tank via strainer, and primed pump. had problems with pump star looks like this is about the limit.

1140 Started hot oil system

1330 Temp increasing slowly to avoid the bumping we had last time (we added 15 gal of water to system during screen clean out) RG Cowan

When cleaning out strainer it was 1/4 - 1/3 full, slightly more than last time. We should be able to run the full week without cleaning.

We emptied out the hexane containers from inside the fence into the strainer housing and allowed it to return to tank. We placed all used containers into 55 gallon satellite storage drum. Forgot to get inventory of drum contents.

After installing valve bypass, we hydro tested new lines by closing valves 4 & 5 and then pressurized with water. Only leak was at wet hose connection which we fixed.

RJC

SWING SHIFT RJC, JWB, DDP

16:40 Setpoint up 2°F to 244°F

17:01 Setpoint 249°F from 244°F

1705 Changing over N_2 dewar to full one; 1st dewar pressure down to 80 psig.

17:10 As we get closer to the bottom of the tank KEED a

we pump the closer we get to cavitating/cavitching the pump. Eventually we will have to keep a continuous watch on the pump.

1710 Raised oil H to setpoint from 249°F to 254°F
1723 Raised oil Setpoint to 259°F
1740 Raised oil Setpoint to 265°F
1801 Raised Oil Setpoint to 270°F
1822 Raised Oil Setpoint to 275°F; Shut H_2O Valve #6
AT 1801 New LN_2 dewar liquid valve was dripping. It tightened down & drip stopped. Will check again in ~30-40 minutes.
1850 Oil Temp Setup 2°F to 277°F; LN_2 Liquid valve still frosted, but not dripping
1920 Raised oil setpoint to 278°F
2017 Raised Oil Temp. setpt 1°F TO 280°F; Temp was raised one degree ~ 2000 Hrs.

2200 -141 TK VOLUME = 4268 > Δ = 45 gal in 60 min
2100 -141 " " = 4313 = 0.75 gpm

2200 RAISED OIL TEMP to 281°F
ΔP BEFORE ~ 27.6 to 28 psig. WE'LL SEE WHAT HAPP

2250 TK 141 Vol = 4232 Vs. 4358 @ ~~2150~~ 1950 = 126 in 180 min
= 0.7 gpm
ΔP across System slowly decreasing - 30.K To 29.2 psi
Outside Temp = 84°F

2310 ΔP = 32.0 after we raised Temp of Oil to 282°F @ 11:00PM (2300 HRS)

GRAVEYARD WOG, ERW, RGW.

0141 Raised temp to 283° F. Red
Need to pressize the eye washers -. Red

0510 Raised temp to 284° F Red

LA: SHIFT 7 AUG 90 (PAVLINA, COWAN, MCKENZIE)

Thanks to swingshift for cleaning up the trailer

I talked to RICH WEISS about tritium analyses. They have performed a beta & spectrum analysis that shows that our source of beta is tritium. Copies of the data are attached to page #47.

The first sheet is the Hexane sample, which the shows a symmetric peak at channel 160. This peak is symmetrical with a slightly steeper slope on the higher energy side.

The second sheet is a clean tritium sample. Its peak occurs at 200 and has the same shape as the Hexane sample. The reason for the shift is the quenching effect of the "dirty" sample, as noted by the Avg. H data.

The third sheet is analysis of a C-14 sample. This is the only other likely candidate in the same area of the spectrum. This does not look like our sample spectrum (more trailing on the low energy slope) and peak is at ≈ 560. The only other isotopes that show in this spec. are chromium & nickel.

2950 I increased hot oil set point 1°F to 28

The ΔP on #1 demister is not as high as previously ~~for the~~ 1.8 as opposed to 2.7 inches. Possible break thru?

RJC

Swing Shift 8/7/90 RJC, DDP, JWB 1530-2400

:700 141 Volume 3108 Vs yesterday-same time = 4429
Δ = 1321 in 24 Hrs 0.55 gpm

Rail car volume @ 10:55 8/7/90 = ~7200
@ 16:40 8/7 = ~7420 > Δ = 220

141 @ 10:55 = 3458
141 @ 16:40 3138 Poor agreement
Δ = 320

We had Switched to Tank Car 3687 on 7-31-90 @ 1430 Hrs
Volume 141 was ~9450 gal
8-7-90 1700 Volume 141 was ~3108
6342 gal in Tank car + any H_2O as to System say 6360 gal. 7420 is off over 1000 gal
We need to use a dowell and get an accurate rec

SPECTRUM ANALYSIS
ID:HEXONE SAMPL
SAMPLE DPM:N PRESET TIME: 2.50 2SIGMA: 0.00

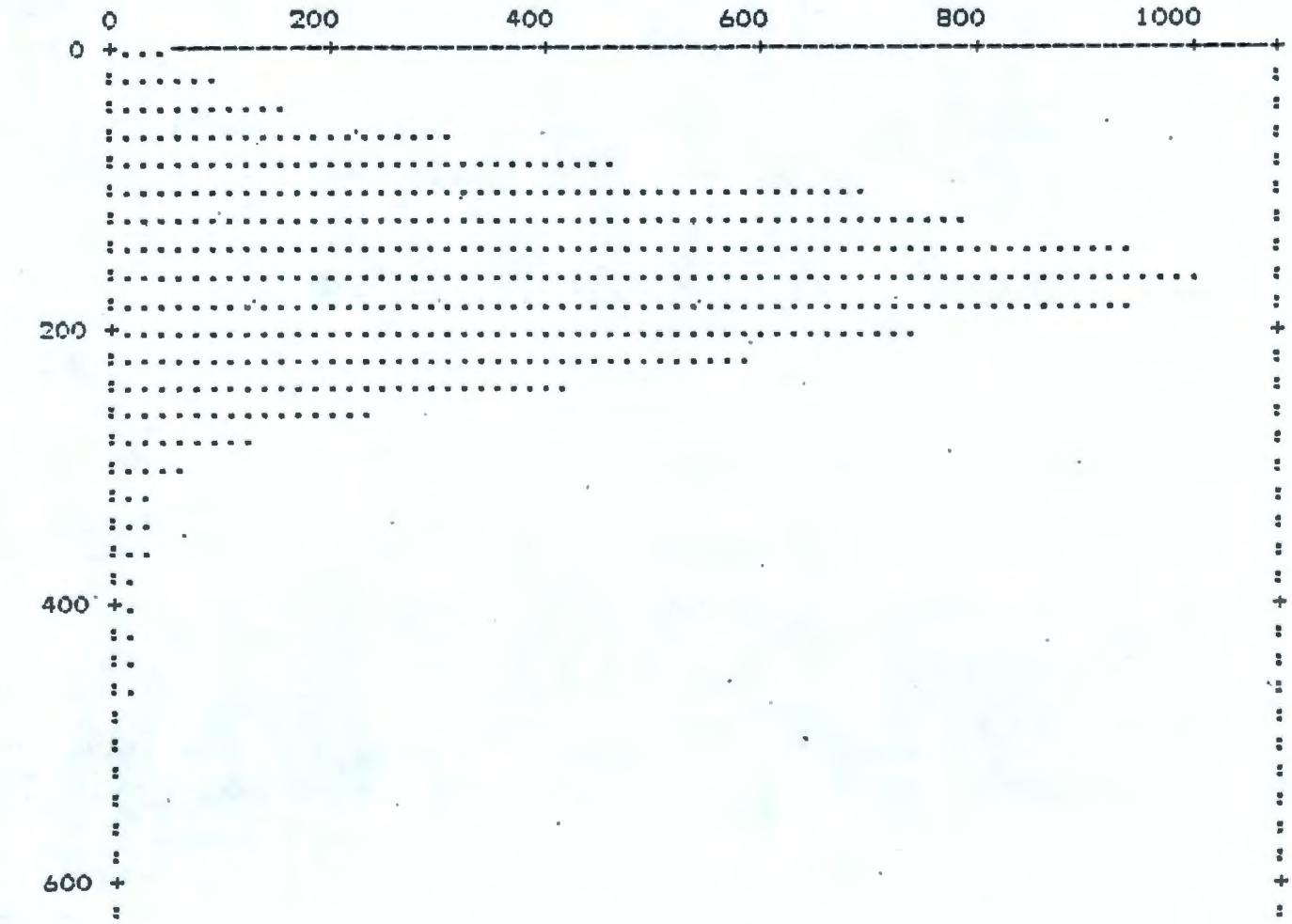
SAMPLE: 1 POSITION:**- 1
TOTAL COUNTS: 7821 CPM: 3128.40 2SIG: 2.26 TIME: 2.50 ERR

LIMIT SEARCH	LIMIT SETTING	AVG H#	UNQUENCHED SETTING
1.00	472	163.0	
5.00	284	163.0	
10.00	253	163.0	

REGION(0-1000) INCREMENT SUM:20
COUNTS: 7821 CPM: 3128.40 2SIG: 2.26 TIME: 2.50

SAMPLE SPECTRUM

X AXIS: CHANNELS Y AXIS: COUNTS



8-7-90 1740 WIND IS OUT OF EAST - GETTING O
OUTSIDE TEMP = ~~98°F~~ 97°F

1800 3074 gal in 141

1920 Raised Temp of Oil to 286°F

2000 ΔP = 31.0 psig 141 level 2966 gals

2100 141 = 2920 gal ΔP = 30.7 Outside Temp 91°F

2258 Raised Temp of Oil to 287°F .

2300 141 = 2821 0.84 gpm over last 5 Hrs - Rate falling off - from last night. Coils fo ? or not?

Check pump (feed) frequently - every 30 minu or so - make sure its pumping OK.

GEAR gAd - WOG, ERW & RCW.

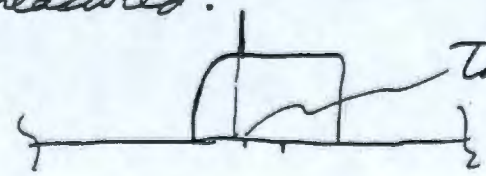
0357 Raised temp to 288° F RH

0502 Raised oil Control/Ea to 289°F RH.

We are getting approx. 50gal/hr. RH

8/8/90 Wednesday Day Shift

1000 Tried to take a level reading on car 3684 by going down the ves to get a true level reading. The 1/4" copper tube hit the rail car tank top inside the dome and no level could be measured.



This area is not cut au under the vent line

8/8/90 Wed - Day shift cont

1300 at present rate of ~0.95 gpm and an inventory of about 1816 we will lose prime about the end of swing on Thursday

When the rail car level was check by blowing out the transfer line and then reducing the purge rate back to 2 cfh it was 7565 gal when T-146 was 1994 thus the rail car should fill to 9559

We modified the feed pump recirculation valve to provide a feed sample point. We took 3 vials of samples at this point simultaneously with samples of the product.

To take sample we shut off the feed pump, turned valve to off. (handle pointed to trailer) opened the tubing plug, and then turned valve to let the piping from the vessel to the pump gravity drain. System worked well.

A definite visible difference in the inlet/outlet samples. The feed sample appeared to be like ~~Kuwaiti~~ Iragi crude and the product a clear yellow solvent. We took some photos of the samples for future reference and reports.

Sent the samples to 222-S lab for the following Gamma Energy Analysis (GEA), Total Alpha, Total Beta and, Total activity. The GEA/TA/TB will give us the necessary characterization data and the total activity will give us results that can be compared to the previous data.
* NOTE: the total activity method is used for low activity samples (50 - 10,000 pCi/g) and measures alpha/beta not gamma. Also it does not accurately measure softer betas like tritium. i.e. Total ~~Alpha~~ Activity is not really what the name implies

Wilbur: The samples going to 222-S; do they require an RSR or not? If not please write up the reasoning in the log so that we can pass that info on to whomever needs it.

Ah.: as noted above if the feed is like a light crude and the outlet is clean.

8-8-90

~~Other~~ The pump is showing some difficulties on restarting. Its not keeping its prime well on restart. We are getting to the point where we need to keep a close watch on the pump or loose it. Two HOUR OBSERVATIONS are ~~probably~~ not enough. Remember we have about 1 minute to shut off the pump before we loose it if it starts sucking air.

~~Mark~~

PLEASE DO NOT SHUT OFF FEED PUMP FOR SAMPLING WHEN IT'S AT THIS LOW LEVEL WE MAY NOT GET IT RESTARTED — HEAD DIFFERE IS TOO GREAT.

WATCH PUMP ~ 15 MINUTES
Air Temp 100°F

1930 Checked both emergency air horns for pressure. Both are OK
Put on tags
Last 80 min 18:00 to 19:20 76 gal/80 min = 0.95 gpm
Setpoint still 289°F ΔP=29.3 psig Working great! Pump

2120 Set point to 290°F; Outside Air Temp to 90°F

1600	1789
2200	1506
6 HRS	283 gal

= 0.79 gpm. AT THAT RATE 1506 gal will be gone in 1915 min = 31.9 HRS OR ~ 0600 HR 8-10-90.

2000 HR	1591
2200 HR	1506
120 min	85

= 0.71 gpm

BUT

2200 HR 8/7/90		2875
2200	8/8/90	1506
24 HR		1369

= 0.95 gpm

Last 2 days

2200	8/6	4268
2200	8/8	1506

2762 ÷ 48 Hr = 57.5 g/HR = 0.

8-8-90 2215 I've been checking feed pump ~15-20 minutes. It's sounds good, and pressure is good. When we get down to near empty in -141 we'll have to babysit pump in order to shut it off in just a few seconds of running out of feed. RJK

relieved WOG, ERW, RGC

8-8-90 2337 Raised temperature to 291. RGC.

8-9-90 0135 Raised temp. to 292. RJC
8-9-90 0600 Raised Temp. to 293 RJC
0730 We have less than 1000 gals to go.

8/9/90 Day shift RGC, Geo, Pat

920 Down to 876 gal indicated in tank. The calculation of volume is based on the dip tube being 1.6" from the bottom of the tank if it is higher we should see a slower than expected change in level in tank 141.

"What to do when the pump runs dry" by RSP & RGC.

1) At the first sign/noise of pump loosing suction, SHUT OFF THE PUMP. Vendor data says we have about 1 minute to shut off pump when running dry or unlubricated.

clarification: when either pump suction or pump outlet pressure goes to zero and remains there

2) Keep the heaters & condensors on.

3) Maintain Hot Oil temperature, but do NOT INCREASE.

4) When #1 vessel pressure drops to 10 inches or less go thru normal shut down (i.e. shut off heaters, then condensors later). No Hurry on shutting off condensors.

CM

8-9-90

WILBUR: I AM WRITING THIS E
SO YOU WILL NOTICE. ON PAGE 49 I
ASKED ABOUT THE RSR'S FOR THE SAM.
SINCE THERE WAS MUCH DISCUSSION AND
HPT'S & SHIPPING. ABOUT THIS, WOULD YOU PL:
WRITE UP SOMETHING IN THE LOG FOR US ON THIS
SUBJECT. THANKS

PAV (A.K.A. Mr Subtle)

PAV There are RSR's and HMSR's (Haz Mat. Ship Records).
side drawer. A shipper, myself or John Biglin etc would hav
1500 complete. Switched rail cars, now usin
whatever is appropriate for the specific
3688, installed placards but not c
MSD'S

Volume 8950 gal

8-9-90 SWING RJC, DDP, JWB

18:20 SWITCHED TO 10 MINUTE READOUTS/PRIN
BABY SITTING PUMP W CHAIR ON FLATCAR. 20 mi
MAN. PUMP SOUNDS GOOD. 364 GAL TO GO —
SETPOINT ~~292°F~~. OUTSIDE MUGGY TEMP 99-100
293°F 8/9/90

1900 8-8 1638
1900 8-9 334
1304 in 24 Hr 54.3 gal/Hr = 0.91 gpm

1930 increase air setpoint to 294°F

2130 Product tank still filling @ ~9 gallons every
but 141 TK level is only about 6.6 gal/10 min

Talked with Dick Cowan a few minutes ago ab
shutting off bypass loop on feed pump, in order t
lose prime if several gas bubbles are sucked
pump We agreed to

8-9-90

than risk drawing up more crud/debris that would probably result from greater overflow back to 141 from Distillation Vessel #1. If we lose prime, we'll just shut off feed pumps. We have to come back to 141 and steam it anyway to clean it out before Yank Tank operations begin. RJL

2240 TANK ~~X~~ 141 LEVEL IS NOT CHANGING, BUT PRODUCT TANK IS STILL FILLING AT ~ 9 gal EVERY 10 MINUTES - I HAD CRANKED UP 141 & 142 FLOWRATES TO BUBBLERS (141 TO TOP OF SCALE) AND 142 TO ~1.5 SCFH. VOLUME IN 141 CHANGED TO 554 AND 142 CHANGED TO 16011 FM 15981. FM 275. RJL

I BELIEVE 141 BUBBLER HAS RUN OUT OF LIQUID. WILL TRY NOW A SETTING OF 0.5 SCFH.

2250 VALUE ON 141 DROPPED TO 156 GAL AND 142 TO 15972. LARGE CHANGE ON 141 ALSO SUGGESTS 141 BUBBLER IS NO LONGER UNDER LIQUID. GOING BACK TO 1.0 SCFH RJL

GREAEqAid Wilbur, Red, Dick

2333 Raised temp to 295° EGW.

0030 Raised temp to 296° ERW

.315 Feed pump pressure went to 0 immediately turned off, no pump noise. RJL. Need out @ 310 tank 141 → 177 gal

0400 Vessel Pressure 7.5 inches - turned oil temp setting to ambient Temp.

.515 power to heaters off RH - oil in 261°F.

0700 oil pump shut off

RJL

8/10/90 Friday day shift

0830 The bubbler tube in 141 seems to have become plugged during the last shift. I went to max flow on the N_2 to blow the line out then cut back to .2 scfh to get reading. Then opened the transmitter ~~cross~~ cross over valve and isolated the transmitter. This gave a true zero which was off by 0.03 inches then

Final Tank volume, corrected for Tank pressure, is 50 gal. (No liquid at the end of the level dip pipe)

MONDAY DAYSHIFT 13 AUG 90 George McD, Bob Paulina, and the new guy C

We changed out the inlet filter this morning successfully without spi a single drop. Hydro tested the system, using the pump water prime set-up, to 100+PSI. Note: That there is a new gauge in the system This gauge gives us the true pressure on the suction side of the pum The older gauge was on the recirculation loop. ~~and~~ Use the new gaug for all readings from now on.

Successfully primed pump, but ran into data logger problems "Error line 280 "volume"" So rebooted (twice, I goofed on first) and data logger gave error message for line 280 again. After a short period of pan (that Dick had sabatoged the program then went on vacation) I dis that I had reinserted a <u>used</u> "new" disc instead of a new "new" disc. So that when the set-up disc interfaced w/ the data disc it was finding something wrong. BOTTOM LINE; we started up at 11:30 AM.

12:55 We've brought the system up quickly to 180° and (hot oil set point) then let the #1 vessel temp equilibrate. Increasing hot oil temp now in 2° increments since there is an unknown mixture of a heavy organic and water (assumining about 50-50 mix). With this unknown mixture it is better to make smaller changes more frequently than large changes.

*

8-13-90

14:45 Changed out nitrogen dewars. However.... The new dewar was at 2600 psi pressure and when it was valved in it hammered the demand regulator. The tank pressure was 1,07 inches and backing off the sensing regulator had no effect. (i.e. we could raise but not lower). Closed the ball valve from the dewar to the demand regulator and let the demand regulator and tank pressures drop to normal operating range. Then slowly opened the ball valve and throttled the N_2 with the needle valve. The demand regulators were then adjusted back to normal operating range.

Bottom Line: When changing out dewars only crack the needle valve slightly open if the cylinder is at high pressure (100+). As the cylinder pressure drops the needle valve may have to be opened further. which leads us to ...

Bottom Line, Part II: Swing shift should keep a close eye on the demand regulator and dewar pressures. Open the needle valve if necessary as the dewar pressure falls.

16:15 Hot oil set point NOW: 231°F
Solution temperature: 200°F

16:30 N_2 Dewar down to 140 psi ~~looks~~ opened needle valve. N_2 Demand regulator should be normal now

Driving: [illegible], [illegible] and new guy Olaf who is waiting for Bob Cash to come on swing.

1617 Raised temp to 242°F

1625 Raised temp. to 244°F [illegible]

1640 Raised temp. to 245° [illegible]

1700 Raised oil Temp to 246°F [illegible]

1705 Dist Vessel #1 @ 210°F but Vessel #2 is 214°, we're distilling heavy over to transfer tank from Vessel #2 but #1 is probably not distilling over very much.

Product tank level @ ~~15:55~~ 15:45 = 20.1 gal @ 16:15 = ~~25.~~ 36.

8-13-90 17:25

1725 Hrs
1615 Hrs
70 min 7 x 5.5 = 38.5 gal

SINCE 15:45 TO 17:25 PRODUCT TK HAS ACCUMULATED ~16.5 + ~38.5 = 55 gal – probably mostly Hexane.

17:45 Dist Vessel #2 temp. jumping around. 220 down to 213°F @ 17:35 back to 218°F @ 1747. HOLDING Temp of Oil to 246°F for awhile. ΔP of System #

1822 Vessel #2 temp up to 223°F. We're going to bump Oil setpt. to 247°F to see if we can raise Dist. Vessel #1 temp above 211°F.

1837 Oil Temp to ~~248~~°F 249°F (2°F Bump up) ΔP = 15.4 psi – 2nd Vessel may be nearly empty. Transfer TK only increasing ~2 gal in 10 minutes

1902 Oil Temp up to 257°F DV#1 = 212°F DV#2 25°F

1115 oil temp to 253°F.

1935 Oil Temp to 255°F ΔP = 20.9 psig in

1957 Oil Temp to 257°F ΔP 27.3 psig Dist Vessels #1 & both at 213°F

2010 Oil Temp to 258°F ΔP 28.6 psig Vessels #1 & 2 @ 213

2016 AT 2006 WE GOT AN ALARM ACROSS DEMISTER PAD #1 ΔP > 8 (alarm point). Set Temp of Oil back 2° to 256°F

2019 DROPPED OIL TEMP TO 254°F ΔP Demister still at 8.2 p

2021 Alarm cleared on ΔP ACROSS DEMISTER #1

2100 Alarm OK ΔP now at 7.0 psig.

2320 I THINK DIST VESSEL #2 IS NOT FULL. RATE is

8-13-90 ONLY ~2.5 gal/MIN 10 MIN. ΔP FOR DEMISTOR #1 CONTINUE ~7.5. WE'VE HELD BACK AND NOT PUSHED IT.
ΔP SYSTEM = 24 psi

DAY SHIFT — PLS PUT UP MSDS SIGNS ON 3RD TANK CAR 37688. RJR

23:30 Grave Yard - W Greenhalgh, J. Biglin, D. Paine

00:45 ΔP dropped below 7, Raised Temp. 1°F to 255.

01:15 magnehelic pressure 0.6 and not-adjustable. We ~~found~~ adjusted other diaphram values to get back to ~.22. It appears sometime earlier the system might have got a jolt of pressure since there is a broken dial guage as well as the system being out of adjustment — Need to check out on Day Shift if possible! Thanks

SEE ~~more~~ Log Entry on page 55 PAV

02:45 ΔP 7.1, raised Temp. 1° to 256.

05:45 ΔP 6.4, Vessel P 28, raised Temp 1° to 257.

05:50 ΔP & UP still dropping, raised Temp Setting To 25

05:55 ΔP & UP still dropping, raised Temp Setting to 260

06:30 Vessel Pressure 25 & ΔP low, Raised Temp to 261.

06:55 Vessel Press. 28 & ΔP low, Raised Temp to 262. RJR

DAY SHIFT: MR. "O", PAV, GEORGE. 14 AUG 90

Someone needs to check their units! All pressures are in "inches". Common error thru-out log. Sorry! You're right!

The "OFFICIAL" STARTING VOLUME FOR TK-142 is 16,036 gallon (not psig)
This is from the first log at start up 11.58, 13 AUG 90.
461 gallons process so far from 142.

8-14-90.

BOB (CASH): A REMINDER THAT OLAFS GONE WEDNESDAY NIGHT SO COME THURSDAY DAY SHIFT ITS JUST GEORGE & MYSELF. ACCORDING TO YOUR EDICT WE NEED ANOTHER PERSON OR SHUTDOWN. PLEASE ARRANGE. ALSO ON FRIDAY WE'LL BE THREE PEOPLE SHORT (OLAF, COWAN, MYSELF) WE MAY WANT TO JUGGLE HOURS SO THAT WE RUN DURING DAY AND SHUT DOWN ON SWING. W/NO GRAVEYARD. WE'LL STICK SOME SUGGESTIONS ON BULLETIN BOARD.

09:30 THE HPT IS MEASURING A HIGH BACKGROUND 2500 cpm from OUR NEW FEED LINE. WHATEVER WE'RE PROCESSING, ITS RADIOACTIVE. ITS NOT TRITIUM EITHER, TRITIUM'S A SOFT BETA THAT WE WOULDNT BE MEASURING THRU THE FEED LINE.

Other Operational Observations

- Product tank is filling at rate of 3.7 gallon/10 min = 0.37 gpm as hexane. This calculation uses a hexane density correction so we are actually processing @ 0.30 gpm as water.

- I think we have a new distillation vessel #1
  We obviously accumulated some organics (byproducts) during 141 processing. Since we had boiling solvent in the vessel the byproducts would not coat the vessel/coils but probably stay in solution. When we started tank 142, were pumping the water from the bottom of the tank, which will displace the organic in distillation vessel #1. The organic would then overflow back to tank 142. Leaving us with a "clean" vessel filled with water. This is supported by the ΔT for hot oil in/out is 14° the highest since start up.

- The distillation vessel pressure: We've been running at 30" for hexane but with water in the seal loop, this would be equivalent to 37.5 inches. So we can run at higher pressures since the water in the seal loop will not blow out as easy as when hexane in the seal loop.

10:45 ~~Samples~~ Took two samples on both inlet & product lines (1 archive & 1 lab analysis) Both were aqueous samples, indicating we've flushed the organic out and have only water in the system. Also the sample bottles for the feed read 400 cpm above background and the product read background. We really are decontaminating the stuff!

8-14-90

1:30. Placed ~~out~~ old inlet filter/piping into satellite storage drum. Also took inventory and attached inventory sheet in the blue log book. ~~on~~ (hanging from fence)

INVENTORY OF WASTE DRUM

- Plastic bottles and tin sample container. These contained liquid which had dripped from pump/filter maintenance. liquid later returned to tanks, so there is no liquid in containers
- used face mask chemical cartridges & surgeons gloves, tape.
- used pump head that failed on feed pump.
- plastic bags with rust/debris from inlet filter.
- bag of used solvex gloves.
- yellow bag containing pump inlet filter & flex lines w/ misc pipe fittings. ~~Rep~~

updated 14 Aug 90 RS Paulin

Rgd.

1:30 ~~Graveyard~~ Swing, Starts with Dick, Red & Bob C.

1700 625 gal since startup on 8-13-90 ~ 1530 = 24.5 g/hr
Not too bad for water

1900 Setpoint still at 263°F; 100% power on heater
Temp Oil in = 260°F

2100 Hot Oil Temp still 260°F w 13°F ΔT. Distil. rate still about 13.5 gal every 10 minutes. ΔP still 29

2300 ~~DAY~~ SHIFT & GRAVEYARD SWITCH – PLS READ MY MEMO ON SHIFT CHANGES. RJ Cash

GM

ALMOST 900 GALLONS DISTILLED OVER. OIL TEMP AT 259°F. POWER STILL AT 100%. MAYBE ONE OF HEATER WIRES IS BURNED OFF – OR FUSE IS GONE (BLOWN)?

8/14/90 23:30 Graveyard Shift: D Paine, J. Biglin & W Greenhalgh

00:00 8-15-90 Vessel Press is 29, & ΔP is 4, Raised Oil Temp Setting to 264(.

00:30 Vessel Press 27, [redacted] Oil Temp. not changing, heater must be full power as indicated earlier. The vessel pressure is going to continue to drop as night gets cooler. Oil Temp. display does show heater to be at 100% power.

00:15 Adjusted Magnehelic reading to ≈ .24 from .18. Production Rate for 1 hr is ≈ 0.35 gpm.

07:00 Production Rate still about .36 gpm; oil heater controller still says it is on 100% power. Is this because of the water or is part of the heating unit out?

DAYSHIFT PAV, O, & GEORGE 8/15/90

General notes that I write in the log to amuse myself;

1) The rail car liquid level is rising deceptively fast because we are filling it with water and the data logger sees it as hexane. (1" of water shows as 1¼" hexane) This is the same with all liquid levels (product tank). Just something to remember when you use the product tank volumes to calculate production rates. (i.e. production rate of 0.37 → is really 0.3 gpm.)

2) The "shine" for the system is increasing. We are now seeing 5500 cpm at the bottom of the feed line flex hose, 200 cpm at the bottom of the filter housing and 500 cpm from the #1 vessel inlet port.

3) The inlet filter is "hottest" (radiologically not thermal) at the bottom of the housing with no "shine" on the top. We can probably use this to our advantage in determining how full the filter is. By using the GM along the side of the housing we should be able to see the readings drop off where there is no crud built up.

4). Using the "sand method" it has been determined that the new filter holds 4 times the volume of the old. However, it is 80 mesh as opp to 40 mesh, and 142 may have more sludge than 141. Therefore we a probably fill up at least 2x as fast. When we started 141 it 1 week to completely fill the filter and tear up the pump. We

8/15/90

want to wait that long again. (I'm not sure how well the pump could pull thru a full filter the size of a small coffee can.) Since graveyard is leading the week, the process will have to be shut down next week on days to check/clean the filter.

5) When cleaning the filter remember this TANK 142 SLUDGE HAS ALPHA CONTAMINATION. It's a whole new ballgame this time with 100 times the contamination, 4 times the volume, and alpha present. Cleaning the filter will not be as easy radiologically as before.

6) Sometimes I wonder if anyone really reads this log, or if I'm wasting my time. If you've read this far please initial next to the appropriate spc:
I've Read this: Rich, RJC, ERW
You're wasting your time PAV: [illegible]

7) If tritium is 6,000 pci/ml (as per Meylan Weiler) then we could only ship 44 gallons of 141 hexone at any time and keep under the 1,000 uci H_3 limit for non-rad shipment — WHERE DID YOU GET THIS INFO? DOT REG'S MUST BE MUCH HIGHER THAN THIS. How do you ship Lomanous D, WAT

8) Samples 1909 & 1910 have not been completed yet since for TX & TB samples are placed in planchets and evaporated. This also means we would not see tritium and or I-129 (partially) in the T.B. TRUE!! RGH RJC

8-15-90 SWING SHIFT RJC, ERW, RGH

1815 DISTILLATION VESSEL #1 TEMP HAS BEEN JUMPING AROUND ALL AFTERNOON. #2 HAS BEEN HOLDING STEADY. #1 VARIES FROM 210°F (LOWER THAN BEFORE FOR WATER) TO 196°F. THERE WERE SOME READINGS AT 213°F. ARE WE GETTING AZEOTROPE OVER? AND GETTING CLOSE TO RUNNING OUT OF WATER? (~1,000 gallon @ 0.3gpm = 4.6 days) Shouldn't be running out PAV

1827 TRANSFER PUMP IS MUCH NOISIER THAN LAST WEEK. WITH MOSTLY WATER IN BOTTOM OF TK 142, THE TWO PUMPS ARE PUMPING H_2O. THE VENDOR SAID H_2O WAS NOT A GOOD LUBRICANT, SO WE MAY BE SLOWLY (MORE QUICKLY) WEARING OUT PUMPS. KEEP A WATCH OUT FOR TRANSFER PUMP. — EACH SHIFT SHOULD LISTEN TO IT FOR REFERENCE IN ORDER TO KNOW IF IT IS GETTING WORSE. RJC

IMP!

DAP IM

8-15-90 22:20 TEMP OF DIST. VESSEL #1 CONTINUES TO ~~FLUATE~~ FLUCTUATE 208°F TO ~178°F. IN FA WE GOT A LOW ALARM AT 21:52 HRS AND IT CLEARED ~~AT~~ RIGHT AWAY. OUR TC MAY BE BAD OR HAVE A LOOSE CONNECTION.

DISTILLATION RATE STILL ABOUT ~3.6 GAL EVERY 10 M

16:~~26~~05 HRS TO 22:00 HRS TK VOLUME (142) WENT FROM 14749 TO 14584. Δ = 165 gal IN 6 HRS = 27.5 gal/Hr

ΔP's ACROSS SYSTEM AND DEMISTERS ARE OK!

TRANSFER PUMP COMES ON WHEN PRODUCT TK HAS ABO. 37-38 GALLONS

TC's IN DIST VESSELS ARE 96" LONG AND EXTEND IN ~48" SO THEY ARE NEAR THE BOTTOM ~ 1/4 TO 1/2 OF DISTANCE FROM BOTTOM TO TOP.

ALL INDICATORS ~~SAY~~ VESSEL #1 IS FULL OF LIQUID TO OVERFLOW LINE. WIND IS BLOWING 15+ MPH, SO LOOSE WIRES COULD BE THE CAUSE.

→ DAYSHIFT - CAN YOU HAVE INSTRUMENT MAN OR ELECTRICIAN CHECK IT FOR LOOSE WIRES IF IT CONTINUES TO FLUCTUATE?

Graveyard Shift 8/15/90 - D Paine, J Biglin, & W. Greenha

23:45 We listened to ~~the~~ product transfer pump; it sounded okay.

3:00 Rate approx. 0.36 gpm, everything running smoothly.

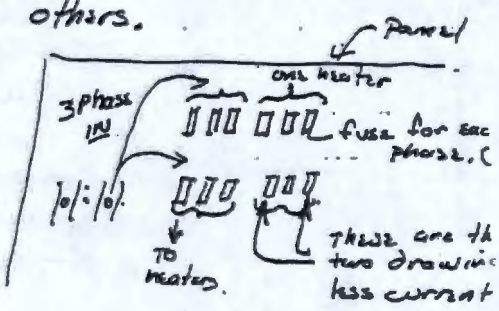
6:00 Rate still ≈ 0.36 gpm, any luck yesterday in finding out if heater output can be increased?

8-16-90

PAYSHIFT: PAV, GEORGE, JOHN. (16 AUG-90)

BOB (CASH): You're right in that the manufacturer states not to run water thru pump. However Cowan has stated that there is enough kerosene in the water to provide adequate lubrication. All indications so far show that to be true. The transfer pump has always made a little more noise than the feed. Also, you're question on if were running out of water --- If there's 2000 gallon of water and we're processing at 0.30 gpm we'll run out in 4.6 days. Its not likely we're running out of water. Also, when you see variations in the distillation vessel temp, you can determine if its real or not by also observing the vapor out temp and vessel pressure to see if they change. If its the fault of the Temp controller the hot oil in temps would have been fluctuating also.

We shut down the system at 09:00 this morning to inspect the heater electrical. There are two heater units, each unit has two heaters, and each heater has three elements (one for each phase). This totals to 12 heater elements and 12 fuses in the panel. With the heater system at 100% 10 of the 12 heaters draw about 36 amp (50 amp fuses), ~~two~~ the remaining 2 only draw about 20 amp, meaning they have more resistance than the others. All heater circuits are drawing current so there are no burnt fuses, just 2 elements at 56% of the others. I'm not sure how the heater elements are made but these two may be slightly damaged. meaning the unit is only 93% capacity.
Bottom line: <u>NO PROBLEM</u>.



When we opened up panel, the 3-phase contactor for the power in had one discolored contactor and some melted insulation. This was due to ~~some~~ a loose screw on the contact. We tightened all connections, cleaned the one contactor and reassembled.

The one heater fuse that had discolored, we cleaned and also trimmed up the wire leading from it. I see no need to replace the fuse block. But at least we now have a spare.

We restarted about 10:30. Had to prime the pump to get it restarted.

Tried to ~~schedule~~ clean out the inlet filter while we were down but there were no operators available. The soonest we can schedule operators is Wednesday afternoon. This is not going to be acceptable.

cont'd?

16AUG90 dayshift cont'd

We need to check/clean that filter either friday or early next week. (For personnel reasons, I prefer next week when we're fully manned on days.)

To schedule operators call John Renken (at Marys #), he's filling in for Mary who's on vacation until Tues or Wed of next week. They had scheduled 2 operators for sampling at CX-70 (or 71), but when I talked to the responsible engineer for that job he said that they would only need 2 operators monday morning, then one afterwards. So we should be able to pick that operator up. We need an operator, RHP, and pipefi Dayshift (i.e. OLAF) please check up on this first thing monday, or we ma loose the opportunity.

To clean the filter it is relatively easy to shut down. We killed the heaters and it only took about 10 minutes for the vessel pressures to drop down to 1". Then we shut down the condensors and pumps. Changing the filter should take about ½ hr to shut down, ½ hour to clean, Then restart. The engineer should go on mask so that he can personally see what is in the strainer and how much. (thats what I did).

14:20 Apparently shutting the controller off had an effect on it. We are able to raise the temp set point up higher than before and we're showing 88% not 100%. Nothing else we did ~~would~~ should have made any difference. DAYSHIFT just has that majic touch. (next week it's graveyard!)

14:30 Transfer pump was a little noisy this time, has been pretty quiet all day.

☆ Changed out gas cylinders/dewars. No PROBLEM. the secret now is to vent the new dewar down to < 150 lb. from the vent valve before making valve changes. this takes about 30-40 seconds. ~~Make~~ Open all valves SLOWLY and no problem, and no shock to the demand regulator.
P.S. GEORGE: please check w/ stores first thing friday morn to make sure WE have the new dewar out here in the afternoon, we don't really want to repeat that one friday

15:30 269° is the temp set point and we're at 98-100% on the heaters a little better. production rate up slightly.

8-16-90 Swing Shift - WEG

1600 ~~Green yard~~ - Dick Henderson, Red Wood + Bob P. + C.

1823 #1 OVERFLOW LINE ALARM: - REAL - PIPE WAS VERY HOT - BUT PRESSURE HAS BE RUNNING RIGHT ABOUT 30" OR 29.X" ALSO NOTED AN ALARM ON #1 @ 16491

DICK COWAN COULD TAKE FLOPPY AND READ IT TO SEE IF PRESSURE SURGED AT SAME TIMES.

It only looks on the disc. Data is the same. Data printed out! RSP 20 Aug 90

WE PROBABLY GOT A BURST/OR VIGOROUS MOMENTARY BOILING WHICH CAUSED A PRESSURE SURGE.

18:39 THIRD ALARM ON OVERFLOW LINE #1; SETTING OIL TEMP SETPT. BACK TO ~~266°F~~ 267°F RJK

1841 ΔP System 31.7"

1845 Dropped temp. setting to 267° it was 269° Red

19:25 NO MORE ALARMS ON OVERFLOW LINE. Oil #1 IN = 266 VOLUME DECREASE IN -142 ~~#9 15~~ 16036 - 14117 = 191 GAL (H2O OUT OF 142).

WE COULD BE STARTING TO GET SOME ORGANIC INTO THE 1ST DIST. VESSEL.

1940 HEATER PULLING ~88% POWER

1944 SET UP 1°F TO 268°F (OIL TEMP).

2033 BLEW LEG OVER AGAIN - 4TH ALARM ON OVERFLOW LINE. ORGANIC IS APPARENTLY COMING OVER; - IT'S LIGHTER THAN H_2O AND WATER BOILS AT LOWER TEMP. STEAM POCKET BUILDS UP AND BLOWS OVE WITH RESULTANT PRESSURE INCREASE WHEN IT BUMPS.

OIL TEMP LOWERED TO 267°F.

HEAT REQUIRED TO DISTILL OVER 2X 3 GAL/10 MIN

$$\Delta H = \frac{0.6\,GAL}{min} \times \frac{62.4\,\#H_2O}{7.5\,GAL} \times \frac{969.7\,BTU}{\#H_2O} \times \frac{1\,KW}{3213\,BTU} \times \frac{60\,m}{1\,HR} = 90.4\,\frac{K}{Hr}$$

$\Delta H_v (H_2O) \cong 969.7\,\frac{BTU}{\#}$ @ 212°F. TO FIGURE HEAT LOSSES

WE WOULD HAVE TO SEE WHAT % OF POWER IT TAKES TO MAINTAIN DIST. VESSELS AT ~ 211°F WITHOUT BOILING

HEATING UNIT PUTS OUT ~ 125 to 130 KW/HR SO IT'S REASONABLE THAT 35 KW OR SO IS REQUIRED TO OVERCOME SYSTEM HEAT LOSSES AT 211-212°F. WHEN VESSELS ARE FILLED W H_2O.

2:25 MUST STILL BE WATER TEMPS IN VESSEL STILL ABOUT 212-215°F :9f

Grave Yard Shift - 23:30 - J. Biglin, D. Paine, & W. Green.

23:30 - Because of bumping problems on swing shift we will keep oil set Temp at same as Swing

0:30 Heater is running at 100%, it apparently has been running that way for some time.

4:00 Production rate still about 0.37 gpm.

5:00 Heater is still at 100% output.

7:00 Smelled slight trace of hexane; couldn't see anything a.m. The slight odor was noticed behind the Flat car & over by the magnehelic guage. WAS WIND BLOWING TOWARD YOU CHARCOAL DRUMS?

7:30 Day Shift - G McDonald, D Paine, & W. Greenhalgh

07:30 Production Rate steady at 0.37 GPM.

08:00 Production Total for 24 hrs is 575 gal, that compares with 610 gal. for yesterday.

08:30 "Clean Screen" - An operator and fitter might be available Monday afternoon; otherwise it will be Wednesday before they are available. He will let us know ahead of Time. (John Renken)

09:00 Oil heater still at 100%.

09:10 Note from Rich Weiss on Alpha content of Tank 142 Sludge. Samples taken previously (before trailer) showed sludge to contain approx. 4000 to 5000 pCi/ml Alpha. This is enough alpha to detect with a pam if smeared on solid surface etc. It is not expected to present any particular problems other than any contamination occurring during operations will have to be cleaned up and released prior to resumption? suspension of the operation.

10:00 Full Nitrogen Dewar Received.

11:15 - Ron Lerch Mgr Env. Div. dropped by to see how we are doing. Everything was functioning allright.

12:20 - Still looks like water being processed; oil heater is still at 100%.

1:00 - Production Rate ≈ 0.35 GPM.

2:00 - Our DOE customer called to check on status. He wanted to know if we had distilled all the water and ~~the~~ answer is "no - not yet".

2:00 - The dewar flask has been connected up.

3:00 - ΔT is 2 deg, so raised oil set Temp. 1 deg. to 269.

Leaving. RED, KGH, KJC.

phone call tank Packards are in — Mike Kalinowski 224 B. 3-5792

6:45 WIND FROM SW ~ 15MPH

OIL TEMP SETPOINT @ 269 °F; POWER LEVEL 100%; OIL TEMP IN 265°F

YOU CAN SMELL HEXONE IF YOU STAND 15-20' DIRECT DOWNWIND OF CHARCOAL DRUMS — ALWAYS COULD IF WIND WAS NOT CALM.

RATE STILL ≈ 0.37 gpm INDICATED ~ 8/10 OF THAT = 0. gallon/min H_2O SINCE EVERYTHING IS SET FOR HEXONE.

8-17-90

16:45 EYEWASH STATION ON EAST SIDE OF FLATCAR HAS AIR PRESSURE – WILL NOT WORK!

1850 OUTSIDE AIR TEMP 78°F

20:25 NOT CHANGES VESSEL #1 & #2 TEMPS STILL 215
WE HAVE DISTILLED OVER ~ 2624 X 0.8 = 2099
(16036 – 13412 = 2624)

2050 SWITCHED TO 10 MINUTE PRINTOUTS
2100 TRANSFER PUMP OFF (AUTOMATICALLY)
2101 SHUT OFF HEATER AFTER REDUCING SET POINT
2103 ~~PLOT A~~ RUN PLOT
SHUT FEED PUMP OFF 2102
2110 SHUT OF XFER PUMP SWITCH
2115 Drain pan valve is open to sump.
~~2227~~
2222 SHUT OFF OIL CIRCULATING PUMP AND TWO CONDENSER FANS
TK 142 VOLUME = 13394
STARTING VOLUME 16036
$\Delta = \frac{}{2642 \text{ GAL}} \times 0.8 =$ _____

22:30 SWITCHED OVER TO K3 - FOR TF Operators

GRAVEYARD 20 AUG-90

00:15 STARTED FEED PUMP SUCCESSFULLY. Did not even try to run without priming. Took about 3 gallon to fill line and reach pressure at pump. Used 12 gallons of water total.

SAME PROBLEM WITH DATA LOGGER on restart as last week.
"ERROR 125 on line 80 : VOLUME"
~~PLOT~~

Just put in NEW data disc and rebooted. No PROBLEM.
WERE OFF & RUNNING

CREW

01:10 The rail car level is reading 3680 (±10) this should be as accurate as the system gets since there have been no transfers since Friday night

According to the data logger we've pumped out 2,631 gallons from 14.2. If a density correction is made (assuming Dick used a dens. of .8 for whole tank) this would be ≈100 gallons of pure water. (our water is anything but pure and density may well be above 1.0). We should be getting into the organic phase, and bumping can be expected. We'll be extra careful upon heating up the vessel above 200

Concerning notes on hexane smell. There is an occassional smell of hexane especially when the wind is calm. This occurs almost daily right at ~~sunrise~~ sunrise (as I'm sure anyone on graveyard had noticed) My observations is that it is not from the activated charcoal drum area but more from the area of the old pumps & valves. Smell has always been faint. Please follow up your observations as best as you can. This is one way how we can can tell if we've lost our seal loops on the tank catch pans. note the wind direction, tank pressure and precise locations.

Brought system up in 25° increments up to a hot oil temp of 200°, will start 10° increments from there. After increasing set point, watch both the oil outlet and vessel temps, as they come up, then I increase hot oil.

BTW: That eyewash leaks pressure faster than others. That's why I switched its location, and moved the one that holds pressure close to the feed pump.

Distillation vessel #2 heating up faster than #1, must have less in it. (as expected)

02:30 distillation vessel #2 over 200°, #1 approaching 200°. reduced hot oil increases to 5° increments (Set point increased from 220 → 225)

02:50 Vessel #1 at 210°F and starting to get vessel pressure. (1+ inches)
vessel #2 at 211°F and starting to get d.p. ~~and product~~. (deceptive readings on product tk)
looks like vessels still have some water.

06:30 Running smoothly. Because of bumping problems will let it hum along at 28 inches pressure instead of 30.

07:45 STILL NO OLAF, NO CHANGE OVER.
OLAF, PLEASE SHOW SOME CONSIDERATION AND SHOW UP ON TIME!

07:46½ [illegible] (DPW)

7:30 Day Shift - W. Greenhalgh, G. Mc Donald, O. Rassmus

8:30 - Called to get operators lined up to change out filter material. No operators available today!

9:50 - Raised Oil Temp setting 2°. to 257.

10:00 - Oil heater maxed out at 100%

10:00 - Raised oil temp. setting 2° to 258°

11:00 - Production Rate ~ 0.35 GPM w/o correction.

11:05 - Raised set point to 259°F — This is not wr over or serial writing. Cro with a sin put n

13:15 - Production Rate ~ 0.35 GPM.

13:30 - Received lab Results on last weeks samples. Tritium results not included, but have been requested. Total activity is 110 pCi/gm.

2:00 PM Raised set point to 260°F
14:00

Could not get operator support for filter cleanout today or Tuesda Made decision to do this work on swing shift (2 operators, fitter (c and HPT) tonight.

Received some sample results (see sample 109)

Plan to sample 2 tank cars Wednesday

3:50 PM Heaters and Feed Pump off in preparation for filter cleanout
15:50

Crew, Wood, Henderson 8/20/90

1905 heat on 215°F

1915 - Shut down hot oil at 1600 to clean screen and check on solids - was only slightly filled but the screen lo

Monday 8/20/90 -

Swing shift - cont.

like it was blinded by fines - Tried to clean it but no luck so install 100 mesh.

The rad levels are now a problem !! we had to do a significant amount of clean up ~2 hours. The used filter was up to 170 mr at contact - the feed line is ~5000 counts/min at bottom of flex - the vessel was reading ~3500 counts at mid point inside the concrete pipe.

We need a better procedure to clean the filter if we have to. Should not try to clean baskets - just use new ones.

1930 Back to boiling and starting to make product.

2000 Back to full rate but oil temp is 9°F lower 249 vs 258 when we shut down. ΔT on oil is 14°F vs 13 before shut down thus heat transfer is

Cleared the low level on Tank 276-S-141 by resetting the alarm to 0. this is only in the program and will default back to ~~the~~ old value with reboot.

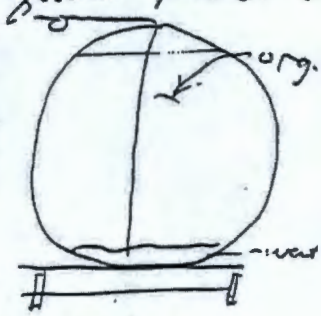
2115 Went over the power calculations to see what was happening. We are running about the max rate per calc. with about 105 KW going into moving the water.

One heater must be out, however, to explain the low amps on two fuses, as the fuses and heaters are wired 3φ. The 11 good heaters are providing ~124 KW* or @ 95% FP = 118 KW net heat input.

RGL

7: GRAVEYARD, Deirell, John & "FREDDY KRUEGER" PAULINA 21 AUG-90

OLAF: When you sample on the tank cars on Wednesday Remember pull samples from both the organic & aqueous phases. We also liquid levels and if possible the volume/level of the aqueous phase. I not sure how you are doing the sampling, but if you're using a pump you can start at the bottom with your sample line and as you pull it up watch for the sample to turn from water to organic and mark the height on the sample tube. Then we would know our water volume, and total volume.



Pirates won again, Mets Lost. Bucs' magic no. is now 39!

04:00 Steady consistant rain. Water trickling into sump, but haven't seen p come on yet.

05:30 Distillation pan filled with rain water & shut down the feed pump a 05:07. Opened drain valve and cleared alarm at 05:14. Tried to restar pump but could not get outlet pressure or inlet vacuum. We mana to loose prime in only 7 minutes. We're trying to locate West a rover (HPT) without much luck. (The normal rover is on vacation an HPT from swing is holding over a double on graveyard) HPT was just at about 04:00.

DARN! Just when we were able to effectively raise the hot oil set and the vessel temp (Boiling point) looked like it was rising. I sho have seen this coming.

The data sheets show the drain pan going from 0.1 to 0.8 in one ten m period. It went to 0.8 on the printout that was skipped by going to 2 interval.

Will leave distillation vessel at temperature. and boiling. Will take hours to bail down.

06:25 HPT office has contacted HPT (MARTA) on the car radio and she's t back as soon as she gets finished w/job. Not looking too hopeful. W keep boiling

06:26 HPT showed up. We reprimed the pump (used 3+ gallons)...

Could not find key to tank fence. Is there a key in anyones pock swing shift? (last ones to go in area). If you have to get in the fence don't have a key just lift the post up and out of the chain

An interesting thing happened when restarting the feed pump. The transfer pump was also running and when it shut off the feed pump also shut off. (Sump pump was also running). There were no alarms that would shut the pumps down and the feed pump restarted with no problem when switch was turned off → on.

LEFT drain valve on distillation pan. OPEN. WHEN RAIN STOPS CLOSE THE valve. so that we have leak detection capabilities again (per procedure).

07:30 Day Shift – O. Rasmussen, G. McDonald, + W. Greenhalgh

9:30 Vessel Press. < 28, so increase Oil Temp. set poin 1° to 260. — Cancel That — heater maxed out at 100% + Set point is already 261.

9:35 Production Rate ≈ 0.35 GPM w/o correction.

10:20 heater dropped off to 95%, so Oil set Temp raised to 26

10:40 Vessel Pressure down to 26, raised Oil set Temp. to 270.

10:45 Level in rain water tank cor 3802 is 6 inches.

11:00 Production Rate still ≈ 0.35 GPM, but appears there may be some organic coming over.

11:00 — Rain lifted; closed drain pan valve.

11:05 Vessel Pressure 32.8, so turned setting down to 268.

11:15 Vessel Pressure unsteady, reduced Temp. setting to 266.

11:20 Vessel Pressure 35, reduced Oil Temp. setting to 264.

11:55 Vessel Pressure > 35, reduced Oil set Temp to 259. The vapor Temp. out of vessel 2 is below 200°.

12:00 Vessel Press > 35, Oil Set Temp reduced to 257.

12:00 Oil Temp. setting reduced to 255.

1:00 Oil Temp setting raised 1 deg to 256.

1:05 Reduced setting to 255.

1:30 Reduced setting to 253, vessel press. > 35.

2:00 Production rate up to ≈ 0.5 GPM w/o correction.

2:15 Reduced Temp setting 2 deg to 243, Vapor Pres. for both vessels < 200°.

2:30 Increased Temp setting to 244°, Vessel Pressure 31.4.

8/21/90 Tuesday

(Days/Continued)

14:40 - Production Rate ≈ 0.57 GPM w/o correction
14:50 - Reduce Oil Temp. setting to 243, Vessel Press. ≈35.
15:00 - Temp Setting reduced to 241°, Vessel Pressure ≈35.

We are feeding organic into the vessel #1 now, with water still on bottom of vessel, so system is very temperature sensitive - boiling off azeotrope. 1° will make a significant difference.

Tank car sampling is scheduled for 1:30 pm tomorrow

RGC
Crew

Swing shift Cowan, Wood, and Henderson

1600 Set point on hot gas reduced to 240 ex pressure #1 vessel exceeded 34" H_2O runni very steady now as we boil off water
RGC

1947 Set temp. to 242° F Red

2000 Set temp. to 243° F Red
2118 Set temp to 244° F Red
2137 " " " 245 RGC.
2155 " " " 246 Red.
2205 " " " 247 RGC
2221 " " " 248 RGH
2236 " " " 249 RGH
2253 " " " 250 RGC
2258 " " " 251 RGC Very small ~~as~~ cha in pressure for increase in Temp - this must be end of Water.
2305 Set temp. to 253° RGC
2320 Set temp to 254 Red

BB

GRAVEYARD : John, Durell, Pav. (22 AUG 90)

Looking over previous data for day shift & swing to see how vessel #1 reacted to organic/water phase. Since there is no pressure sensor on vessel #2 we will go by the demister d.p. Based on previous shift data, if we keep the d.p. about 4 inches or less we should be ok. Will increase hot oil temp based on d.p. and watch overflow temp.

00:30 Raised hot oil set point to 255 °F
00:40 " " " " " 256 °
00:45 " " " " " 258
01:00 " " " " " 260
01:25 " " " " " 261
01:43 " " " " " 262
01:55 " " " " " 264
02:15 " " " " " 265

Limiting factor is still vessel #2. Vessel temp up to 215 and demister d.p. at 4 inch. Vessel #1 is filled with organic and at 251 °F but not boiling much off. (36° difference in vessels). At current rate (146 gpm) it should take about an 8 hour shift to boil off the water mix in vessel #2. Hopefully we be full throttle about the start of dayshift.

02:48 Raised Hot Oil set point 266 °F
02:56 " " " " " 267 °F
03:04 " " " " " 268
03:11 " " " " " 270

03:17 #2 vessel temp starting to climb quickly 216 to 223 in last 10 minu running out of water. Will start increasing oil to start #1 boiling over.
03:22 Raised oil set pt to 272.
03:30 " " " " " 274.

#1 vessel should be boiling off the hexane component of the organic mix and concentrating the NPH & TBP. Meaning that #1 vessel boiling point should be constantly increasing on us

03:40 Raised set point to 276 still not boiling off much from #1, #2 @ 23.
03:55 " " " " 279
04:05 " " " " 281 vapor press in #1 rising
04:15 " " " " 284
04:30 " " " " 287
05:10 " " " " 289

Pressures have been fluctuating between 24" and 6" on vessel #1. Increased feed flow rate to assure seal loop stays filled

CM
Red RGC

Tue. 8/21/90

GRAVEYARD (CONT'D)

05:15 raised oil temp to 291°F
05:27 293
05:37 296
05:47 299
06:15 303 All pressures remain low
06:25 307

Just realized ~~that~~ while looking at sample results there was no feed/product samples taken ~~during~~ since 14 AUG 90. Those were only samples taken of aqueous phase. I'm assuming samples will be taken on day shift when we have an HPT.

OLAF Please call Julie Ring (6-8162) and let her know total tritium inventory for Tank 141. The best data appears to be Malin Wallers which you have a copy of. (I don't). Please call Julie today with this info.

06:40 Raised oil to 311°F

At these temperatures we are probably only distilling the lighter fraction (hexane, boiling point 241°F) its going right thru the system at a rate of 2.5 to 3.0 gallon every 10 minutes. If were to distill anything else we'll be at a lot higher temp. (This is just enough to keep some pressure in the vessels.

06:50 raised oil to 315°
07:10 " " 320

07:30 Day Shift 8/22/90 – O. Rasmussen, G. McDonald, W. Greenlaw

07:45 – Vessel Press, & ΔP's all low, Oil Set Temp. raised to 325, heater 71%
7:50 – Rain Collection Tank (CP-3802) has 6 3/4" of water in it.
8:00 – Raised Oil set Temp to 330. Production Rate < 0.3 GPM
8:10 – Raised Oil set temp to 335.
8:20 – Raised Oil set Temp to 340.
8:30 – Raised Oil set Temp to 345. Production Rate ≈ 0.35
8:45 – Raised Oil set temp to 350. Production Rate ≈ 0.45
9:20 – Raised oil set temp to 360. Production Rate ≈ 0.60
9:45 – Raised Oil Set temp to 370. Production Rate ≈ 0.65
10:10 – Raised Oil Set Temp To 375. Production Rate ≈ 0.67
10:35 – Raised Oil Set Temp to 380. Production Rate ≈ 0.72

Read RGC

Wed 8/22/90

Day Shift (Continued)

11:00 - Raised Oil set Temp. to 385. Production rate ≈ 0.81
11:20 Raised " " " " 390. " " ≈ 0.9
11:45 Raised " " " " 395. " " ≈ 0.92
12:30 Raised " " " " 400. " " ≈ 0.97
1:15 GM sampled rain water in tank 3802. It is listed as Hexone sample 33 (Lab No. E 2030).
1300 1:20 Production Rate is ≈ 0.95 GPM.
1:30 Sample Product liquid. It was essentially all organic. The sample No. is Hexone 32 (lab. # E2029).
1:30 Raised Oil Set Temp. to 405.
1400 2:00 Raised Oil Set Temp. To 410, Heater is bumping up close to 10
2:30 Production Rate ≈ 0.92 GPM, Oil Set Temp. raised to 412.
1500 3:05 Vessel Press. 33, lowered Oil Set Temp to 410. " to 415 at
15:20 Vessel Press. 27, raised Oil Set Temp to 413. RGC

RGC RGH RGH
Swing Sh. ft. - Crowe, Wood, Henderson

1558 Reduced set temp. to 409°F, RGC.
1555 Raised temp. to 410°F, RGH
1600 Change recirculation on feed pump to reduce flow through screen. feed pressure 8 → 4 psig RGC

1716 Things really went strange this last hour. The Temp which had been up at 377°F in #1 vessel and 407°F oil temp just started to drop - the pressure went high with the heaters off. The ΔP on #1 demister is now 730" H_2O the heat is off and we are in deep trouble to get back up - It would seem that the solution in both vessels did a roll over which may be the cause of the high ΔP on the mist elementor in #1. - the system heat is off. RGC

1920 The system has been off for ~2 hours and the vessel pressure is still neg. RGC

RGC 11 WT

Wed
8/22/90 Swing Cont

1930 We are out of ideas on what could cause such a rapid plugging of the demister

- Check the ΔP & Pressure transmitters - they are OK as are the lines into vessels from the transmitters.

- turned up N_2 to Vessel pressure transmitter and it blew out the overflow trap leg on #1 vessel

- Pulled the last 130 logs from the computer and calculated rate of changes and values for Temp. and pressure. Just running OK at 407°F oil temp then high vessel pressure rapid temp drop followed by rapid rise in Demister ΔP on #1 (not #2), soon are vessel pressure except for 1 or 2 inches was in #1 Demister pressure drop even with all heat off! RGC

1950 - After looking at data - this could be a roll over of different temp/comp. material in the vessel with lighter-cooler heavier rich top mixing with TBP rich hotter bottom but why plug the demister?

Could this be TBP decomp. catalyzed by H_3PO_4 formed by TBP breakdown?

2000 Will try to reprime pump and see if steam might restore ΔP on demister. Vessel temp down to 230°F

2130 Vessel cooled to ~212°F, used 2.0 gal H_2O to prime pump. Suction higher than it was before - up to ~17" Hg

2143 Raised temp. to 285°F RGH
2155 " " " 290°F

(2

2208 Raised temp. to 295°F

2300 Demister is plugged again - as soon as we started to get ≈ .3 gpm of product the demister ΔP went Max. Cut back heat but no good still cuting in & out on feed pump - Heat off and still no good - lost seal - lost pumpprime - Down again.

Looks like it is vessel and demister changing time ~~[illegible]~~. There goes at least 2 weeks production.

I suggest we just heat ~~the~~ the system and dry out the vessels per SOP

Heater on and holding at 260°F

GRAVEYARD (LITERALLY) John, Derell, Pav.

Holding at 260 Hot Oil and reviewing each data logger printout since yesterday. It appears we were at our peak operating conditions at 11:00 (am) with the hot oil at 383 and distillation vessel #1 at 356 (and rising), vessel pressure at 23.2 in and production at just less than 16gpm. (which is as good as we got with tank 141!) Unfortunatly instead of fine tuning, temperatures were raised ~~each~~ in rather large increments. My further observations and feelings are not printable.

02:00 Will slowly bring temperature up on vessel #1, to see if we can improve the demister ΔP. Hopefully some warm vapors may help clean the element.

On a good note. There was a fairly impressive display of northern lights on the drive in (10:30 to 11:30 p.m.). Too much light here to see them tho.

Talked to Joel Eucher (our CAM) this morning and we received another $100K. However, we should be have run out of money this week, and were spending at a rate of $25K per week. So at normal operating expenses (which we aren't have) we would run out of money about Sept 21 and have to shut down and not charge to the project for the last week & a half of Sept. (Total Quality thru better management?).

GM

GRAVEYARD Thursday 8/23/90

02:50 Raising hot oil in 2° increments and letting stabilize now at 272° Entire #1 vessel and d.p. are rising slightly @ 3.8 to 4.1 inch.

03:00 Started ~~getting~~ some ~~product in tank~~ when vessel #2 reached 265°. (Therefore we ~~have a fraction that boils off at that point~~).
(PREMATURE conclusion product tank back to original volume.

03:55 Oil temp set point 290, distillation vessel #1 270°, vessel press 5.0, d.p 4.9 vapor out #1 temp shooting up from 61° to 122° this thermocouple is on the pipe inlet to the condensor. So we are vaporizing something at 270° and it is passing thru the demister.

Also shut off the "stadium lights" and wandered out by the last rail cars to witness spectacular northern lights, whole northern sky was filled with fast moving waves of light, very very faint coloring. Ah the joys of graveyard!

04:40 Now at hot oil 309°, vessel #1=239°, vessel #2:285°, vessel #1 press = 12.9 in, DP #1 = 8.1 we're receiving product into tank, and the ratio of #1 demister d.p to #1 vessel pressure has dropped from 0.98 to .63

04:55 vessel #1 temp: 293° and pressures are dropping down to 9.7 (vessel) & 6.0 (D.P. #1). must have boiled off that cut. will heat up to next cut.

05:30 Eastern lights are fairly bright now too. #1 demister d.p. is running about 1/2 or less of vessel pressure. We're cleaning or boiling off whatever is on the demister I'd say lets go for it in the morning; reprime the pump flush some water in the vessel and start pumping again. Crew will be back out at 8 Am (dedicated individual) we'll decide then.

6:20 we've boiled-off 20.6 gallons. and

07:30 Day Shift 8/23/90 - O. Rasmussen, G. McDonald, W. Grea

System down, demister plugged or otherwise not functioning We are to prime pump, add new feed & bring up temp slowly and try to force out overhead material if possible.

Day Shift (Continued). Thursday 8/23/90

9:30 - Took sample of the product tank, sample No Hexane 35 (E20
- Sample log has been moved to the back of the Book -
11:40 Hot oil Temp in ~~vessel~~ #1 still 252°; we are waiting for Temp to cool down so we can add water. Vessel Temp #1 is 251°.

13:45 Vessel #1 Temp & oil Temp 230-232, Primed pump and started Feed pump.

13:50 Vessel # Temp 202°, pump suction high 18 to 20.

14:05 Set Oil Temp setting to 220 Oil Temp is presently 216.
14:15 Oil Temp decreasing, the controller somehow didn't tip in, it is heating now.
14:30 ΔP in vessel is ≈ 30, oil Temp setting reduced.

14:45 — Feed pump off, no appreciable flow through Demister.

Swing Shift - Dick Cowan + Red

825 finally having some luck steaming out the demister - Pressure down getting some vapor out of demister

1827 Oil Temp up to 260°F

1845 oil temp up to 270°F pressure < 1" H_2O
1900 oil temp increased to 280°F
1910 oil temp increased to 290°F
1923 " " " " 300°F
1935 " " " " 310°F
1945 " " " " 320°F
2000 hrs " " " " 330°F
2015 oil temp increased to 340 - this is where Paul was at start of day shift - plan to continue at 5° pump here on and hold #1 V.P. 10-15 # H_2O
2024 Oil temp increased to 345°F
2030 " " " " 350°F
2040 " " " " 355°F Pressure still low ~ 6.5

RS

02

~~Wednesday~~

Swing (cont) Thursday 8/23/90

2045 hot oil temp increased to 360°F
2050 " " " " " 365°F
2100 " " " " " 370°F
2105 " " " " " 375°F

2110 Some light vapor coming from HV-9 on hot oil system back at the insulation on the inside of the hot oil enclosure. This is most likely from the stem leak and the higher temp is boiling off the collected oil. This should be checked when we go down

IMP!

2115 hot oil temp increased to 380°F
2125 " " " " " 385°F
2135 " " " " " 390°F

2140 Still getting a ~~lot of~~ smoke/vapor from oil heating unit! also at end of heating unit - placed a small pan under to see if it drips.

2142 hot oil temp increased to 395°F still have trouble getting any vapor flow pressure still low (2.1)

2148 Oil temp raised to 400°F
2155 " " " " 410°F
2207 " " " " 420°F (New ground for the fry
We're about ready to throw in the French Fries
2217 " " " " 430°F
2226 " " " " 440°F
2240 " " " " 450°F
2255 " " " " 460°F Top of the hi

System pumped out to rail car still getti 1.2 gpm

2330 The plan is to continue cooking the vesse at 460°F until swing shift.

GRAVEYARD, MOE, LARRY & CURLEY (24 AUG 90) PAV, DDP, JWB

Well know that we've evaporated to dryness when
1) Hot oil in = Hot oil out = Vessel temp. (or close)
2) Vapor out for 1 & 2 = ambient temp. (slightly above ambient)
3) Vessel pressure = DP #1 = DP #2 = Ø. 4) No increase in product tank
and we've been there for a shift.

Remember, Any residual hexane in the vessels will require us to take the vessels and reconnect them and reheat them. Evaporating off the hexane is a requisite for any form of land disposal.

00:30 Vapor out (#1 & #2) temps are dropping quickly, 36° & 23° in the last 20 minutes (respectively). Good sign.

03:00 Vapor out temps have been slowly climbing up the last hour. Could be from heating up the steel flex. We're still getting product: 4½ gallons in last hour. Wonder what it is. It would be interesting to get sample late on day shift to see what this high temperature cut is.

When we decide what is happening with the system (probably swing shift), give me a call and leave a message (38-248-8737). Otherwise I'll assume to come out on days next week to set up the vessel change out. Bob Cash will have to work out techs schedule. (I talked w/him at 01:00 this morn)

OLAF: PLEASE CALL JULIE RING WITH THE INFO SHE NEEDS, SEE LOG ENTRY ON PAGE 76 (AUGUST 21)!

A general note on log entries (from something on the wall)

Knowledge not shared is energy wasted.

Never mind Julie called n.r. and I gave her the data. PAV 08:00.

Here are some things you can rely on... I NEED TO get back into town on time today to meet some friends and head to Portland. Naturally I can rely on OLAF to get lost on the way to work and be late! ~~OLAF do you ever consider others~~?
(sorry I shouldn't put that in the log)

JWB. 1/11

Swing shift Friday 8/24/90 RJC, RGC, ERW

1630 Increased N_2 to #1 vessel to purge out vapors in the distillation system & provide heat to the demister elements

7:45 pM = 1948 Hrs RED CHECKED BOTH EVACUATION HOR BOTH A.O.K.

1955 Oil #1 in 457°F #1 out 455°F Δ=2°F Vessel #1 388°
Oil #2 in 457°F #2 out 454°F Δ=3°F Vessel #2 445°-444°

ΔP #1 Demister 14.2 inches ΔP #2 0.2" 15.6" ΔP system

2008 ΔP #1 is partially caused by high nitrogen purge - RGC will lower N_2 purge to 0 ΔP drops to 0 (zero). System pressure drops to 0.3".

2105 Still getting vapor off the hot oil system three ~~leaks~~ leaks observed are
1 - HV 9 packing
2 - Pump discharge
3 - Top heater assembly flange

This must be fixed before restart.

2235 CUT OFF HEATER - START COOLDOWN - PRINTOUT JUST BEFORE ~1M CUTOFF

2248 Pump off on Oil system

2230 Dewar No. 2 value out with 50% remaining and No. 1 is value in service with about 92% full. Cover gas on tanks 141 & 142 is setting on 0.24 in H_2O.

DAYSHIFT TUESDAY, AUGUST 28TH, PAULINA

On monday we opened both vessels at the spare 3/4" port on the top of the vessel. Larry Martin (from personal protection/industrial hygiene) sampled the vapors on each with the following results;

	Vessel #1	Vessel #2
O_2	1%	1%
Organic Vapor	2,000+ *(2900) ppm	2,000+ *(2400) ppm
Hexane	50 ppm (± 50ppm)	50 ppm (± 50 ppm)

* Organic vapor analyzer goes to 2,000 ppm, readings in parenthesis are approximated ~~and~~. Analyzer is not as accurate above 2,000 ppm.

Hexane vapor is by using a ketone drager tube with 100ppm as smallest division. The #1 vessel sample showed a possible interference due to NH_3.

Also, measured the sludge depth in the tanks using a 3/4" stainless tube (end hammered closed and length marked along tube) No build up was noticed in vessel #2. In vessel #1 the rod ~~and~~ hit something fairly solid about ~~10~~ 8 to 10 inches from the bottom. When the rod was pulled there was another 7 or 8 inches of "dirty oil" on the bottom of the rod. From this it appears we have a build up of rust/sludge at the bottom of the tank and 7 or 8 inches of a dark organic liquid (with a high boiling point) also on top of the sludge. Also possible that there is no hard layer and that the rod was hitting a heating coil (crossover pipe at bottom between inner/outer coils)

10 AM walked down system, checked all pressures etc usual inspection. Everything ok, will have to change over dewars about the end of the week.

DAYSHIFT WEDNESDAY 29 AUG 90 PAULINA

08.00 Walked down area. Alls well. N_2 demand regulator crept up to 0.28 inch, reset at 0.22. Dewars are at 60% & 40%

JM

THURSDAY 30 AUG 90 PAV, COWAN, etc.

Afternoon: Disconnected overheads flex line from demister housing and blind flanged. Inside of demister housing and elbow coated with a black "carbon" powder. Willy said it looked like his exhaust pipe when when he had the wrong jets in a carb and was running rich. Don said that the black powder was in the hose but didn't appear to be in the stainless flex. (?)

Disconnected the demister drain line and hand pumped liquid from the space between the demister flange and drain line. Pumped out somewhere between a quart & a half gallon of dirty black solvent. Not looking for the demisters either were bypassing or something getting thru.

Changed out inlet feed filter. It was 2/3 full of "Black Slime". Removed the 100 mesh screen and placed in the temp storage drum. There is some free liquid that wouldn't drain. Placed filter & xs liquid in to scrap can with vermiculite.

Smears and carbon in demister housing didn't show greater than background.

~~MONDAY~~ TUESDAY SEPT 4, 90

MIKE BUSSART, THE LAGGERS PLANNER/SCHEDULER, CALLED TO LET US KNOW THAT WE CANNOT REMOVE & INSTALL THE INSULATING BLANKETS OURSELVES. GIVE HIM A CALL & LAGGERS WILL BE HERE!

Wednesday Sept 5, 90

Checked the flamibility of the liquid removed from the demister and just got a reading ~10% ~~organic vapor when checked~~ RGC LFL at ~80°F Thus we should not have any explosive gas in the vessels

Thursday Sept 6,

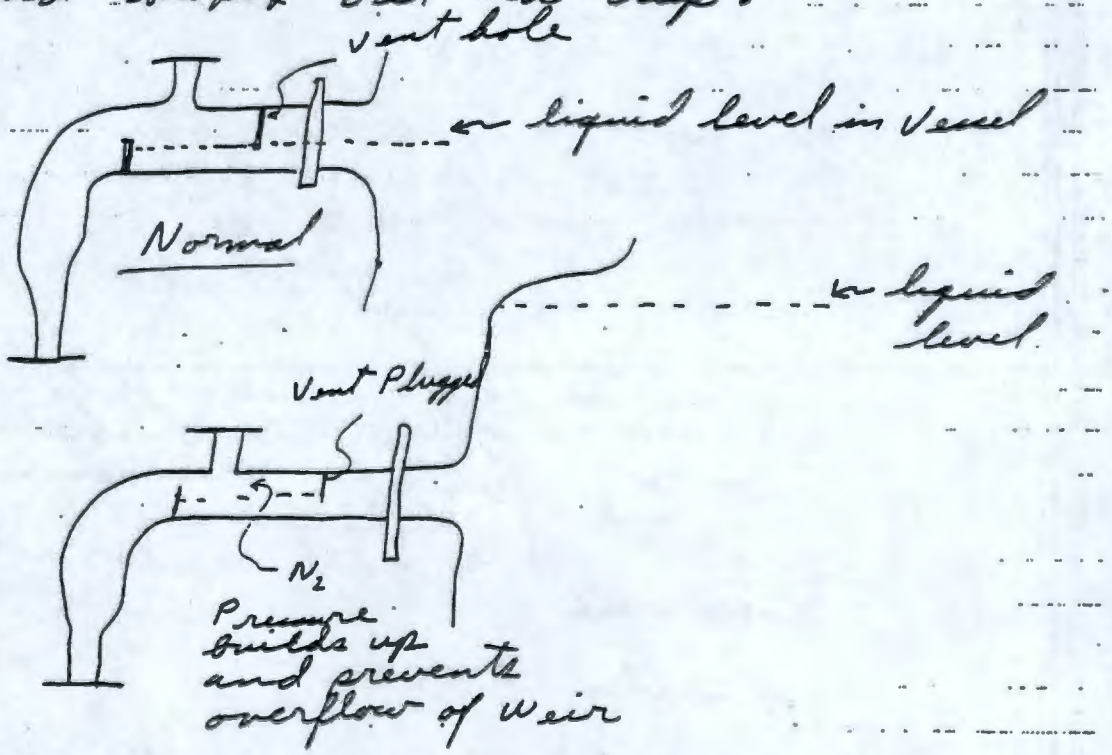
Completed assembly of Demister and refilled hot oil system after replacing pump discharge gasket and tighting

Sept 6 cont.

The stem on HV-9. Also tighten the flange on the upper heater. Completed heat up of oil system to 235 with the steam elimination valve open - need to reset valving. RGC

9/7/90 Friday

Started heat up of system - primed Feed pump filled vessel but got high ΔP on number one demister again! ΔP would cycle and the overflow line would become hot. I am convinced the problem is the small (1/4") hole which vents the overflow weir chamber to the vessel. If this hole becomes plugged the vessel will fill about 4' above the vessel inlet before the pressure builds up and blows out the trap.



LM

9/7/90 Friday cont.

- Action will be to boil out enough liquid with the feed pump off to be sure liquid is below the feed Tee.

Remove the 1" flange monday and use the special tool to clean out vent and restart monday

1415 ~~14~~ The computer went down when the power "blinked" due to storm.
RGC

9/10/90 Monday

845 Cleaned out the vent hole, it was not a hard plug and it was not obv that it was plugged. Some tar and crud was on the tool and seemed to come from the top of the feed tee near the vent - hope its clean and clear

915 Started up the system. Primed feed pump using 7 gal of water, will heat to ~210° and hold for an hour or so

1300 Water in power and ΔP has started to Drop, oil temp increased to 245°F. Starting ramp to operating steady state at about 400°F oil temp

1325 increased to 250°F
1333 increase oil temp setpoint to 255°F
1338 increase oil temp setpoint to 260°F
1348 increase oil temp setpoint to 265°F
1358 increased oil temp set point to 270°F
1410 increased oil temp set point to 275°F
1430 increased oil temp set point to 280°F
1450 increased oil temp Sep Point to 285°F
1530 increased oil temp set point to 290°F RA.
1540 " " " " " " 295 RGC

9/10/90 Monday

1610	increased	oil	temp	set	point	to	300 °F	RGC
1620	increase	oil	temp	Set	Point	To	305 °F	RGC
1642	increased	oil	temp	set	point	to	310 °F	RGC
1700	"	"	"	"	"	"	315 °F	RGC
1720	"	"	"	"	"		320 °F	RGC
1743	"	"	"	"	"		325 °F	CM
1800	"	"	"	"	"	"	330 °F	RGC
1817	"	"	"	"	"	"	335 °F	RGC
1827	"	"	"	"	"	"	340 °F	RGC
1837	"	"	"	"	"	"	345 °F	RGC
1847	"	"	"	"	"	"	350 °F	RGC
1910	"	"	"	"	"	"	355 °F	CM
1920	"	"	"	"	"	"	360	RGC
1940	"	"	"	"	"	"	365	
2000	"	"	"	"	"	"	370	RGC
2012	"	"	"	"	"	"	375	RGC
2030	"	"	"	"	"	"	380	
2050	"	"	"	"	"	"	385 °F	RGC
2123	"	"	"	"	"	"	390 °F	CM
2140	"	"	"	"	"	"	395	RGC
2210	"	"	"	"	"	"	400	RGC

The operating temperature is almost back to the same value where we had the problem on 8/22. The fix of cleaning the hole in the feed tee seems to be working.

Note!! If the ΔP on #1 demister alarms the vessel may be overfilling as the ΔP also reads level above the feed tee. Shut off the feed pump!

The pressure and temperature seem to have started to cycle some - I suspect this is slugs of feed going in - I suggest ~20 pressure average will prevent higher than set pressure. This is 9-10 °F Δ on inlet/outlet oil temps.

CRW

9-10-90 Monday (cont.)

2245 To try to reduce cycling the feed was reduced to give 2-3 PSI on the feed pressure gage. I increased the set point on the hot oil to 402°F

Still getting some smoking on the hot oil system - fix the big leak on the pump and the heater but the leak on HV-9 is not completely stopped. New leak 3 on the orfice plate flanges. Could shut down heater and pump long enough to have Don or Carlos tighten on day shift. R.G.C

QRW

9-10-90 Grave yard. Red Wood, Will Greenhalgh & Olaf Rasmuss

23:30 We went out and looked at the oil heater. There is alot of vapor coming off of it. It appears to be leaking in at least 2 locations. We certainly do need to tighten it down if possible on day shift. The smoke is not that much of a problem, but we don't want to ~~hide~~ any potential risk of a fire.

9/11/90 0:00 We checked the waste being generated. One drum is appar full. We need to open the lid, make an inventory if necessary seal the bag, clamp the lid, and bolt it down! When you get finish date the drum, and place an inventory (waste inventory sheet) inside a plastic cover and tape it to the drum so we can get it disposed of.

for day shift

0:20 The vessel pressure is cycling alot. It got down as low as 13 and as high as 26, but no temp change have been made. Product rate ~ 0.8 GPM.

0:50 Dropped oil temp. set point to 397 to reduce vessel #1 pressure which gradually crept up to 30". Product rate = 1.01 gpm

1:10 Dropped oil temp another 4° to 393 as vessel pressure reached 30

1:20 The production rate is very close to 1 GPM.

1:30 Vessel Pressure 30.2, dropped Temp. setting 3° to 390.

1:50 Dropped temp. to 385°F. Vessel Press. was 30 and Demister No.1 press was 5.9. Red

2:08 Dropped temp to 380°F

2:15 ΔP1 >7, dropped Temp to 375°F.

2:18 ΔP1 rising, dropped Temp to 365°F

2:20 ΔP1 not responding (7.6) so turned Oil Heater off,

2:25 ΔP rising, Turned pump valve so that ~~[illegible]~~ most liquid is recycling rather than going into vessel.

2:35 ΔP still rising, Turned heater back on adjusted setting to 330° (Temp is ~ 225°).

2:40 Adjusted Temp. to 345° to Try and dry out demister.

4:00 Temp setting adjusted to 365°

4:15 temp setting " 367° Vess. Press, 29.7 No.1 Demist 21.0 inches

4:21 temp set to 370°F Vess #1 - 29.7 Dem. #1 21.0 in

4:32 Temp set to 375°F Vess #1 29.5 Demister #1 - 21.0 in

4:50	Temp.	set	to	378,	Vess #	29.5	Demister #1	20.6
5:05	"	"	"	381	"	29.5	"	20.5
5:20	"	"	"	385	"	29.6	"	20.4
5:52	"	"	"	390	"	28.5	"	20.0
6:10	"	"	"	395	"	29.5	"	19.9
6:30	"	"	"	400	"	29.6	"	18.9
6:45	"	"	"	405	"	28.8	"	18.4
7:00	"	"	"	410	"	28.4	"	17.0
7:07	"	"	"	415	"	28.4	"	16.5
7:20	"	"	"	420	"	29.2	"	15.6
7:37	"	"	"	422	"	26.8	"	13.0

9-11 11:50 Demister D.P. continued to drop to ~6-7" then held steady for a couple of hours. Increased oil temperature to maintain boil off. Decided that level in vessel was likely well below the weir level and increased feed flow (from possibly 0). Increased feed flow resulted in vessel increased pressure (as expected) and reduced oil temperatures. Tried to adjust feed flow to steady state in/vs. out, but level increased to ~18" by noon. Returned pump to total recirculation to boil off inventory.

12:10 Vessel pressure and demister D.P. #1 are dropping steadily. Raising oil temp

12:40 Demister D.P. dropped to 7" by ~12:35. Attempted to increase feed flow, but we seem to have lost over prime. Could not get over ~3 psi on pump discharge with recirculation closed, and ~5 inHg on suction. Shut off pump and set heater to 210°F to cool down system for re-prime

Sampler: Sampled product ~11am (archive + spl.). Sent to lab (see spl

Gordey and.

2015 Oil pump shut off — lost of power to main panel on, Stop pump on. Egd.

9/1 OPR DAY SHIFT — TESTED FEED PUMP WITH WATER (ONLY). PUMP WORKS FINE. ARRANGED OPERATORS, PIPEFITTERS AND HPT (ROD) FOR SWING SHIFT TO INSTALL NEW WEIR PRESSURE EQUALIZING LINE, CHECK PUMP SCREEN, AND TIGHTEN BOLTS ON ORIFICE PLATE IN OIL SYSTEM.

3PM

WORK PACKAGES — MAINTENANCE HAS WORK PACKAGES FOR EQUALIZIN. LINE (NEW) AND FILTER REPLACEMENT (OLD). PACKAGES ALSO INCLUDE J-7 FORMS FOR OTHER MISC. REPAIRS SUCH AS TIGHTENI OIL SYSTEM ORIFICE BOLTS.

Swing 9/12/90 Wed.

17:00 Installed the pressure equalizing tube and valves without incident. Also checked the screen but it look very clean so replaced

18:30 Tried to prime, ran OK at first then lost flow at 25" Hg vacuum on feed side of pump. Tried water flush several times but could not establish flow. Bypassed check valve, purged, water back down to tank, and forced water up to the pump but could not get flow back. feed screen must be plugg will try to get operator, fitter, and HPF back.

21:30 Got all the 5 back and changed the strainer - It was plugged with tar, not at all like what we saw the first time. Start up seems to be the problem. OK now Pump at ~15" Hg and discharge steady with bypass set to 5 psig. Tighted two oil leaks - hope it does the job

2140 Starting Operation - Heat on -

2245 Increased Oil Temp 25°F increment to 225 - #1 vessel is lagging way back Will wait for signs of water boiling before any additional increases.

ERW

Graveyard Shift - Bob Paulina, Wilbur Greenough, Red

#1 vessel temp coming up slowly, We will wait for temp to equilibrate before raising oil to

0:30 Vessel Temp is 149 and raising slowly.
1:00 " " " 157 " " "
1:30 " " " 173 " " "

0:50 Observations on a sleepless night (& day):
The are slowly raising the hot oil temp (began shift at 225, now at 241)
The reason for caution is that we know there is a fair amount of water [illegible] temp increases

is dramatically lagging in its response. At the start of the shift the vessel temp was 100° below the hot oil, it is now 50° below. In the past vessel temps were closer and responded more rapidly to changes in the hot oil system. We have one of the following conditions

1) faulty thermo couple – [illegible]
2) fouled coils
3) high heat capacity "liquid" in vessel.

I believe its #3. The thermocouple is not erratic and is steadily climbing and is comparable to outside temp of the vessel (1). The heating coils were clean when I looked into the vessel last thursday. and we haven't processed that much to foul them (#2).

There is not much ΔT across the hot oil coils so we're not putting much heat into the vessels. But, with 50°–60° difference between the hot oil out and the vessel temp it should be taking more heat. We will continue slow increase and see if we start getting vessel pressure as we approach a vessel temp of 212° if this happens we're boiling water & thermocouples are OK

03:15 Appears that the filter is getting plugged again (some black crud could be building up in the vessel??). Cut back on recirculation and pump inlet at 20 psi and outlet pressure is surging between +1 and +5. May have to shut down pump.

03:20 Shut down feed pump, suction was down to ~ 22 in Hg. and pressure was bouncing between 1 and 3 psi. (pump starting to chatter) NEED TO CLEAN INLET SCREEN. ON DAYSHIFT.

03:35 vessel #1 temp 184 vessel pressure 1.6 inch:
vessel #2 temp 236 (dropped to 216)

We are now getting some vaporization in #1 vessel and some product out of #2.

0342 Raised Oil temp. to 247°F Red

04:50 #1 Vessel boiling, with following observations

#1 distillation vessel temp	184°
Vapor out #1	199°
hot oil out #1	245°

#1 vessel temp is definitely biased low, it can't be less than the vapor out pipe temp. Must be between 199° and 245°. Either the thermocouple is damaged (ie. reading at shorted point) or the thermocouple is deep into the coil at the bottom of the vessel & [illegible]. We should replace the thermoco

next down time and not insert it as far. vessel #2 boiling at 218° #1 boiling point should be close to same.

Whatever is in there is boiling off @ 199° to 240°. Pressures (vessel & demister dip) are varying indicating a fairly violent boiling.

BOTTOM LINE

1) we need to clean inlet screen, there are no spares left.
2) when we clean screen we need to send sample to lab and characterize, at least to determine sp. g. to determine if this is just a sludge layer at the water/organic interface or if its settled at bottom.
3) We need to re-evaluate inlet filter, we may be faced with daily shut down to clean filter. (i.e. NO PROGRESS)
4) demister dip & vessel press acting normal. Dick needs to reset #1 dip. alarm from 8 inches.

WB
JBP

5:45 Oil set temp raised to 252°.
0652 Raised temp to 260°
0722 " " " 265°

DAY SHIFT OPR

REMOVED CLOGGED STRAINER ELEMENT. IT WAS FULL OF BLACK "PUDDING". CARLOS SAYS IT IS THE WORST HE HAS SEEN. WRAPPED STRAINER BASKET IN PLASTIC BAG AND PLACED IN WASTE. DID NOT SAMPLE "PUDDING". THE STRAINER HOUSING DOES NOT CONTAIN A STRAINER!

No screen on New dip leg

CHANGED TANK 142 SUCTION TO SHORT DIP LEG (2" SHORTER THAN REGULAR DIP LEG) TO GET PUMP SUCTION ABOVE THE CRUD/INTERFACE LAYER. THE WEIR RETURN LINE NOW USES THE OLD PUMP SUCTION DIP LEG. HOPEFULLY THIS WILL ALLOW US TO DRAW OUT THE 142 ORGANIC LAYER AND LEAVE THE WATER/CRUD MATERIAL FOR LATER.

STARTED SYSTEM BACK UP AT 2:45 PM. STARTING VESSEL TEMP AT 182°.

DID NOT HAVE A CHANCE TO REPAIR #1 VESSEL TEMPERATURE. WILL LOOK AT THAT JOB TOMORROW, PROVIDING THAT NO OTHER PROBLEMS COME UP.

3:30 OIL TEMP SET TO 210°F. GOOD LUCK!

RGL

9/13/90 Thursday Swing RGC - RGH - GT McD

1545 Increased oil temp to 225°F

Looked at the T/C on #1 vessel but really could not see anything that should cause the type of reading we get. Not clear why #1 vessel temp is so strange now. Vessel could be full of black "pudding"? . RGC

1615 Put ice on the T/C where it goes into the vessel - no change in reading thus the T/C is not damaged where it goes into the vessel and the temperature is likely OK. This is strong evidence of a big block of pudding in the vessel We may be in for real trouble now. RG.

1620 We are getting next to no heat transfer in #1 so I see no alternative except to heat up and try to melt this pudding

I increased hot oil set point to 250°F

1700 I increased hot oil set point to 265°F starting to get some boiling U.P. = 2.1'

1800 I increased hot oil set point to 275°F

1910 " " " " " " 295°F

(1850)

750 Power went off @ 1929 - to trailer computer etc.

Power back on at 1950

2000 I increased hot oil set point to 315°F

2030 " " " " " 340°F

2045 " " " " " 365°F

2100 " " " " " 390°F

2135 " " " " " 415°F

9/13/90 Swing Thursday cont.

2200 Made final increase in oil Temp to 460°F

The difference between the vessel and the hot oil indicates we have fouled the coils in #1 vessel. The T/C seems to be good and the vapor temp is consistant with the vessel temp. I do not have any good ideas on what next. With such low heat transfer I doubt we can get much of the liquid to go overhead. Thus the vessel will have a large amount of liquid when removed.

2240 I do not know how the coils could foul so fast. The log shows a ΔT of ~30°F at full power on Tuesday before we overfilled the vessel. We have removed almost nothing from 142 since then but the ΔT is now ~120°F at 70% power.

OMR

9/14/90 Graveyard Bob Paulina, Rod Wood, Wilbur Greenhalgh

2330 Production rate is approx. 0.5 GPM & vessel #1 Temp is rising.

00:00:01 Nice job on switching the drain and feed lines on tank 142! I think this will help a lot in keeping things running. A good idea that was well implemented. Thanks Olaf.

030 - Production rate has dropped to 0.35 GPM

040 Will keep controller @ 460°, we are limited to this by the procedure. Op specs limit to 500°, with auto cut out at 470°. SAD also states the auto cut off at 470°. If we want to go above 460 (but below 470) need to modify procedure.

1:15 Production Rate steady at 0.35 GPM.

3:00 " Rate is up to 0.5 GPM

As stated previously, the thermocouple in vessel #1 reading out is not accurate. Whatever the reason (on the bottom sludge or scorted) the data can not be taken too seriously let alone used to determine coil fouling or tanks of pudding. (It is tough to believe that a solution boils well below the temp of the liquid [illegible]

03:44 WERE BELOW THE 10,000 gallon remaining mark!

04:24 Production now 5.3 to 5.8 gallon every 10 minutes

04:30 TRENDS SO FAR THIS SHIFT:

- Hot oil temps the same, ΔT on #1 vessel hot oil up to 6° from 5°
- "distil. vessel #1 temp" climbed from 355 and stabilized at 364°
- #1 vapor out still increasing gradually (from 226 to 282)
- #2 " " " " " (from 283 to 302)
- #2 distillation vessel temp has dropped as we process more (from 439 to 412)
- pressures rose and have pretty much equilibrated at; #1 = 9.9, #dip. = 1.5, #2 dip.
- production rate still increasing slightly now at 5.3 to 5.8, up from 3.5 gallon/10 min.

We're equilibrating well. At 5.5 gallon/10 min. process rate we would have to process for 13 days yet. (keeping input rate constantly). Looks like at least another month folks, and thats optimistic (i.e. no equipment failure or vessel change out)

MB
OTR
TOP
RCC

9-14-90 DAY SHIFT

SYSTEM WAS RUNNING GREAT WHEN WE CAME IN ON DAY SHIFT. PRODUCTION RATE WENT UP TO ~ .6 GPM.
ABOUT 10:70 - 11:00 THE VESSEL PRESSURE STARTED CYCLING BETWEEN 8" AND 16" OVER A PERIOD OF A FEW MINUTES/CYCLE.
CHECKED WEIR OVERFLOW TEMPERATURE AND IT WAS HOT!
TRIED SOME CORRECTIVE ACTIONS

- REDUCED PUMP DISCHARGE PRESSURE
- REDUCED OIL TEMP
- BLEW OUT PRESSURE EQUALIZATION LINE (BOTH DIRECTIONS)

BLOWING OUT THE EQUALIZATION LINE SEEMED TO STOP THE CYCLING FOR A SHORT TIME, THEN IT RESUMED. STARTED GETTING INCREASED DEMISTER #1 D.P. AND NOTED THAT PUMP SUCTION LINE WAS GETTING HOT (INDICATING THAT PUMP WAS PICKING UP HOT LIQUID FROM THE 142 TANK)
DECIDED TO STOP PUMP WITHOUT LOSING PRIME BY CLOSING PUMP SUCTION VALVE AT THE SAME TIME AS SHUTTING OFF THE PUMP. ATTACHED A PIECE OF INSULATED WIRE TO THE 142 FEED VALVE TO ALLOW CLOSING THE VALVE FROM OUTSIDE THE FENCE. A 1-SECOND OPEN/CLOSE TEST WAS SUCCESSFUL, BUT MAY HAVE COST US THE PUMP PRIME. WHEN TRYING TO RE-START PUMP LATER IT WOULD NOT START TO PICK UP VACUUM.
SPENT AFTERNOON BOILING OUT VESSEL #1.
IN PM. FOUND THAT 142 TANK WAS PRESSURIZED TO 2.5-3.0" WATER. F
EVIDENCE OF PRESSURIZATION WAS INCREASE IN 142 + 144 LIQUID LEVEL READOUT, AND IN PRODUCT TANK LEVEL RISE. TANK PRESSURES ARE NOW

9/14/90 Friday
EXCESS N_2 FLOW ?

TRUCK PICKED UP DEWAR #2 IN PM

VESSEL PRESSURE 0.4" DEMISTER D.P. 0.1" EACH. TURNED OFF HEATER @ 3:20 PM . OIL AT 460°F ON CONTROLLER.

RGC

1700 Swing RGC, RGH, GTMcD

Tried to reprime pump with very small amount of water but could not get it to hold, started but then lost pressure. I have fears for this pump it does not seem to deliver correctly the last few days - it would pull 25" Hg But not ~~it~~ now!

2040 All system down - spent time going over logs for today. Significant ΔP caused by tank car pressure? why I do not know. Start at 11:56 which is after Olaf was working on the ~~vac~~ vacuum pressure equalizing lines.

After looking at data I conclude

1- We did not have a serious problem until after adjustments to flow/temp.

2. There is not a problem with the coil - the cycling may be unavoidable given the ~~we~~ very high oil temp, poor heat transfer, and the volatility of the feed.

3- The operation was gaining rate as we ran thus it may be worth trying to run next week.

4. Something happened to pressurize the tank car about noon today and this was not a direct result of the other problems. This caused a high vacuum pressure and high pressure in the underground

DRP

GRAVEYARD: SUNDAY NIGHT TO MONDAY MORNING (SEPT 17TH)
DEZ WUNDERKIDS: DEIRELL, JOHN & PAV.

PRIMED PUMP WITH water and restarted. Had everything going by 01:00. On priming the pump; The pump had trouble keeping its prime at first so we continously added some water when suction leg to 142 was opened (leg ~~then~~ 1 gpm), then slowly backed off the water to Ø flow as the pump settled out. Put a total of 9.0 gallons of water into the system. Pump will require close surveillance, we can maintain a constant suction pressure of about (-15). but the out let pressure occasionally drops close to zero, and you can here the pump strain at that point. Will try to check pump about every 15 min.

02:05 Oil temp set point now up to 200°; will bring up to 250 then hold for ow. to boil off any water. Pump still looking good, seems to be stable.

03:40 Around 03:00 our #1 vessel press & #1 d.p. both shot up simultaneously to 38 inches (hot oil temp only 200°) from nothing. There appeared to be no overheads, because vapor temp never climbed. Because of this. the feed pump went off. We purged both of the #1 pressure lines, and while pressurizing the weir side must ~~of~~ have blown out the seal leg. All vessel & d.p. went to zero. So we restarted the feed pump. (Due to lightning quick response and a lot of luck, we did not loose our p. We're operating still/again. I'd like to hear some "theories/excuses"... this one, so take a look at the data sheets starting around 03:00... Su. prizes will be given for "most plausible" and "most ridiculous" theories (Judges decision will be final)

04:34 Boiling started. Vapor out jumped from 58 to 182 in 10 minutes #1 vessel temp 194°, hot oil set pt. 233°

05:55 Water appears to be boiled out; distillation vessel temp climbing, pressures drop, about 10 + gallons in product tank: Starting to raise temp. set point for hot oil in 5° increments watching all temps & pressures,

07:30 Hot oil set point now 300°F & RAISING. We've pumped 200 gallons from TK-142. This was mostly topping off the distillation vessels at st. up. Plus we've pumped out the product tank twice. When bringing syst to max temp remember: The higher the temp, the greater the potential for an upset that could shut us down. (also will give minimal increase in productn

7:00 Day Shift: 9/17/90 - Clat Rasmussen, Geo McDonald, Wilber Greenlaw, Ch

7:55 Production Rate 0.34 GPM.
8:15 Raised oil set temp 5° to 307.
8:45 Production Rate has increased to 0.73 GPM
8:55 Raised oil ~~set~~ Temp 3° to 310. UP is 11.7.
9:15 UP is 10.6, raised oil set Temp. to 315.
10:00 UP is 8.5, " " " " " 318.
10:00 UP is 7.8, " " " " " 321. Production rate 0.56 GPM.
10:45 UP is 7.2, " " " " " 324. " " 0.52 "
11:00 UP is 6.8, " " " " " 327, " " 0.50 "
11:15 UP is 7.8, " " " " " 330, " " 0.52 "
11:30 UP is 7.1, " " " " " 333, " " 0.53 "
11:45 UP is 6.8, " " " " " 336, " " 0.49 "
12:00 UP is 6.7, " " " " " 339, " " 0.49 "
12:05 " " " " " 340, " " 0.49 "
12:10 UP is 6.7. " " " " " 343, " " 0.49 "
12:30 UP is 6.4, " " " " " 346, " " 0.49 "
12:40 UP is 6.7, " " " " " 349, " " 0.43 "
12:55 UP is 6.2, " " " " " 353, " " 0.45
13:05 UP is 6.5, " " " " " 356, " " No Reading (Sampling
13:25 UP is 5.5, " " " " " 359, " " 0.40
13:35 UP is 6.8, " " " " " 363, " " 0.42
13:42 UP is 6.7, " " " " " 366, " " 0.42
14:00 UP is 5.8, " " " " " 369, " " 0.44
14:12 UP is 5.5, " " " " " 372, " " 0.41
14:20 UP is 5.8, " " " " " 375, " " 0.39
14:32 UP is 5.8, " " " " " 378, " " 0.40
14:45 UP is 5.5, " " " " " 381, " " 0.40
14:55 UP is 5.8, " " " " " 385, " " 0.40
15:10 UP is 5.0, " " " " " 388 " " 0.38
15:20 UP is 33, " " " " " 391, " " 0.40
15:25 UP is 5.3, " " " " " 394, " " 0.38

Ran steady all shift. Did not adjust pump. Only took temperature up SLOWLY
Just try to keep it going like this at a modest production rate

0530 Swing Shift Dick-N-Dick and Red
1550 UP is 4.8 set Oil to 397 production rate .35
1615 Restarted Datalogger to change title and start new disk. Last weeks was still installed - Note: need to install a new disk each week

9/17/90 Saving Monday RGC, ERW, RGH (co.

1615	setpoint	To	402	VP = 3.9
1625	set point	to	407	VP = 3.8
1642	"	"	412	VP = 4.5
1652	"	"	417	VP = 5.4
1715	"	"	420°F	VP = 6.1
1735	"	"	425°F	VP = 6.2
1853	"	"	427 F	VP 12.7
1945	"	"	430°F	VP 13.6
2100	"	"	433°F	VP 14.4
2130	"	"	435	VP 16.0

2130 The system is operating very smoothly little or no variation in pressures. The temp continues to slowly increase on the #1 vessel. The calculated equilibrium temperature is 383°F but who knows how good that will be.

The heat tranfer has improved remarka with "only" 47°F between boiling an hot oil out. I do not know why the improvement but this is near the 50° used as basis of design limit. The rate is lower than expected but at 0.85 gpm the balance of tank 142 should be processed in less than two weeks of steady operatio or before the end of the FY. This will require perfect operation but it is not yet impossible if we can keep runn

2240 Increased hot oil set point to 438°F the Vessel pressure is 17.0 rate is 8.1 gal/day

2245 After blowing the product line to the tank car the level is 7155 gal. This is likely a bit ~~too~~ RGC high as the water in the bottom does ~~not~~ give a high readin

4/(+2)

9/17/90 Monday evening

At the present rate we will be near the 9500 gal limit by Thursday morning.

We continue to have an oil leak on the hot oil system at the upstream side flange of HV-9. Need to remove lagging to tighten the bolts. Also must have hot oil off, if not cooled.

GRAVEYARD

12:05 ~~Production rate remaining at 8.6 gallons/hr = 0.43 gpm. (not 0.85 gpm) with 9000 gallons remaining thats 15 days of operating time.~~

never mind this is still a 10 minute cycle & if prints out only 2 minutes RSP

Cabo feed to #2, vapor out #2

Vapor out #1 temp is steadily dropping (14 degree / 30 min) since shift change, must be less vaporization. Increased hot oil another degree (up 2° RSP this shift so far).

02:15 Still humming along.. keeping #1 vessel pressure about 20(+) inches. Some difference in tank calibrations, most prior to since pumpout from 142 greater than product tank increase. Since beginning of shift we've averaged 0.91 gpm from 142. and about 0.85 for product.

05:00 All's well. Hot oil set point is 444° (raised 6° so far this shift, in 1° increments over 4 or 5 hours). Vessel pressure remaining about 21 inches and vapor out temps about 310°. Everything is stable so doubt that I'll increase temp set point unless vessel pressure drops. It ain't broke -- So I ain't goin to fix it! We've pumped 323 gallons so far (59 gallon/hr or 0.98 gpm. average for shift. At this rate there's 148 hours remaining, if only we could maintain this rate.. hot oil system is smoking from two spots; from around the insulation wrap on the supply valve (H-9) - not the valve packing (previously - on Friday) and from a flange directly below expansion tank.

07:00 Distillation vessel #2 temp has dropped consistently 12° or less in 20 min no other temps are changing. Vessel #2 is now almost the same as #1 temp. (where it had been running higher)

0730 9/18/90 - Day Shift - Olaf Rasmussen, Geo McDonald, Willow Carter

The System appears to be running smoothly. It is heating alot better than last week oil coils must have sluffed off their deposites. 10° different Oil Temp set point 444.

1150 System still running steadily. Have not adjusted temperatures. Maintaining 0.9 gpm product as indicated on product tank.

Took 3 samples: Archive sample, sample to REDOX lab, and sample for PNL to analyze for hexane, NPH and TBP. Main purpose of PNL sample is to find out what is happening to the TBP. Shipped to PNL (Rich Lucke) about 11 AM.

1300 - Oil set Temp, raised 1° to 446, Vessel Pressure is 18.4, Production Rate
1330 - Production Rate 0.87GPM., Vessel Press. cycling 13 through 20 a back.
1420 - Must be having some weir problems, The return lines are Temp. hot.
* 1520 - Throttle back pump to reduce liquid level in vessel.

Left 1530 Swing shift RGC, RGH, ERW

1 Came in and things were in bad shape but they did get worse. The ΔP on #1 demister went down but the total pressure started to climb. The underground tanks were at 0.9" and then went off scale. The product level went up as did all tanks due to the generation of pressure in the system.

What time did this happen?

The bad things that happenned

- lost the prime on the pump when it shut off at ~38" H_2O in #1 Vessel
- Ran the transfer pump dry and may have damaged it
- Had to shut down the system heaters as pressure was over 38" H_2O

1900 Had to cool off and let all pressure went off before reheating to see if this was done reacting.

2000 Tried to reheat the vessel but the heat transfer was still. 2° ΔT on the hot oil with 110°F between the oil and the vessel, After talking to Pav, decided to cool down and try a restart - this seem to work last time. If we should have another pressurization like this, just sit back and what? Temp.? know?

JGR
RSP
JJP

THE GRAVEYARD CLEAN-UP CREW: Deirral, John & Pav "the wonder dog"

00:50 Letting the system cool down to below 212° so that we can add water. Were dreadfully low on SCWP's (been out of gloves for a few days) could someone on day shift get the cabinet re-stocked? 233-5 is not open on the back shift.

Just some observations; (but not necessarily conclusions)

Going back through the ~~logs~~ data sheets it looks like vessel #1 stopped (actually slowed) boiling about 14:00 (vessel pressures dropped, vapor out temp dropped) the #1 vessel temp. had lowered to 367° this is below the 380° where it had been boiling. The #1 overflow also went up to 140° at this time, this could be because the feed was about that temp (based on the feed pump temperature) tho there was never any fluctuations in the charcoal bed temps indicating we never saw a heavy loading of organic vapor during the pressurization.

02:00 Restarted pump, used 13.2 gallons of water, this cooled #1 vessel down fairly quickly to 90°. Set the pump valve to a little recirculation and an outlet pressure of 5psig and suction, vel less than -15. Suction can get down to -20 if needed but didn't want to work the pump that hard. Hot oil set point at 235° (just above existing temp to start with)

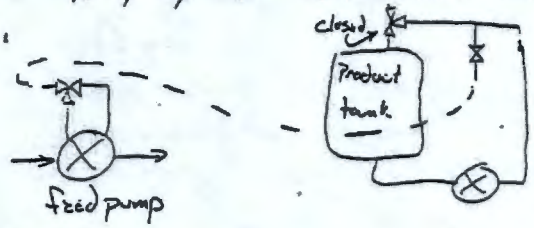
02:25 All system pressures have gone up; underground tanks up from 0.2 to 0.6 and all tank liquid levels are up. I backed off regulator for N_2 demand and system pressure is slowly dropping. Not sure if the two are related since I couldn't hear any N_2 flow at the demand regulator before making a change.

03:00 We have been getting good distillation on #1 at hot oil temp of 240° vessel temp 193° just started to get vapors off of #2 (vapor out #2 temp rising).

03:45 Wed. Sept 19th Cont'd
oil delta T decreasing, vessel temp increasing, vapor out temp decreasing pressures decreasing. I think were about finished boiling water; time to pump up the temps. and get the show rolling again!

04:20 First transfer of product, The pump sounded ok. sounded like a little debris in pump at first but quieted out. HOWEVER --- Its not pumping! we cycled the pump a few times, and tried pressurizing the tank by increasing the N_2 purge dramatically., To no avail. after running the pump for 20-30 minutes the product tank volume continued to increase. At 4:45 we shut off the pump and dropped the heater back to ambient. Looks like we lost the pump when it ran dry last night.
Now the rest of the story. The product tank is full and we need to remove & replace the pump head. there are no isolation values ~~(from around pump tilted on out of adding these)~~ for the pump. ~~or But we is to pressurize the tank and blow~~ liquid out ~~thru sample port and back to tanks via inlet filter housing. This would work IF the pump outlet port isn't plugged~~. Cant pressurize tank because of overflow, would have to pressurize underground tanks etc.

We could use the two sample ports to pump out the tank. We got the fittings together to do this.
Use feed pump suction to pump out product tank ~~and~~ into vessel #1 and overflow.



6:45. Shut off feed pump and closed 142 value via new "wireless remote"

07:30 9/19/90 Day Shift - Clat Rasmussen, Geo McDonald, Wilbur Greenha

We need to repair Transfer pump and deciding best how to isolate the product vessel.

* The maintenance RWP expires tomorrow - Rod Garrett has told his management. We need to make sure we get one drafted and signed (Bob Paulina).

There was a question about the level of Product Tank. It sounded MT to the clang, but full via Temp. contrast. We finally had to dip it with a piece of wood before finding out it was full!

we had the instrument man check and verify level instruments are reading fine.

Afternoon - Set-up to ~~pump~~ pressurize product tank and blow the product liquid to the ~~ta~~ tank return to 142.
Had a problem with connecting tank car line to product tank. Not complete. Everything else is ready for blowing the product tank liquid to the tank car

Swing 9/19/90 Wed.

Repaired the pump with no major problems we used both the freeze plug and the pressure transfer to empty the tanks. In the future I see no problem with just using a freeze plug as this is quick and ~~eas~~ easy. The pump was released and the shop has it. Red plans to order parts to repair plus a new pump head.

@ 2030 started up system, used only 1.5 gal of water - pump output is very low and I suspect we will have to change the pump before too long.

Plan to heat to boil off water then slowly increase to ~350°F.

GRAVEYARD - PNL cannot analyze any more samples for us unless it is an emergency until aft The new year! - [illegible]

As discussed with dayshift wednesday morning: we had a perfect opportunity with the feed pump off to obtain a feed sample. All we had to do was open the feed port & turn the valve to drain the feed line. If we are to characterize what is happening during these pressurizations we need to define the situation with data. such as inlet & outlet samples. The we agreed to take the sample I noticed it was not taken. I am disappointed to say the least. - PAV.

The RWP expires today. I had talked to Rod about it a few weeks ago, he had also talked to Conan and they didn't foresee any problems in renewing it. Dayshift will need to follow up to make sure it occurs today.

TRAVEYARD 20 SEPT 90 (CONT'D)

01:50 Hot oil set point up to 260 running well. Everything is tracking almost identical to the same point during sunday nights start-up. (Sept 17, 06:44). I'm keeping a marker in the data log, which corresponds to the same point during the previous start up/Heat-up for comparison purposes.

Our rail car levels are all low (400-500 gallon range) compared to previous (9,000 gal
Did someone change rail cars? There's no log entries or placarding on tanks, so this shouldn't be the case. N2 purge is at 2 cfh and tmtr valving is ok.
No, were still on #3 rail car and levels returned to normal several hours later
cause of errant readings is unknown.

Note: whenever we do go to the fourth car we will need a pipe fit. because the fill line valve is the one that looked like it was leak. and Don had to tighten the packing nut fairly tight. He had said that we'll have to loosen the packing nut, hit the stem to loosen the tapered stem.

06:30 When the transfer pump kicked on everything hummed along well except for one thing ---- it didn't transfer any liquid. After several varied and assor remedies failed, we got an HPT and took a sample, The sample flow was good, N2 flowed easily to the tank cars, the only thing in between is the check val. A few raps of the check value with the old "manual transfer pump starter" (hammer). I Started the pump and it does transfer. However the pump rate is SLOW. Its taken 1/2 hour to pump down from 51.2 to 13.5 gallons. and its still runnin Its real slow at the bottom of the tank, this pump needs all the head i can get! Pump shut off at 11.8 gallon. The "sample" we pulled was mostly water, and was archived.

07:10 Some differences from the previous start up (with the hot oil in temp of 309°)
- our ΔT for the vessels is slightly less
- our vessel temps are closer to the hot oil now
- Vapor out temps are lower now
- pressures are lower.

Bottomline. What we have in there now is less volatile than what we had earlier in the week. So don't expect the same production rates & temps.

As we bring this up to temp please look over old data so that we may be able to learn from past experience. And whatever you learn is USELESS if you don't pa it on. (i.e. Use the log!)

8,124 gallons pumped from 142
27,399 " total

9/20/90 - Day Shift - Olaf Rasmussen, Geo McDonald, Wilbur Greenhalgh

Transfer pump working okay, but it vibrates and is noisier than before.

0730	Raised Oil Temp To				320,	Vessel Press	7.3,	Production Rate		0.49
0755	"	"	"	"	325,	"	" 6.2,	"	"	0.48
0813	"	"	"	"	330,	"	" 6.0,	"	"	0.5
0835	"	"	"	"	335,	"	" 6.1,	"	"	0.51

0850 Transfer pump working fine and sounds good!

0855	Raised	Oil	Temp	To	340,	Vessel Press.	5.9,	Production Rate		0.47
0910	"	"	"	"	345,	"	" 5.6,	"	"	0.46
0920	"	"	"	"	348,	"	" 6.6,	"	"	0.46
0930	"	"	"	"	351,	"	" 6.6,	"	"	0.48
0940	"	"	"	"	354,	"	" 6.3,	"	"	0.48
0955	"	"	"	"	357,	"	" 6.0,	"	"	0.45
1000	"	"	"	"	360,	"	" 6.8,	"	"	0.45
1010	"	"	"	"	363	"	" 6.5,	"	"	—
1025	"	"	"	"	366,	"	" 6.2,	"	"	0.41
1040	"	"	"	"	370	"	" 5.5	"	"	0.41
1050	"	"	"	"	373	"	" 5.1	"	"	0.42
1105	"	"	"	"	376	"	" 4.6	"	"	0.36
1110	"	"	"	"	379	"	" 4.7	"	"	0.36
1120	"	"	"	"	382	"	" 5.2	"	"	0.36
1130	"	"	"	"	385	"	" 5.4	"	"	—
1145	"	"	"	"	388	"	" 4.4	"	"	0.32
1155	"	"	"	"	391	"	" 4.2	"	"	0.32
1205	"	"	"	"	395	"	" 4.0	"	"	0.36
1210	"	"	"	"	398	"	" 4.1	"	"	0.30

Rich Lucke (PNL) called with results Preliminary: NPH = 11.49%, TBP = 0.6
(product sample)

								Balance Hexane & other		
1305	Raised Oil temp Setting				401	"	" 2.0	"	"	0.18
1315	"	"	"	"	404	"	" 1.7	"	"	0.18
1322	"	"	"	"	408	"	" 2.3	"	"	0.06
1335	"	"	"	"	411,	"	" 2.1	"	"	0.15
1340	"	"	"	"	415,	"	" 1.8,	"	"	0.15
1405	"	"	"	"	418,	"	"			0.14
1410	"	"	"	"	421,	"	" 2.1,	"	"	
1435	"	"	"	"	424,	"	" 2.3,	"	"	0.14
1445	"	"	"	"	427,	"	" 2.6,	"	"	0.2
1455	"	"	"	"	430	"	" 3.6	"	"	0.2
1505	"	"	"	"	432	"	" 4.5	"	"	0.26

13:30 Took product samples. Hexane 50 & 51, Sent 51 to The Lab.

Day Shift

NOTE: We called and talked to Dr Lee Burger, PNL 6-3327 about the TBP distillation. He doesn't think what we are doing with the TBP would be a problem. The TBP is normally very stable except at or near the boiling (decomposition) temperature where it typically decomposes to butyl alcohol and dibutyl phosphate or similar products which would distill over. However, if we want to have our organic feed analyzed for organics to 1% or sometimes a fractional percent with IR, we could probably arrange to have a couple of samples analyzed. He is unsure presently about how fast they could do it however. Also, the ESL Bldg has NMR capability through Tim Franz.

All day long production has slowly declined despite steady oil temperature increased until about 3pm when vessel pressure and demister D.P. came up, along with the production rate.
For a time we suspected that the feed pump was not getting liquid into the vessel (bypass valve is closed with ~2psi pump pressure). Rapped the check valve (~2:50pm) just before vessel pressure started rising although we think this was probably unrelated. Pipeline is cool, indicating flow.

Wrote PCA to cover high-temperature pressure cycling in vessel #1. Please look over, comment (if you want), I'll get signed & released tomorrow. — ANY OTHER CHANGES YOU WANT? ?

Swing Shift Dick-N-Dick-N-Rl

That a boring night - No adjustments to the process this shift, it would have run just the same if we had not been here.

GRAVEYARD, PAN, BIGLIN, PAINE.

04:30

GENERAL NOTE: The system has a long term cycle where pressures and vapor out temp increases then the vessel temp drops, followed by drop in pressures and vapor temps and increase in vessel temp. At this operating temperature the cycle is minor and we avoided the temptation to play with the hot oil temps. Our production rate is satisfactory while maintaining temps & pressures below those of Sept 18th when the "pressurization" occurred, so we will not play with the temps. At the lower temperature (vessel temp) any gas evolution should occur at a slower rate and we have more operating range to

DAY SHIFT 9-21-90 RASMUSSEN & McDONALD

0845 HRS. TRAK CAR 3688 WAS VALVED OUT and TANK CAR 3685 WAS VALVED IN. **NOTE**: Fitters had major problems trying to free rusted-up valve on # 3685

0900 TOOK Product sample Hexane 53 E.2406

1:40	- Raised Oil set Temp. To				435	Vessel Press. = 2.4,	Prod. Rate = .21
1:45	- "	"	"	"	" 437	" " 2.9	
1:50	"	"	"	"	" 440		
2:00	"	"	"	"	" 442		
2:05	"	"	"	"	" 444,	VP = 2.0,	Prod. Rate = 0.
2:10	"	"	"	"	" 446,	VP = 2.0,	
2:15	"	"	"	"	" 448,	VP = ,	Prod. Rate = 0.0
2:20	"	"	"	"	" 450,	VP = 2.6,	Prod Rate = 0.2
2:30	"	"	"	"	" 452,	VP = 2.6,	Prod. Rate = —
2:35	"	"	"	"	" 454,	VP = 3.6,	Prod. Rate = 0.2
2:40	"	"	"	"	" 456,	VP = 3.9,	Prod. Rate ≈ 0.5
2:55	"	"	"	"	" 458,	VP = 1.8,	—
3:00	"	"	"	"	" 460,	VP = 1.6,	

The 460' is the Operating limit; No more Temp. increases will be made

3:20 - There is little or no production rate, Vessell #1 is about 80° below Vessel #2. We don't know why nothing is happening.

System ran well until just before noon when vessel pressure & D.P's started steadily dropping off, along with the production rate. Started raising oil temperature in an attempt to revive the system.

RGC

Swing Friday RGC, RGH, ERW

The system was not producing any distillate. The pump was pumping but slowly only 2-3 psig at discharge. The vessel temp had increased to 391°F. The assumption was that the #1 vessel was empty.

To check this we closed the Feed weir bypass valve to allow pressure to build up for the feed Tee and force feed into the vessel even if

9/21/90 Friday Swing (cont

the vessel was full. (This works because the vent hole in the feed tee weir is plugged). It was assumed that if the feed pump was pumping, the level would build up as indicated by #1 demister ΔP. Then the level could be returned to the height of the weir by opening the bypass vent valve. The overflow line would get very hot as the excess liquid drained back to 142.

In fact the #1 demister and vessel pressure did increase when the vent bypass was closed. But, when the valve was reopened no overflow and the pressures went back to .2-.4 ? the only conclusion is the 3" feed tee is plugged where it goes into the vessel. Most likely where the heating coil runs near the 3" vessel nozzle

After being a bit stagger by this we decided to run for a time with the vent bypass closed and see if feed could be forced into the vessel with less than 30" H_2O. This worked and the production rate come up to ~.6 gpm with 6"-14" head on the feed tee but we never could fill the vessel.

Conclusions:

1. The 3" nozzel into the vessel is plugg with mud and should be cleaned someho
2. The Feed pump is about shot (we did run it by during one of the ~~prob~~ probl times) and the production rate is set by the pump (~.6 gpm is all it will do)
3. We can run as is until we get everyth together for repairs

Pav - I hope the water you use for priming the pump will wash out the mud allow us to operate with the vent valve open. If not we run about 8" ΔP to get the feed into the vessel. I hope you can get the pump started one more time, We will get a new one monday.

We need a holdover to cover day shift if we are running (Dick H will be on vacation). Does Geo want 4 or 8

2000 Heater off
2310 Feed Pump is off & Valve #2 (going to 142 tank is closed)

9/24/90
Grave Yard Shift - Paulina, McDonald, & Greenhalgh

We are going to try and take 2 Feed Samples (for chemist characterization). We will just Archive them for now.
01:20 - Samples Taken - Liquid is dark, but still very fluid.

01:40 - Feed Pump primed and operating - used 3.4 gallons of water. The hot oil system turned on. The Feed line checkvalve is working fine - can hear flow going through it.

01:50 Oil Temp setting 100°
02:10 " " " 125° - Condensers on.

TRYING TO DETERMINE FLOWS BY PIPE TEMPERATURES. THE FEED LINE FROM THE PUMP TO THE INLET TEE IS COLD, SO WE ARE PUMPING THAT FAR. AFTER PUMPING FOR 15-20 MINUTE THE OVERFLOW LINE STILL IS AT AMBIENT TEMP INDICATING THAT THERES NOT ANY/MUCH OVERFLOW AND THE PUMP IS FILLING THE VESSEL (FILL RATE IS UNKNOWN)

02:34 observed the first decrease in 142 levels down 17 gallon in ten minutes
02:44 observed an increase in 142 levels up 13 gallon " " "

03:15 tank 142 has been pumped down from 6602 (@ 01:14) to 6590 for a pumpout of 12 g.
03:20 all the 1" lines are cold to the touch (ambient 64°) except 2: the feed line and the #1 overflow [illegible]

However, the #1 distillation vessel is not rising and the hot oil ΔT is only 1°
seems we have one of 2 situations;
1) the vessel is full of something with a low heat transfer coefficient
2) pump is pumping but nothing is going into the vessel (UNLIKELY)
or 3) we're just not up to a temperature that's accomplishing anything

Vessel #2 responds quickly to changes in hot oil temp, #1 does not.
i.e. hot oil temp set pt is 200°, #1 vessel is 101°, #2 vessel is 164°

P.S. Used 3.4 gallons of water on pump priming

3:45 Oil Set temp. raised to 225°.

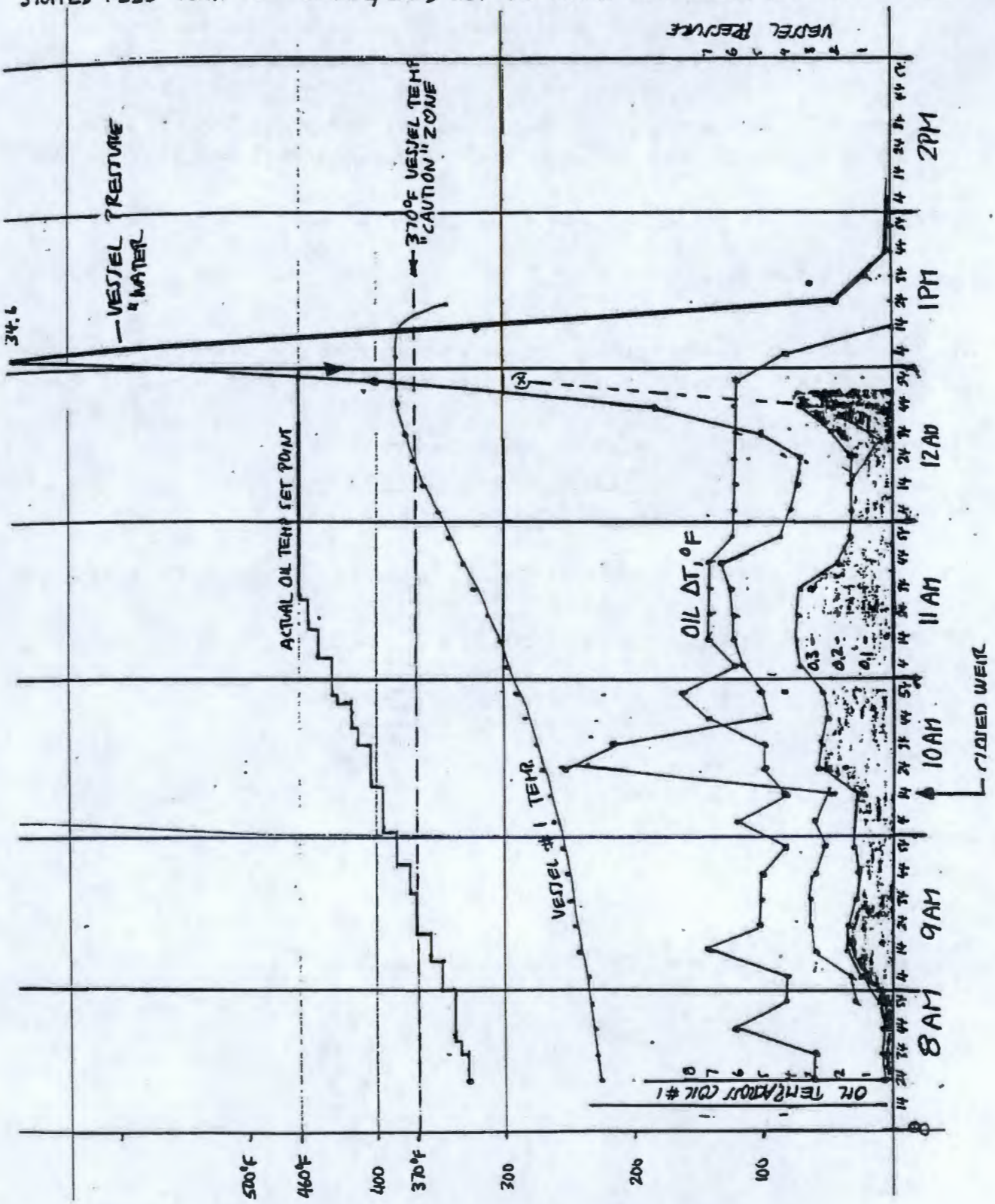
06:40 We are finally getting some vaporization in vessel #1. Hot oil at 29
287 out, vessel temp 194°, vapor out 140°, vessel press. 0.6 inch

Production to date 19,275 gallons from 141
9,430 gallons from 142
28,705 gallon total

Day Shift Olat, Red and George McDonald holding over.

0825	temp.	was	set	330°F
0835	"	"	"	335
0840	"	"	"	340
0900	"	"	"	350
0912	"	"	"	360
0920	"	"	"	370
0938	"	"	"	375
0945	"	"	"	375
0[illegible]	"	"	"	37[illegible]
:018	"	"	"	405
1023	(Closed weir bypass valve)			
1035	"	"	"	415
1040	"	"	"	420
1050	"	"	"	430
1055	"	"	"	435
1110	"	"	"	445
1117	"	"	"	450
1128	"	"	"	460

WELL, WE DID IT AGAIN. HAD OIL TEMP. TOO HIGH WHEN WE HIT THE "CAUTION ZONE" AT 370°F VESSEL TEMP! SHOULD HAVE BEEN AT ~400° OIL TEMP INSTEAD OF 460°. SEE CHART BELOW. KILLED HEAT, BUT TOO LATE. PRESSURE IN VESSEL #1 WENT ABOVE 35" AND INTERLOCK STOPPED FEED PUMP. LOST PRIME & COULD NOT RE-START. NOW COOLING DOWN TO RE-PRIME PUMP.



WE LET OURSELVES BE DECEIVED BY LOW VESSEL PRESSURE AND VERY LOW PRODUCT RATE BEFORE THE VESSEL TEMP ENTERED THE 370°F CAUTION ZONE. THE LESSON: MUST HAVE LOW OIL/VESSEL ΔT WHEN APPROACHING 370°F. WE WENT THRU THE 370°F ZONE FRIDAY, BUT NOTHING HAPPENED BECAUSE THE INLET WEIR WAS PLUGGED. THIS TIME THE CLOSED WEIR VENT VALVE ALLOWED THE PUMP TO FORCE FEED INTO THE VESSEL AND MAINTAIN THE VESSEL FULL, RATHER THAN MOSTLY EMPTY.

OHL RGC BSB

NEW PUMP PARTS ARRIVED THIS SHIFT.

1830 Cooled to ~240°F and restarted pump. No real problems, used 3.0 gal Water. Started heating ~1900 hours. Plan to increase oil temp to 400°F and hold until #1 Vessel temp is steady.

2100 Tried to crack the pump (I suspected it was not pumping). Closed the feed tee vent valve (red handle) and allowed the level to build up.

The first thing that happenned was a rapid buildup of pressure on the feed Tee to 19" H_2O. Then it broke through and went back down to ~~~~ 0-2". After one hour we could not be sure there was any level buildup. Feed pump was very hot. (130°F) and concluded there was no flow. As a last ditch try we iced down the pump, ~~with~~ the pump suction seemed to increase but no induction of flow.

Check Valve could be plugged or ~~stick~~ stuck shut. Our normal priming add water down stream of the check valve and forces it closed.

Thing that need to be fixed

- The pump is not making it and needs to be changed.
- The feed tee is plugged and needs to have a snake run down it.
- There is still one Hot oil leak on the flange to HV-9

GRAVEYARD PAUL, WILBUR, GEORGE

REVIEWED DATA LOGGER PRINTOUTS FROM DAYSHIFT. NO PRINTABLE COMMENT.

00:45 Shut off valve on N2 purge to tee, opened vessel purge line, raised hot oil set point to 405 from 400. When the N2 purge to the inlet tee was shut off pump outlet pressure increased back to 1 to 2 psig. (Still not much pressure) ~~the~~ the pump was fighting the N2 pressure in the weir. Since we are going to shut down anyway, I will run the temp up first and see what kind of process info I can obtain.

02:00 KILLED THE FEED PUMP. IT WAS STARTING TO MAKE NOISE AND THE PUMP HEAD WAS HOT, 132° on data logger.

06:15 We took the hot oil system to the max (slowly) of 460° and let the system equilibrate. The VESSEL #1 temp and #2 temp were 401° & 404° respectively. The vapor out temps for both vessels were essentially ambient (66°), all pressures were essentially zero (true vessel pressure: weir valved out, vessel valved in) and no change in the product tank. This leads to two possibilities that I can see:

1) Both vessels are empty
2) If there is any liquid in the vessels its boiling point is beyond the capacity of our system. (i.e. BIG TROUBLE)

third possibility is) none of this is happening, I'm just hallucinating from being on graveyard too long.

for final data check data logger printout for 05:40 (marked with a post-it) We shut off heaters at 05:45.

cont'd.

My recommendations for what they're worth.

1) Replace pump. This may be the root of all evil.

2) Pull the weir piece (elbow, tee.) off and inspect for build-up. We are doing a lot of SPECULATING on the condition of this and if its plugged. Lets get a DEFINITIVE observation of the condition. The piece should not be that difficult to remove and if it is filled with crude then we would be able to sample and determine cause. We would have to have plastic or sheet metal under flanged joint to assure nothing dropped between vessel & concrete and to protect porous concrete surface from contamination.

3) Sample vessel #1 contents, either thru a top port or thru inlet when weir is removed. We have a feed sample (Sunday night graveyard) and a product sample (Friday days) to indicate any changes. a vessel sample would indicate what may be building up and/or breaking down in the vessel.

4) Get some priority from management to get the samples analyzed for organic components (e.g. NPH, TBP, MIBK)

5) Hot oil valve (H-9) was smoking heavily tonight. The valve flanges should be tightened when were down.

DAY SHIFT : ARRANGED CRAFT SUPPORT FOR SWING SHIFT : 2 OPS, 2 FITTERS, HPT AND MILLWRIGHT. Mg, Red, Henderson

TESTED FEED PUMP & CHECK VALVE BY PRIMING PUMP : 1½ PSI, 12" Hg suction. OPENED CHECK VALVE BYPASS – NO CHANGE IN READINGS

TESTED PRODUCT PUMP BY TRYING TO TAKE SAMPLE. COMPUTER SHOWS 3 GAL IN VESSEL, AND WE COULD HEAR WEIGHT FACTOR BUBBLING IN TANK. COULD NOT GET MORE THAN FAST DRIPPING FROM SAMPLER, NEED TO CHECK PUMP. MILLWRIGHT TOOK NEW GEAR AND USED GASKET TO SHOP.

WATER FLUSHED & DRAINED FEED LINE (4 GAL). MAY HAVE ADDED 6-7 GAL TO VESSEL BY PRIMING PUMP, FLOW CHECK & LINE FLUSH.

9/25/90 - Tuesday
Swing shift

Replaced the feed pump and check the vessel contents with a stick and a flashlight.

- did not ~~change~~ install the screen in the strainer as it seemed a better test to find out what a new pump would do in the same location as the old one.

- Could not remove and rod out the feed Tee as the liquid appeared to be as high as the Tee.

- The sticking of the vessel indicated it was full of a mud like solid which could just be penetrated with the ~6" stick used to check the vessel. The liquid layer must have been very thin.

11:30 - Oil Set temp increased from 400 to 400, Vessel #1 coming on good.

9/25/90 - Graveyard Shift - Paulina, McDonald, Greenhalgh.

00:00	- Oil Set Temp	411,	Vessel #1	Temp	386,	Product Tank level 12.5.	
00:20	- " " "	412,	"	"	"	387	
00:40	- " " "	415,	"	"	"	387	
00:45	- " " "	417,	"	"	"	387	
00:50	" " "	419,	"	"	"	387	
01:00	" " "	422,	"	"	"	385	
01:15	" " "	424,	"	"	"	376	
01:25	" "	426	"	"	"	375	; Prod Rate < 0.1
01:30	" " "	428,	"	"	"	373	
01:40	" " "	430,	"	"	"	370	
01:45	" " "	432,	"	"	"	365	
02:00	" " "	434,	"	"	"	363	, Product Tank level 16.0
02:35	" " "	440,	"	"	"	,	" " " 17.8

Waste Note:

The 90-Day clock was initiated for the drum when it was completely filled, inner bag Tape sealed, and lid bolted down on Sept. 12, 1990. An SDAR (Storage/Disposal Request) was written earlier and submitted to Solid Wastemanagement. When the drum become full and the clock was initiated, solid waste management was notified so that they could finalized the SDAR. An approved SDAR is required before disposal or storage of the waste drum can be initiated. Willbur

Willbur performed weekly inspection of waste drums.

0240 Oil set Temp. 442, Vessel #1 Temp 368, Product Tank level 18.0
0300 " " " 450, " " " 364, " " " 18.6
0305 " " " , " " " 361, " " " 19.0

03:27 NOW MAXED OUT AT 460° ON HOT OIL SET POINT

04:30 Well here we sit at 460° hot oil set point. There is a ΔT across the heating coils of 3° (both vessels). #1 vessel @ 395° and #2 @ 410°. I expect both to equilibrate at a little over 400°. The vapor out temps are cooling and approaching ambient temp. The vessel & demister pressures are just above zero. In all we have distilled 7.3 gallons into the product tank this shift. The product transfer pump is switched off and will remain off until the tank is near full. This would prevent it from pumping dry in case of an (now unlikely) pressurization

From Dicks observations we are now sitting with the #1 vessel filled with the same slime that was filling our inlet filters. When we changed out the demisters there was about 8" of sludge (Aug 25th, page 85) in the bottom of the vessels and the rest of the vessels ~~were~~ was clean. Since then we have processed 4,890 gallons and have filled up the vessel with slime, pudding, sludge, peanut butter chocolate mouse ... If we change out the vessel now, we still have another 6600 gallons to go. Therefore we may (or may not) have to change out a second time before finishing. My recommendation at this point is to shut off the feed pump, keep the hot oil at max temp for the rest of the week and work at changing out the vessel next week. We should keep the heat on until we are ready to change out the vessel to assure that we are ~~at~~ as close as possible to dry in the vessel and to assure that we have driven off all benzene. If we don't we may not pass the TCLP analysis and have to reconnect the vessel and dry it out again (too much work!) If the feed pump is off then we can run at [illegible]

05:25 After talking to Olaf we decided to go ahead and shut down the flow pump and up the N_2 flow to the vessel, and start baking out the sludge and driving off any residual liquid/hexane.

TOTAL PUMPED TO DATE: 28,743 gallon.

Vessel #2 has shown some high temp spikes up to 429° then will suddenly drop back down to lower (410) value.

NOTE: Vessel pressure & demister dp's are due to increased N_2 flow.

Kyd. GRW

~~Graveyard~~ Day Shift: Olaf, Dick H. & Red

~1405 Oil system running @ 454°/457°F.
Alarm (horn went off twice) —
Cut heat to 455°F off. Turn horn. Blips were of short duration.

Wrote JSA, J-1, J-4 and J-7 for vessel changeout. Current plan is to run till ~11pm tonite, then start cooling for prep work Thursday afternoon, and vessel changeout Friday. Not sure we'll have the crane crew. Will find out Thursday morning.
I'll be in town Thursday AM to get QA signature from Dieffenbacher's manager (Ron is out til Friday morning)
Alarm on Oil System — this may be a coincidence, but that constant churning in vessel #2 has stopped. Could this have been an air trap that finally went thru the system and setting off the low-flow switch alarm/heater interlock for a couple of seconds? Every thing else is normal.
Had fairly high flow on N_2 into vessel #2 a couple of times to sweep fumes out. Pressurized underground tanks took 4" this morning with N_2 then backed off. UG tanks went back to 0.2. Doing another high-flow this pm (3:15). This morning the increased N_2 flow boosted the offgas temp on #1 about 40°.
Plan to PCA water boil out from procedure. Given the semi-liquid condition of the tar, I don't think it's a good idea, in fact might give us some major problems.

DJH
RGC
JMB

Swing 9/26/90 Wed.

Continuing heating to boil off as much Heron as possible.

1745 increase N_2 purge to #1 vessel to strip Heron vapors from demister and vessel. Will use this in place of water boil off.

2230 Placed oil heater setpoint to ambient, turned off oil heater; turned off condenser fans; Reset N_2 purge to #1 vessel

2245 turned off oil pump reset N_2 purge

Day Shift 9/27/90 Dick H. + Red W

0845 - We had to break into the trailer. the key I had stashed is gone. A mouse must have taken it.

0900 - Started Oil pump because Oil temp was 177°F and Vessel No. 1 was 239°F

1042 Stopped oil pump; Control panel power off. Unit must be worked on until tomorrow.

9.28.90 Day shift RJ, RJ, ERW Friday

Filters pulled all the piping connections from the vessel and removed the vessel. There was a large release on the feed tank and in the vessel. The vessel was cleaned out and prepared for ready for bigger disposal. RJ

RJ

10/1/90 Placed rags from clean-up in waste drum
Removed old vessel, moved demister set to new vessel, and installed new vessel. Started installing piping. Not complete.
Connected weir & weir piping.
Looked inside old vessel. Amazingly, .. it looked CLEAN!
Vessel looked filled with black liquid, with ~ 1 ft. of coils

OM

exposed (maybe a little more). Coil looked like they were coated with a thin layer of tar (maybe 1/8" or 1/4" thick). Lower demister housing also was coated with the same layer of tar

RSP

10-1-90 Swing Pump - Henderson

A water truck hooked up to a hose & water is running out of it - enough to cause a real mess of it runs all night - None is around. Is called operators (2-2920) & informed them of the problem.

2 OCT 90 PAUL & OLAF

- As noted above: water ran all night and it did make a bit of a mess (no problem)

- Installed a new thermocouple into vessel #1. It extends down to about a foot and a half (maybe two ft) from the bottom of the vessel.

Completed restoration of system, re-filled oil, and started system up at 2:15 pm.
Laggers did not complete all jacketing. They will install metal jacket tomorrow. Lagging of the short section of oil line leading into #1 caisson may be delayed beyond tomorrow.

PSL

Temp Controller #75 F @ 1540 BH.
1555 Increased temp controller to 200°F on hot oil - OK

1600 Found the #1 Vessel T/C is a type J in a Type K system all vessel temperature for #1 are lower than printed on the log. Trying to locate a type K T/C ~ 6-8 feet long.

May have to wait until day shift to change

1700 There is a lot of water in the hot oil system this time. Remember to drain the coils in a new vessel before installing (may have antifreeze in it.)

PD

Oct 2, 1990 Tuesday - Swing (cont)

I hope we did not have antifreeze in the coils as this may require changing the hot oil and we don't have that much.

2245 The water boil out has been difficult, at one point ~1900 I did consider shutting down as the hot oil pump was cavitating. Finally got the water out by purging N_2 through the surge tank. Used about 1/3 of a tank of liq. N_2 but finally got the pump to steady out and started increasing temp.

The new T/C is here but is an 84" so it will be a little shorter. Insert ~4" into the vessel - if we can not get to it I will make up a correction table tomorrow.

Chw BSP

Oct 2→3, 1990 GRAVEYARD

Hexane is running
Germany is united
and Kuwait is still apart of Iraq
Somethings change some don't.

01:00 Oil set point is 282°, have been bringing it up in 3° increments since the water boiled off. Vessel #1 pressure now at 15" and "indicated" vessel #1 temp is 315° vessel #2 is 241°. last 10 min printout had a production rate of 0.72 gpm will start raising in 1° or 2° increments since we are at hexane b.p.

03:00 Oil set point is 299°, keeping vessel pressure about 15 to 20 inch, production rate has been about 0.92 gpm. We keep chasing up the oil temp. 1° or 2° at a time depending on vessel pressure.

Started to rain, so we opened the drain pan valve to prevent the feed pump from shutting down. It stopped raining so valve is once again closed to give us leak detection/protection. When shutting down the process for the week end, we need to remember to open the valve so that in case it rains the pan will not fill with water.

Note: PNL - (Ron Brodazynski) is scheduled to come out here on Friday, Oct. 5th to assay the 2 ~~[illegible]~~ waste drums. Please look for him and help him set-up his trailer rig.

Thanks, Wilbur

04:00 Started getting alarms for "low flow thru the heaters" at the heater control panel. Pump discharge pressure was varying wildly from 5 to 30 inch, while the inlet pressure remained constant. Pump was sounding like it was pulling air accassionally. We dropped the oil temp from 302 to 290. and opened up pipe cap on reservoir tank to vent any moisture. Pump stabilized (with a discharge pressure varying about 30 in) in a few minutes. We've brought the oil temp back up to over 300 with no problem. Evidently there was still some water remaining in the oil, which hopefully will be eliminated in the separator.

05:05 Steam is visible from the hot oil reservoir vent. (the one we removed cap from). and water droplets have formed around the lip. Another interesting note is that it is trying to snow!

06:05 not much steam visible from hot oil reservoir vent and hot oil pump outlet pressure improved from 30 to 35

06:30 water in the oil caused pump cavitation to the point that we shut down the heater & then the pump. You can hear the oil/water boiling in the expansion tank.

tk	141	385	16,036.	142 - start
	142	5972	- 6,357	remaining
		6357 in tanks.	9,679	pumped from 142
			19,275	" " 141.
			28,954	total pumped.

OLAP call this into Sylvia (3-5521) she didn't answer when I called.

DAYSHIFT MM - Allowed oil system to cool down. Spent rest of day trying to resolve the anti-freeze in coil

10/3/90 Wed. Dayshift

scenario. Here are the known facts:

3 vessels have aluminum tags stating 2 gal antifreeze added 1-89 : #1 vessel now by fence
#2 vessel still installed
one vessel on flat car

2 vessels have no tag : replacement #1 vessel
: one vessel on flat car

Crane crew came out and set un-tagged vessel on flat car upside down — coil was DRY !

This does not guarantee the replacement #1 vessel coil did not contain antifreeze but makes it unlikely.

Antifreeze per American Hydrotherm may cause crud formation in the oil, with coating of the heaters and possible abrasion of the pump seal, as well as extreme difficulty in steaming out the system (potential cavitation and pump damage).

Took samples of oil from: fill pipe (lowest point in system)
suction strainer
reservoir drain
fill pipe after 15 min pump recirculation.

All samples were somewhat milky looking, but no second phase. Saw some crud in reservoir drain (mud leg).

CONCLUSION : 90+ % sure we don't have antifreeze, but do have water in the system. Plan to do EXTENDED steam out for at LEAST 1 shift, perhaps more, then VERY SLOW TEMPERATURE RISE beyond this point.

Started oil back up @ 3:15 pm. With H-3 (bypass) open, H-9 (coil feed) closed and H-5 (steam out) 2 turns open, could not get enough flow thru orifice to keep heater on. NEED TO KEEP H-9 closed and throttle H-5 a little more during steam out.

Shut off feed pump, ~1pm. Closed tank suction valve within 2 seconds of shutting down pump to avoid loss of prime.

RGC RGH.
RBP

Swing shift RGC, RGH, DDP

Started increasing oil temp to boilout water. Had to clock HV-9 to get 8+ PSID on flow meter. No problem up to 250°F but did get steam off of system

Plan slow water boiloff - 2 hr at minimum coil recirculation - 2 hr with increasing flow to coils and H-5 open (was throttled) - 2 hr max recirculation with increasing oil temp to ~ 350°F

GRN

GRAVEYARD PAV, RED, WILBUR OCT 3 → 4, 1990

00:00 closed off steam elimination valve H-5, keeping N_2 flow to xpansion tank
Hot oil set point is now 315° and were getting product out.
Looks and feels like only oil mist coming out of the xpansion tank vent.

01:30 There were no fumes coming out of the xpansion tank vent, so cut off N_2.
Still about 1/4 dewar of N_2, will change over dewars later.

Because of the J-type thermocouple, we are have a high temp alarm (#9) on the distillation vessel. The alarm is in error until we replace the thermocouple.

05:45 Were closing in on the "sweet spot". Hot oil temp is 403°, vessel pressure is 17+ inch, and product rate is 8+ gallon/min. We will be fine tuning from here. (i.e. 1 or 2 degree changes in the hot oil temp) I aim to have/keep the vessel pressure just under 20" to keep a good production rate, but leave some operating room, & in case of any sudden pressurizations.

06:10 Hot oil set point = 406°, vessel press. = 203, production rate = 9.3 gallon/min
JUST RIGHT!

07:30 WE'VE treated 5972 ~~5637~~ 5627 (07:30 reading)

141 389
142 + 5627
6,016 remaining in both

16036
- 6016
10,020 pumped
+ 19,275
29,295 total

10/4/90

AM SHIFT — Left all settings alone until noon. Vessel pressure and production rate dropped off gradually, while vessel #1 temp continued to increase. Decided to go slow thru temperature range of past pressurizations (370-400°F).

Raised oil temp to 407° @ 12:00
408° @ 12:15
409 @ 12:40
410 @ 12:55
411 @ 1:05
413 @ 1:25
414 @ 1:20
416 @ 1:25
418 @ 1:30
420 @ 1:40

Backed off oil temp to 418 @ 2:40
416 @ 3:20
414 @ 3:28

Raised temperature 13° in 1 hr. 40 minutes. Production rate and pressures came up gradually. Got rate from .2-.3 to .6-.7 gpm. Started backing off on temperature to keep vessel pressure ~ 10-12".

Filters disconnected denver.

WE'VE BEEN KEEPING PRODUCT PUMP OFF

DON'T FORGET TO TURN ON MANUALLY FOR TRANSFERS

Swing RGC, RGH, DDP

Slowly increasing temp to get 15-20" Vessel Pressure rate coming up nicely. Had a slight indication of gas generation at above 1530.

1900 Running at 90% - 95% Power. Running very smooth tonight

10/4/90 Swing (Cont)

2200 checked product rate for ~20 min exact rate is 1.01 gpm which is high but running very well. At this rate we need to watch it closely.

BP

GRAVEYARD RED, WILBURN & PAV OCT 4th → 5th, 1990

The process is running well.
The Pirates won game one of the national league championship
The world is a wonderful place.

00.10 The #1 vessel pressure is swinging from 17 to 28 inches in relatively short time (a minute or two). We dropped the oil set pt from 412° to 410° to see if this limits the swings

00:30 Within one 10 minute period vessel press ranged from 10 to 29 inches, dropped oil down 1° to 413°

Day Shift Notes: A second drum of waste was filled, taped, and sealed. The 90-Day clock was started (10/4/90). The waste drums HEX-90-001 and HEX-90-002 were Assayed Today by the PNL portable drum counter unit. Assays are necessary prior to storage/disposal. Also, a Memo supplying additional information on the waste drums were mailed to Harlan Boynton. He has the request for the SDAK and should be able to process it. —Wilbur—

03:20 We are getting some "wier'd cycling. The vessel pressure will build up to 27 or 28 inches then drop, coinciding with the drop we will get a high overflow temp alarm (130°+), when the high overflow alarm clears we start building pressure again. At the time of the overflow alarm vessel pressure will drop to 8 or 9 inches and #1 demister d.p. goes to almost zero. Cycle time is 10 to 20 minutes

04:00 On this cycle the vessel pressure rose to 36+ inch, I dropped oil temp down to 400 and shut off product pump power. Pressure dropped gradually until overflow cycled, and reduced pressure.

(CONTINUES)

06:35 The affect of dropping the hot oil set point from 416 to 400 and slightly reducing the feed pump rate (from 6" to 5") has been great! We have not had a high overflow temp alarm since 04:30 and our production rate has REMAINED UNCHANGED at just under 1 gpm. We have processed 500 gallons on this shift with an hour yet to go. It appears that running at those higher temps did not accomplish anything but make the system more erratic.

in past 24 hrs
5627
- 4291
1,336 gallons (540 gallon on graveyard)

NEED TO SUBTRACT 100-GALLON RISE IN 141 TANK! BUT STILL PRETTY GOOD!!

AM SHIFT - Rate has slowly dropped to 0.68 gpm over last 1.5 hours

9:00	Raised	Oil	1°	to	403°	
10:50	"	"	"	"	404°	rate = 0.7 gpm
(Cycling 9" - 20" vessel pressure)						
11:25	"	"	"	"	405°	rate = 0.74 gpm
11:40	"	"	"	"	406°	rate = 0.74 gpm
11:55	"	"	"	"	407°	rate = 0.75 gpm
12:10	"	"	"	"	408	rate = 0.71 gpm
12:20	"	"	"	"	409	rate = 0.75 gpm

Disconnected faulty thermocouple on product tank to stop continuous printout of alarm messages.

Took 2 samples (archive & lab) and took product sample Hexane 57 to lab

BROKE 142-TANK 4,000-GALLON BARRIER AT NOON!

12:40	"	"	"	"	410°	
12:55					411	rate = 0.76

NO FURTHER CHANGES. CYCLED BETWEEN 8 - 22 ALL AFTERNOON. RATE HAS CLIMBED TO ~ 0.8+ IN LATER PART OF SHIFT.

141-TANK CONTINUES TO RISE. AT 642 GALLONS BY 3:15PM. COULD BE THAT CLEAN #1 VESSEL IS OUTPACING OLD #2 VESSEL

10-5-90
Swing Friday

No problems this shift! The cycling vessel pressure is not sensitive to feed rate, I have no idea why this is happening this way.

We continue to transfer to Tank 141

For this shift

	1537 Start	2230 End	diff	Rate
T-141	669	~~3155~~ 893	+224 gal	+ 0.54 gpM
T-142	3760	3155	-605	-1.46 gpM
Total	4429	4048	-381	+.92 gpM

The net rate is a little higher than the transfer Tank value of 0.8.8 gpM that seems about ave for the shift.

2240 All off at this time - cooling down.

Changed computer program to add type J T/C this is still not real accurate as we use type K lead wire.

DSP

GRAVEYARD OCT 7-8th, 1990. PAU, JOHN, DERRAL. & SPECIAL GUEST ALAN OLANDER.

SYNOPSIS OF SHIFT.

When we came in the one N2 Dewar was caked in ice (Dewar #1, closest to rail cars) it appeared to be leaking at the "liquid" valve. Just changed over to new dewar.

We started up pump using the wired remote valving system and 9 gallons of water. Had some problems with the hot oil system at first; we had a low flow alarm and low d.p. across the orifice due to the cold (42°) oil. once the oil heated up flow alarm cleared and d.p. rose.

We then rebooted computer. For some reason when entering the file name it would not accept a title with any spaces, and responded "too many inputs". that is why the strange file name of "OCTOBER8START".

Once we started up the computer we noticed a vessel pressure of 24.6 and a #1 demister d.p. of 24.2. Since the oil temp was just 100° this looked suspicious. The pressures kept climbing too high of 44 inches (999 on d.p.)

- why was it not cut off by High Pressure interlock?

figuring it may be related to the liquid level we "offed" the #pump. and the pressures stabilized in lower 40 inch range. We blew out the vessel and inlet weir lines by increasing N_2 flow to each (individually). Good flows, so the lines aren't plugged. Assuring adequate N_2 flow to the xmtr. we opened the xmtr crossover valve and the valved out inlet vessel pressure zeroed. Therefore the pressure xmtr is okay. Everything points to high liquid level in the vessel and no flow thru the overflow leg.

A possibility is that the same tars that plugged the 3" pipe of the weir piece may have plugged the overflow seal loop down at the bottom where it narrows to 1 inch. It may be a problem now because of the cold temperature causing further congealing. This may also have been the cause of our cycling last week. Yes

If the vessel was indeed full, we opened the feed line valves to 142 hoping for some drain back (thru the check valve). Also, if the flow-out the overflow is temperature dependent, we also started to heat up the system, once pressure became low enough to keep the heater from tripping out.

06:00 We are now running at a set point of 225, and vessel pressure & d.p. have just gone below 20 inches, no sign of vaporization of anything (i.e. water) yet. Will try to start up pump and see if pressures climb with liquid addition again.

06:30 We restarted the feed pump, it ran awhile before it lost its prime. Although it only ran a minute or two (at outlet press 4") the pressures rose. so we did not restart the pump.

06:50 #1 vessel pressure @ 0.5 inches !
#1 demister d.p. @ 0.4 inches !

YOU FIGURE ?!!

07:12. IN our JOY & EXUBERATION over the sudden reduction of pressures, we tried to restart the feed pump. After the pump was restarted, the vessel pressure and demister d.p. climbed simultaneously and slowly to 16 inches. At this point we killed the pump and pressures stabilized (i.e. stayed the same). I am at a loss as to why BOTH the vessel pressure and demister d.p. are directly related the liquid added. Its as if the demister is plugged and the overflow is 90% plugged. Recommend heating to see what happens.

P.S. This is my last week of graveyard. If the project takes longer Olaf and Dick need to work something out. PAV

10/8/90 -07:30 Day Shift Olaf Rasmussen, Geo McDonald, & Will Greenlee

We are heating up the vessels with feed pump off to hopefully lower the liquid level. The vessel pressure and ΔP Demister for #1 are both about 19. The liquid level for tank #1 as near as we can tell is just about the weir.

13:30 System is distilling, 11.0 gals has been condensed into product tank.

14:45 We activated the feed pump after the Vessel Pressure and ΔP Demister had gotten down to >10 and >2 respectively. The pressures built right back up to 24 & 18. So Turned off valve and feed pump.

CONCLUSION – Drain end of weir or drain line itself is plugged. We are able to get liquid into the vessel, but not into the drain line. Tried opening the diversion valve on drain line (to 141 tank) made no difference, so the pluggage is on the vessel #1 end.

PLAN – Lined up fitters and operators for tomorrow morning to pull the weir, clean it out, and possibly also clean out the drain

REQUIREMENT – MUST HAVE SYSTEM COOL ENOUGH TO WORK ON AND LIQUID LEVEL IN VESSEL BELOW WEIR LEVEL

Even tho this system is not working right, we did make some product (pumped out product tank once & now refilling)

10-8-90 Monday Swing

1600 Filled up vessel and then put on max N_2 purge, increased vessel pressure to ~100 inches H_2O No luck, could not break loose the plug

Started heating up and boiling down to reduce level enought to allow removal of the weir.

Openned the valve to 142 and got flow back through the feed line. The hope was that the

10-9-90 Graveyard Paine & Beglin

0000 Reduced Htr setpoint to 90° - Turned off Htr. continuing oil recirculation.

Oil Temp 436° F
Vessel #1 418°F
VESSEL #2 443°F

DAYSHIFT PAV, OLAF, GEORGE, WILBUR, COWAN

- unplugged product tank thermocouple as we were getting spurious readings and alarms. it will now read -9999

- MORNING Rodded the #1 vessel and determined liquid level to be about 1 foot lower than inlet. Replaced the #1 vessel thermocouple with a 6 ft type K. It is sticking out about 1 foot, so it should extend down close to the bottom of the vessel. If a lot of sludge builds up this would affect the temps. Since the TC went in easily there is probably no sludge there now.

WASTE DRUM - ADDED 1 thermocouple
3 pair Tolrex gloves

Drum #3 90 Filled - Day Clock start 9/10/90

- AFTERNOON: Using the N_2 cylinder we blew the drain line seal loop back into the vessel. It broke thru at 15 psi. Removed the weir piece and and it both the weir piece and the 2" vertical pipe had a considerable amount of black watery pudding. We sealed things up and will attack the clean out in the morn

10-11-90 Day Shift - Olaf R., Geo McDonald, W. Greenhalgh & PAV

We completed the cleaning of the weir. In the process we took a solid sample of the weir and put it in the Archive for future Analysis, etc. The vessel was put back together after cleaning the weir. New flange gaskets were used. Waste Drum # 4 was filled during clean up and was removed to the accumulation area. The ninety-day clock start date is 10/11/90. Inventory includes flange gaskets, copper tubing used for cleaning rods, Solver gloves, plastic bags + sheeting, cloth rags for cleaning, mask cannisters, paper sheeting (Scotch-Kraft liner paper), masking tape, black tar junk.

Swing RGC, RGH, ERW

Heating system back up but getting high ΔT on coils to #1 distillation

1630 Had a problem getting the overflow to work - Plugged the overflow line it worked loose after we cut off the pump at 18" ΔP #1 C (this is liquid well up in the demister housing) P Feed Pump is off oil at 235°F and starting to boil will hold for equilibrium here then on up.

Heated on up and set pump to deliver the rate at which we are processing. The overflow is plugged again - May be necessary to blow out line again.

GRAVEYARD (AGAIN!) PAV, JOHN, DEIRELL

Note - The critical stuff is that we added about a quart (1 or 2 lb) of tars and crud that we scraped and cleaned out of the weir and overflow piping. Also on days, when we were connecting the weir, we scraped any excess tars into the vessel, and looked inside. It appeared that there was 3/8 to 1/2 inch build up on the top coil and about 1/4 in. on the 2nd coil.

It appears our #1 drain line is plugged down stream of the seal loop ~~since~~ if we flow out the overflow the 3/8" stainless vent line on the seal loop gets hot not the 1" line. In case of vessel pressurization

the seal loop will still be effective since it would blow out thru the vent line to tank 141 instead of the drain line to 142.

Philosophy for this shift has been to throttle the feed pump to about 95% recirculation ~~to get the liquid low~~ and to raise the temperature up to maximize vaporization. I am doing this to lower the liquid level to the weir height. We started the shift with the liquid level about 5 inches above the weir. (determined by 15" pressure at weir and 10" vessel pressure). Once we are up to max temp and liquid level is down (vessel pressure = weir pressure), we will increase the feed pump flow in EXTREMELY SMALL amounts until equilibrium is established

On the pressure valving; We have been using only the weir pressure tap (black value open) and not the vessel pressure tap (red value closed). Because, when you open the vessel tap (red value open) it ~~bto~~ forces hot solution (that is above the weir) to flow out the overflow vent and into tank 141. Flow from the distillation vessel to 141 is counter productive so avoid opening the vessel line except when you need to determine the real vessel pressure.

03:05 now at a Hot oil set point of 460

03:10 Powerful winds from out of the west. Some temperatures are varying a little due to wind blowing the thermocouples.

	For Graveyard			Since Restart Thurs (16:00)		
tank 142	2498 - 2253	=	245 gallons pumped	3033 - 2253	=	780 pumped
tank 141	1359 - 1472	=	113 gallon increase	919 - 1472	=	553 increase
			132 gallon net processed			227 gallon net

current production rate is 0.8 to 0.9 gpm. (Just right)

03:45 turned oil set point down to 450°. the heater is running 100% and can only maintain a 449° temp.

04:45 We are just a fine tuned machine right now with a production rate of almost 1 gallon per minute. I would recommend to keep runnin and don't touch a thing!

5:50 Last few pump rates have been over a gallon per minute.

6:30 Once the winds died down we were able to maintain a steady hotoil temp.

10/12/90 – Day Shift Geo McDonald, Wilbur Greenhalgh, etc.

Gallons treated to date.

	141		142		
start	19,325		16,036		
current	1515		2963		
	17,810	+	14,073	=	31,883 gallons to date

	1,515	
Since restart	919	30.33
	596	1963
		1,070
		596
		474 gallons distilled since restart

Product Sample Results Todate:
(WOG)

Tank 141 Product – Total Activity 1550 ± 70 pCi/gm
1 sample only → – Tritium 4970 pCi/ml

Tank 142 Product – Total Activity 1330 ± 340 pCi/gm
– Tritium 4290 ± 1280 pCi/ml

The first Product sample from tank 142 was also analyzed for GEA (gamma energy analysis), Total beta, Total alpha, and Total activity. The Results are

GEA (Cs-137)	4370 pCi/L
Total beta	670 pCi/L
Total Alpha	137 pCi/L
Total Activity	1,410 pCi/gm

12:10 – Turned Feed pump off and closed tank value simultaneously after receiving call from R J Cash to do so. He is concerned about operating with a portion of the safety system impaired (plugged drain line).

13:45 – We sampled ~~the~~ product tank. We requested ~~total~~ alpha, total beta, and total gamma in addition to total activity and Tritium.

After turning off feed pump, pressures (Vessel 1 & DP 1) rose sharply so we back off the oil set Temps. Also, we encountered problems with the product tank pressure and had to manually operate Tranfer pump. (Transfer pump is off!)

10-12-90 Swing Friday

1600 Continuing to boil off what liquid we can to insure the level is below the weir in #1 distillation vessel.

1918 Set point reduced to 40 F.

1925 Oil Pump stopped

10-15-90 Monday Days

Spent the day blowing out all the vent and return line. Blew through all lines that tie in to #1 weir. The pressure required to ~~fl~~ blow through was 10PSI for the return to 141 and 142. The leg down into 142 required ~40 psi to blow out. All lines were blown out at well below the 100 psi design pressure.

RP
JAB DDP

~~Graveyard~~ SWING! ... PAU, JOHN, Deirall

- Started feed pump @ 3:30, used 4 gallons of water for priming
- Swing shift is pretty nice, thanks for switching Dick! Is it Olafs turn for graveyard next?
- In case this log is ever read for historical reasons, it needs to be stated that the system was being operated safely on friday before shutdown. As stated in the log for that day, we had a pathway to vent any off gases (due to a sudden pressurization) thru the overflow vent line. This was tested and verified. The true cause of the shut down was an uninformed management call, by someone who ~~should~~ was not in the position to make that call.
- Feed pump thermocouple was off of pump. Reconnected it. Also plugged in product tank thermocouple since problem appears to be in datalogger.
- 16:55 hot oil set point is 220° and vessel temp is 185° started boiling off water phase when we hit 185°. (vessel press & demister d.p. increased)
- 17:35 we are boiling off water (vessel temp 195°), vessel pressure is up to 25 inch and demister d.p. is just slightly lower at 24.4 inch. We are getting vapor thru the demister because the vapor out temp is up to 175°. The water must be condensing on the demister and blinding it slightly. Will keep the hot oil set point at 220° and wait for everything to equilibrate

there has been no affect on #2 vessel as of yet.

17:50 Vessel & demister pressures both hit 32 inches. I valved out the weir pressure tap and there was no change. therefore the pressure is real and not due to a build up of liquid above the weir. Reduced the hot oil set point to 210°

18:00 Vessel press. 34.5 inch, vessel temp 200, dropped oil set point to 198.

18:15 feed pump temp started up, and outlet pressure fluctuating from +5 to negative, Increased flow, but that only made the fluctuations larger. I shut down pump and closed tank valve. The feed pump could not overcome the vessel pressure of 37.5 inch. (If only we hadn't shut down!)

18:30 Vessel #1 overflowed, overflow line was warm thru the seal loop and gradually cooled to ambient temp about the underground tank fence line. This occured after the pressures started downward

22:38 Well, the vessel pressure & demister d.p. were due to liquid buildup. We opened the feed line check valve bypass loop and the feed pump bypass then drained the vessel back. We used an increased N_2 purge to the vessel to help. Before we drained back to tank 142, we pressurized the vessel with N_2 and no flow out the overflow.

Bottom Line: The overflow is plugged, but this time we don't have the alternate route of the vent line is not available. After shutting down a system that was operating we now have one that is not.

Graveyard – Oct 15, 16 – Dick, George, & Wilbur

The thick goo will not flow through the overflow system at the low outside temp. and has plugged off again but now we know that the lines can be blown out at well under the design pressure and this is not a safety problem. ~~so~~ for the balance of tank 142 I propose to operate with the inlet and distillation flows matched or with the no net overflow. This will require operating the pump bypass ~~to~~ valve as required to hold #1 Vessel ΔP at 3-5".

0:20 – We have started up the system and are making product (0.4 gpm).

02:00 – We lost pump suction so had to shut it down untill vessels cooled back down ~~to~~ below water Boiling Temp.

10/16/90 Graveyard (cont)

Had problems keeping the pump going and had to set at ≈ 3½ psi.

10-16-90 DAY SHIFT

Ran system at a steady 0.7 gpm product rate. NO PROBLEMS, DID NOT TOUCH A THING ALL SHIFT

Graveyard had everything set up in a slight "STARVATION MODE", i.e. system can boil off slightly faster than we are putting feed in. Rad survey suggests that liquid level is ≈ 8" below weir level. Oil Temp 450°. Pump pressure (as left by GY) = 2.5 psi. Did not make any changes.
Did not sample product due to marginal condition of plug on sampler valve (actually, below valve). Did not want to risk a leak in our pump-out piping. Plug is VERY hard to remove.

If we can keep running at this rate, we should pump out 142 tank by Wednesday afternoon. 142 tank level at 3:00 pm = 1,048 gallons.

Red went into 300 Area early to order spare pump head and spare 80-mesh and 60-mesh screen baskets. Alan Olander was here most of the shift

Please READ & REVIEW proposed operating plan. Comments?

SWING SHIFT

16:05 With the vessel liquid level below the weir, vessel pressures dropping and solution boiling point rising, we bumped the pump rate up about a hair and a half. This will provide some heat to help carry over the TBP that should be concentrating in the vessel. This extremely small change took the vessel pressure from 9 to 18 inch and demister d.p. from 2+ to 3+ inch.

Dick, re: your note last night on proving we could blow the system out. This is a good point. It is also a point that we proved when we blew the drain line out with N_2 on THURSDAY.

17:00 RAISED HOT OIL 1°.

18:10 Running smooth. We are checking feed pump at least every half hour (on the hour & the half hour), and whenever

17:45 Still producing at .7 to .8 gpm, looking good --- so far.

23:00 The half body counter is apparently working. We must have got it to work since no other shift noted it in the log.

Late in the shift we were discussing the next steps and decided there would be a definite benefit in going directly to tank 141 when we suck air from 142. If we go to 141 and pump out the 1500 gallons, then we would not have to go thru the cool down phase, and could accomplish it with a simple valving change. This would save time and all the problems we get when we cool down & restart. However, this would mean running without a screen on the inlet, (no worse than we are now). I like this idea since we would be emptying tanks when the process is still running well. (i.e. most gallon per day), and would take the least amount of time.

10/17/90 Wed Graveyard

0000 I increased oil temp to 455 to lower level - Readings may not be accurate as to level in vessel / ΔP #1 / demister. May need to blow these lines but not now.

0100 Found Feed Pump not feeding during frequent pump checks, ~10 min. The output was pulsing from 0-3 psi and pump suction was ~14" Hg. Tried to reset pump. would start to Pump then pump would cut out on overpressure. Could get pump to put out 10-15 psi but when bypass was opened the output became erratic. Seems like air going through pump. Restarted many times but could not reach steady operation of pump. Finally lost it completely.

0200 Heat and feed pump off. Check level measurement on T-142 and found the reading to be solid at 550 gal ± 25 gal. Thus at least 5½" of liquid is still in the bottom of the tank

10/17/90 Wed (cont)

0200 The pump ~~[illegible]~~ seems like it is pulling air. The ECN indicates that the suction leg should be less than 4" off the bottom but one demension is only ref.
Does anyone have 'As Built' info on the 142 dip tubes? Also is it possible that the tank is not level?

If the dip pipes in 142 are actually over 4" different in length ~~to~~ we would have to add ~500 gal of water to 142 which seems excessive.

I suggest we ~~[illegible]~~ go back to 141 and then change ~~[illegible]~~ back to the dip legs on 142 as they were. (draw from the long one)
RGC

0630 Changed to Tank 141 - took a lot of water to get pump going as the line was almost empty (used ~ 8 gal). Pump tripped out due to high vessel pressure so closed valve to 141 and started boiling out the water. When all the water is gone, cool to ~220°F then start pump and heat as fast as possible to keep up with pump. Set pressure on Pump discharge at ~3 psi. This is real touchy as there is still air in the line.

0720 May have a problem with #1 demister as it seems to have a permanent ΔP now.

10-17-90 Day Shift Ann, Olaf, Red, Richard.

0818 - Raised temp controller to 280°

1125 Lowered Controller from 350 → 220° (F)

We boiled down the liquid in the vessel until about 10:45 and then allowed the system to cool down. At about 220°F we (15:45) we started the feed pump and were able to get away with almost no prime water. Because no water was added we rapidly increased the temperature to above the boiling point of hexane so that the vessel would not overfill. We stopped increasing the temperature at 270°F but had a pressurization. The demister ΔP tracked up with the vessel pressure and went off scale. The goal was to get the process going without overfilling. If a higher starting temperature were used (meaning no water prime were used) then there might be some better results. AHB

oil Temp Vessel Temp was below 220

Evening Shift Pau, John, Deirell OCT 17, 90

At the start of the shift the "1 vessel press was 23.8" and demist. d.p. was 20.6. the vessel had overfilled due to input > output. We have been letting the pressures (i.e. liquid level) come down. At 17:10 the pressures were 4.3"/1.6", since the overflow temp never varied from 51°, it means the liquid had to have boiled off. The vessel temps were #1=221°, #2=220°. To boil off at these temps means there was probably water in the feed/vessel. When we have some operating room in the vessel we will try to restart with the feed pump at minimal flow. Restart would be a piece of cake if the overflow was open.

18:00 Killed heat to take vessels down to about 200° to try restart. Since we know the stuff boils at 210-220 we will go just below that initially. Opened valve H-5 to the expansion tank to help cool the oil. This really made a difference.

21:45 We've had an exciting last few hours. We started the pump with no difficulty and no priming, and promptly throttled it back to where the outlet pressure was barely above zero (between first two divisions). While constantly watching the pump we had to chase the temperature up, as both the vessel press and demister d.p. steadily climbed up. Both pressures were approaching 40 inches before we reached a temperature/flow rate combination that turned it around.

(CONTINUED)

22:15 Well, we discovered why it was so easy to control the ~~the~~ feed pump to just above zero output. With the pump recycle all the way off we could only get 1 or 2 inches outlet pressure, inlet suction was 5-10 cm (Hg). Seems like we only pumped 7 gallons of water out of 141, the rest of whats in there may be too thick to pump. We may not even have 1500 gallons in 141 if the sp.G. is above the 0.8 of hexane. On that happy note we shut down the pump, and were boiling down whatever is in vessel #1

For tank 141 we need to rod the tank to determine liquid level and pull a sample to determine what it is we are trying to pump & treat.

Until then we might as well pump out of 142, if the feed line isn't plugged with stuff from 141. We may have to blow the feed line back to the underground tanks.

(21)

10/18/90

0000 Boiling down vessel, oil to 460°F. Will cool off and try again later to set feed.

0120 The ΔP on #1 demister are ~2.2" at 1.7 gal/log as this is a linear function of flow at normal operating rate ΔP will be ~12" H_2O. This is up by ~10" and may be a problem.

0240 We started getting real good distillation rate as we got up in Temp to 460 oil then got an outgassing reaction. Turned off transfer pump. Underground tanks went to >1" H_2O, Heater cut off. Very typical TBP reaction, Seems to happen after all the water + Hexane is gone.

0300 - Heater off -

0600 Opened HV-3 to use oil tank to cool the system. Checked level ~~sys~~ instruments on 141 + 142. Zero OK effect of flow normal ~.1" level

DAY SHIFT

Started up system and ran with feed pump on for 3 hours. Vessel gradually filled with feed. Couldn't get rate up fast enough to keep up with pump feed rate.
Shut down 1 hour to boil out inventory.
Couldn't get pump going. Cooled system down and managed to get pump going with a little water.
Now racing feed pump again... trying to get boiloff to equal feed rate of approximately 0.7 - 0.9 gpm. Oil @ 430°F.
Pumped product ~~pump~~ tank 5 times today. So making some progress.

PSP

THE SHIFT OF SWING STARRING JOHN, DEIRELL & PAU. (18 OCT 90)

18:00 Everything has equilibrated and were running well. Product rate is staying at 0.88 gpm. Hot oil set point is 460. If we keep this up, we'll finish 141 on Friday.

RGC

10/19/90

0030 Level (ΔP #1 demister) continues to climb but slowly. Distillation temp. also going up slowly and rate has fallen off to ~.6 gpm

Material Balance Start to date
Start 35,550 (H_2O incl.) Now 35308 Δ ≃ 242 gal ~.7%

0640 Just finished a very interesting pressure / temp excursion in the system. Pump and heat cut out, got pump restarted and left heat off. When pressure dropped back restarted heat. ΔP #1 went way down as did vessel temp but rate is up! Very strange, But looks real good now. Valved to lower Pressure top on #1.
RGC

Day shift

Cooled to around 300°F and started the system again. It took ≃5 gallons of prime to get the pump going. We brought the temperature up rapidly and had to make adjustments to the feed rate to keep equillibrium. Demister ΔP is higher than we would like and the product rate is rather low. Pump pressure has been between 3.5 - 4.5. Good luck

Sample Results

Sample No	Lab No	Date	Time	Results
Hexone 38	E2315	9/17	11:10	Tritium - 5736 pCi/ml Total Act - 1337 pCi/ml
Hexone 46	PNL-329 Bldg	9/20	PM	Preliminary NPH = 11.4%, TBP = 0.699% Balance Hexone & other
Hexone 57	E-2570	10/9	10 AM	Tritium 3398 pCi/ml Total Activity 1161 pCi/gm

Sample log

Sample No	Lab No	Date	Time	Type of Sample
Hexone 34	Archive ~~Total Activity 2494~~	Aug 23, 1990	09:30	Product
Hexone 35	E 2035	Aug 23, 1990	09:30	Product
	Note: Sample taken after Demister problem. H3 = 6770.78 PCI/ML TOT-ACT = 2490.83			
Hexone 36	Archive	8-24-90	1000	Product
	H3 = 1561.18 PCI/ML TOT-ACT = 574.57 PCI/ML			
Hexone 37	E-2039	8-24-90	1000	Product
Hexone 38	E-2315	9-11-90	1110	Product
	TOT ACT 1,336.7	H-3	5735 pCi/g	
Hexone 39	ARCHIVE	9-11-90	1110	Product
Hexone 40	ARCHIVE	9-14-90		Product
Hexone 41	E-2333	9-14-90		Product
Hexone 42	Archive	9/17/90	13:15	Product
Hexone 43	E-2337	9/17/90	13:15	Product
Hexone 44	Archive	9-18-90		Product
Hexone 45	E 2344	9-18-90		Product
Hexone 46	PNL 329 Bldg.	9-18-90		Product
Hexone 47	ARCHIVE	20 mi 9-19-90	1610	PRODUCT
Hexone 48	didn't use			
" 49	didn't use			
" 50	Archive	9/20/90	1330	Product
" 51	E-2375	9/20/90	1330	Product

Sample Log

Sample #	Lab #	Date	Time	Type of Sample
Hexone 52	Archive	9-21-90	0900	Product
Hexone 53	E2406	9-21-90	0905	Product
Hexone 54	Archive	9-24-90	0045	Feed
Hexone 55	Archive	9-24-90	0045	Feed
Hexone 56	Archive	10-5-90	1025	Product
Hexone 57	E-2570	10-5-90	1025	Product
Hexone 58	Archive	10-12-90	1345	Product
Hexone 59	E-2641	10-12-90	1345	Product

Requested Total Activity, Tritium, α_{Total}, β_{Total}, γ_{Total}

SWING SHIFT Deirell, John & Pav.

15:31 Things were going downhill, so dayshift ran away. As we were coming on shift the vessel pressure dropped from 29.9. to 9 inch., d.p. did likewise. Pump outlet pressure was less than 2 and inlet suction dropped from 13 to less than 5. We lost the pump, so we shut ~~down~~ off the feed pump. #1 vessel temp is 430°.

We tried to restart the feed pump by water priming with no luck. Used 2 gallons of water. Interesting enough the water did not flash, ~~at~~ the vessel pressure built up slow and to decreasing slowly. We will try again. P.S. pressure only built up to a max of ≈22 inch

Also found N_2 system out of tilt. Whenever the N_2 Dewars were changed over on day shift (not logged) did you remember to vent the pressure off the dewar first? We also need to follow the N_2 system for awhile after changing to assure its working right. Perhaps we need to change the surveillance frequency to hourly.

18.00 After 4 failed attempts at restart under hot conditions, we will boil off. cool down & shut down for the weekend.

10-24-90 DAY SHIFT 10-24-90

Flushed drain line into 141 and 142. Pluggage was in seal loop. Re-connected short dipleg to overflow and long dipleg to pump suction. Did not remove weir - it was empty. Vessel down ~1' below weir level. Used 100 gallons of water.
PSP Started up oil system at 2:45 pm

24 OCT 90 SWING SHIFT PAV & JOHN

Heated vessel to 220°(+). This took longer for #1 than #2, meaning more thermal mass in #1. Started pump shortly after 20:00, adding 13.2 gallons in all, expecting water to flash and steam out system. However, vessel pressures did not change, vessel and vapor out temps did not change, and the overflow (which had no liquid in) stayed ice cold. Situation stayed like this for 20 minutes. We were able to run pump at max outlet pressure of 5 and inlet suction of 15 in Hg. Since no change in vessel and cold overflow, it appeared that water had just gone down overflow and not into the vessel, i.e. a plug in the weir. The feed pump pressures started to fluctuate and decline so we shut pump off. About 10 min. after shuting pump off, vessel pressure & vapor out temp all went up and we were boiling water and overflow line was hot all the way down to fence. Not sure why there was a 1/2 hour time delay to evaporate H_2O after it was added. One possible theory is there was a thin crust layer above

LDU

took till 22:00 to stabilize & equilibrate system. It is now just boiling off the water added. We will try to start feed pump.
Tried once more to start feed pump. Can get outlet pressure and inlet suction only when adding water. We added another 5 gallons (18.2 total). When the fitters took apart the 142 feed line (previously and now drain line) it was fairly well coated with sludge on the inside. Our whole feed line is probably coated, and if we were to open the strainer housing, I would bet its mostly filled with tars. Either the pump can't pump any more or we need to check our feed line by opening up the filter housing. We will boil down water for remaining hour of shift.
P.S. I noted every thing I could on data logger printouts, review it your interested.

☆ The new response to RPR: DD-096-90 is on the message board. Would someone on dayshift see that a copy is faxed to Bill Heine ASAP. And that Heine passes it on to Bill Osborne at T-plant so that it can be closed. I have also made an extra copy to be given to Rod Garrett.

OLAP, I Also reviewed your schedule. The one that we were all supposed to discuss yesterday morning. My abbreviated comments are also on the message board. And We still need to sit down and discuss among the three of us.

10/25/90 Graveyard

Restarted feed to distillation. Used ~ 30 gal. in 4 tries. Had system pumped OK for ~1 hr then lost flow. When the pump had liquid feed the suction was 17" Hg Max. @ 4 psi out. We need at least 18" Hg to effectively pull water off the bottom of tank 141 & 142. It seems like the pump just does not have enough suction now. I think it is time to flush and repair the Feed Pump.

0500 Tried to pump from 141 just to see if it would be easier. It is after one reclaim we were able to pump from 141 @ 6 psi discharge and 15" Hg. Also overflow working OK this shift we did get some high overflow temp and started up by blowing out the seal loop and steaming the piping.

DAY SHIFT -
Ran steadily until ~ 10 AM when pressures & D.P. started dropping slightly. Started raising temp in 1-2° increments. Up 6° to 287 by 10:30
Product rate 4.2 - 4.6 gp/10min

10:40	Raised Oil to	289	Started contant surveillance on the feed pump ~10:30 as we're not sure when we will lose suction
10:45	" " "	292	
10:50	" "	294	
10:55		297	
11:00		300	
11:05		303	141 level rose for an hour. Blew out dip tube & level dropped 116 gal.
11:10		307	
11:15		311	
11:20		316	
11:35		320	
12:50		400	(Taken up in increments)
13:10		405	Have held here to let reval catch up (529) Production rate is LOW (~2.6)

Pump pressure dropped gradually during the shift from 5 psi to ~3 psi. About 2pm the discharge pressure dropped to ~1 psi. Let it run for a while to see if pressure would come back, but it didn't. Closed 141 suction valve but it didn't make any difference. Shut down pump and started raising temperature for boiling vessel inventory down. Should get to minimum inventory for line flush. Tank 141 inventory is down to ~ 355 gallons.

TOMORROW → arranged for operators, fitters and millwright to install new (spare) pump head tomorrow morning.

SWING SHIFT 25 OCT PAV, HENDERSON

19:40 We are continuing to just "Boil-down" except at right now we have minimal pressures, our vapor out temps are ambient and we haven't seen any product in a long time. So we will soon start to cool down.

SWING SHIFT - 26 OCT PAV, HENDERSON & OLAF

We tried every conceivable way to start the feed pump, it will run for awhile and then reach a point where the tank contents are unpumpable (i.e. -20 in Hg suction & 0 outlet pressure). With all the trying and priming during day shift and start of swing we added 95 gallons of water to the system, (created 95 gallons of waste). The system acts like the only thing left in the underground tanks is an unpumpable

0022. We may be finished. If not at least this log is.
Recommend we actually stick and sample the underground tanks and find out what is there.

JBC
1/5/90

RECORD COPY
NOT FOR CIRCULATION

OCT 25 1990

RECORDS
DOCUMENT CONTROL

NOTEBOOK NUMBER
WHC-N-436

DATE OF ISSUE
10-25-90

SERIES AND COPY
— 1

TITLE (INDICATE SUBJECT CONTENT)
Hexone #2

AUTHOR (S) - DEPARTMENT AND SECTION
Olaf Rasmussen

PERIOD COVERED (INCLUSIVE - MONTH, DAY, YEAR)
FROM TO

IF CONTINUED FROM ANOTHER NOTEBOOK,
GIVE NOTEBOOK NUMBER

IF CONTINUED IN ANOTHER NOTEBOOK,
GIVE NOTEBOOK NUMBER

ROUTE TO	P. R. NO.	LOCATION	ROUTE DATE	SIGNATURE & DATE
OR Rasmussen	61911	R1-51	OCT 25 1990	

Date Received 12-23-92 cw

INFORMATION RELEASE REQUEST

Reference: WHC-CM-3-4

Complete for all Types of Release

Purpose

- ☐ Speech or Presentation
  - ☐ Full Paper
  - ☐ Summary
  - ☐ Abstract
  - ☐ Visual Aid
  - (Check only one suffix)
- ☐ Speakers Bureau
- ☐ Poster Session
- ☐ Videotape
- ☐ Reference
- ☐ Technical Report
- ☐ Thesis or Dissertation
- ☐ Manual
- ☐ Brochure/Flier
- ☐ Software/Database
- ☒ Controlled Document
- ☐ Other _____

ID Number (include revision, volume, etc.): WHC-N-436

List attachments.

Date Release Required: 12-29-92

Title: Notebook HEXONE#2

Unclassified Category: UC-

Impact Level: 4

New or novel (patentable) subject matter? ☒ No ☐ Yes
If "Yes", has disclosure been submitted by WHC or other company?
☐ No ☐ Yes Disclosure No(s). _____

Information received from others in confidence, such as proprietary data, trade secrets, and/or inventions?
☒ No ☐ Yes (Identify)

Copyrights? ☒ No ☐ Yes
If "Yes", has written permission been granted?
☐ No ☐ Yes (Attach Permission)

Trademarks?
☒ No ☐ Yes (Identify)

Complete for Speech or Presentation

Title of Conference or Meeting: N.A.

Group or Society Sponsoring

Date(s) of Conference or Meeting

City/State

Will proceedings be published? ☐ Yes ☐ No
Will material be handed out? ☐ Yes ☐ No

Title of Journal

CHECKLIST FOR SIGNATORIES

Review Required per WHC-CM-3-4	Yes	No	Reviewer - Signature Indicates Approval: Name (printed)	Signature	Date
Classification/Unclassified Controlled Nuclear Information	☐	☑			
Patent-General Counsel	☑	☐	BD Williamson	BD Williamson	12/21/92
Legal-General Counsel	☒	☐	BD Williamson	BD Williamson	12/21/92
Applied Technology/Export Controlled Information or International Program	☐	☑			
WHC Program/Project	☒	☐	G.T. BERLIN	[signature]	12-17-92
Communications	☐	☑			
RL Program/Project	☒	☐ cw	N/A		
Publications Services	☐	☑			
Other Program/Project	☐	☐			

Information conforms to all applicable requirements. The above information is certified to be correct.

	Yes	No
References Available to Intended Audience	☐	☐ N.A.
Transmit to DOE-HQ/Office of Scientific and Technical Information	☐	☒

Author/Requestor (Printed/Signature): O.R. RASMUSSEN [signature] Date: 12-17-92

Intended Audience: ☐ Internal ☐ Sponsor ☒ External

Responsible Manager (Printed/Signature): G.T. BERLIN [signature] Date: 12/17/92

INFORMATION RELEASE ADMINISTRATION APPROVAL STAMP

Stamp is required before release. Release is contingent upon resolution of mandatory comments.

APPROVED FOR RELEASE — OFFICIAL DOCUMENT — cw — Date 12-23-92

INFORMATION

Date Cancelled

Date Disapproved

BD-7600-062 (08/91)

ID Number

Lead Author	Phone	MSIN	Other Author(s) or Requester
OR RASMUSEN	3-4147	R1-51	

Project or Program	Lead Org Code	Sponsor Agency (DOE, DOT, NRC, USGS, etc.)
HEXONE REMEDIATION	TC420	

Editor	Phone	MSIN	DOE/HQ Program (DP, EH, EM, NE, etc.)
	3-4147	R1-51	

Mandatory Comments (Only mandatory comments are to be documented. All other comments should be made on a copy of the information submitted for review and returned to the author.)	Reviewer Name & Signature	Date	Resolution	Reviewer Name & Signature	Date

Legends/Notices/Markings (required per WHC-CM-3-4 or guidance organization.) (Reviewer Initials)

	Yes	No		Yes	No
Applied Technology	☐	☐	Predecisional Information	☐	☐
Business-Sensitive Information	☐	☐	Programmatic Notice	☐	☐
Computer Software Notice	☐	☐	Proprietary Information	☐	☐
Copyright License Notice	☐	☐	Purpose and Use	☐	☐
Export Controlled Information	☐	☐	Thesis/Dissertation	☐	☐
Legal Disclaimer	☐	~~☒~~ BOW	Trademark Disclaimer	☐	☐
Limited Disclosure	☐	☐	Unclassified Controlled Nuclear Information/Official Use Only	☐	☐
Patent Status	☐	☐			

Additional Information

ID Number _____

INSTRUCTIONS FOR INFORMATION RELEASE REQUEST SIGNATORIES

This list provides the instructions for each reviewer. It is meant to be used as a checklist and requires no signatures. The list is not all-inclusive but covers the key areas needing review. Areas needing further review or other areas needing review should be indicated under comments.

Author/Requestor

- ☐ Project or program manager concurs with the issuance of this report.
- ☐ Reference citations are necessary, correct and appropriate for the intended audience and will be publicly available at the time the document is published.
- ☐ If a journal article, the reference style agrees with guidance from the client or journal publisher.
- ☐ Contributions from authors of other organizations indicated in the comment section of this form.
- ☐ Document is responsive to the client's scope of work.
- ☐ Client approved this for release.
- ☐ Document reports or reflects quality technical work.
- ☐ Scientific methodology used is logical and reasonable.
- ☐ Inferences and conclusions are soundly based.

Responsible Manager

Indicated at the bottom of the comment section of this form that General Counsel's office should review this document with particular attention to:

- ☐ An invention (reported or unreported)
- ☐ New software developed at WHC
- ☐ Sensitive subject matter (policies, issues, concerns)
- ☐ Copyrighted information
- ☐ Trademark information
- ☐ Proprietary information.

If the funding for the program represented by the document is from the Assistant Secretary for Nuclear Energy (NE), and public clearance or limited clearance for foreign exchange (sponsor-limited) is requested, the following information is required:

- ☐ Information generation dates
- ☐ Prior dissemination to DOE contractors
- ☐ Prior dissemination under international agreements
- ☐ Substantive value for international exchange
- ☐ Justification for release of information.

Authorized Derivative Classifier

Document reviewed for (and indicated on the front of the form) the presence of the following:

- ☐ Classified Information
- ☐ Unclassified Controlled Nuclear Information (UCNI)

General Counsel Reviewer

Document reviewed for (and indicated on the front of the form) the presence of the following:

- ☐ Previously reported or as yet unreported patentable subject matter
- ☐ Copyrighted material
- ☐ Trademarks
- ☐ Proprietary information
- ☐ Product comparisons
- ☐ Inventions
- ☐ New software developments
- ☐ Information meets environmental compliance.

Communications Department Reviewer

- ☐ This document contains no unresolved company policy issue.
- ☐ WHC logotype is used correctly.

WHC and RL Program Reviewer

Document reviewed for (and indicated on the front of the form) the presence of the following:

- ☐ Applied Technology Information
- ☐ Limited Disclosure Information (foreign exchange)
- ☐ Distribution list discussed with the client
- ☐ Others who should review this document before release indicated in the comment section of this form.

Publications Services Reviewer

- ☐ Structure and format of the document are appropriate for the subject matter and comply with company standards.
- ☐ Restrictive legends, markings, and notices are affixed as appropriate.
- ☐ Required elements are included, such as disclaimer, cover, distribution list, approvals, etc.

Information Release Administration

- ☐ All reviews are complete and this publication or presentation is approved for release (after resolution of mandatory comments).

Hetone Log Book #2 (See WHC-N-408-1)

1700 11/5/90 Monday

Tried unsuccessfully to start feed to vessel from tank 141. The vacuum would come down to #5 in Hg with 8 psi discharge - hold for a few minutes L1 and then discharge would drop to 1-2 psi. Looks like an air leak in the new piping.

1830 Got HPT over and inspected the Piping inside enclosure. found the Plug loose on the vertical into the tank. Tighten the plug, taped around 1" pipe and inspected balance of system.

Restarted Pump, works fine now running water at max power took a lot of indicated level from Tank 141 to fill the vessel.
690 → 620 / 70 * .8 = 56 gal

2100 Still running water at full power.

11/6/90 Tuesday

0200 Have processed about 200 gallons of water since startup and temp starting to increase rapidly, oil to 320°F and pressure dropping pump still holding 3-4 psi.

05:00 We are running full throttle (460°). Vessel pressures are gradually dropping. Feed pump is running and pumping (-15 inlet, +3 outlet) but starting to make some noise, then quieted down.

05:12. Overflow is gradually warming and 141 levels are barely decreasing. Even tho outlet pressure on the pump is still +3, it doesn't appear like we are moving much feed.

APPROVED FOR
PUBLIC RELEASE
CW 12/23/92

05:58 6 NOV 90

Not processing very much. Tank 141 has gone from 349 to 334 in an hour (15 gallon/Hr., 0.25 gpm). Product for same time was 12.4 gallon (0.21 gpm)

06:30 Feed Pump can only pull 15" vacuum, where it used to pull 22". If we want to pump out the rest of the tank, I think its time to replace the pump with the new factory assembled unit. This one should give us 20 to 25 in vacuum.

07:00 Still maintaining a 15 gallon/Hr pump out rate slow but steady. We could keep going until pump gives out, then replace pump.

07:30 pump out rate has been improving, last ½ hr we've been maintaining a 20 gallon/Hr pump out rate, and increased product. Slow but positive!

DAY SHIFT Ran steadily until ~3:30 pm when pump dropped to 10" vacuum and ~1 psi discharge. At the same time the vessel #2 pressure started dropping. Shut off pump after closing suction valve. Re-started pump later - got ~4 psi discharge, then everything went back as before (10" vac, 1 psi). Stopped pump and left heat.

Process rate was very slow all shift, about 0.2 gpm with heater maxed out at 460°F. Wrote PCA to raise heater limit to 495° and obtained phone approvals.

CRAFTS lined up WEDNESDAY AM to install new stainless steel pump and piping. Operator lined up also.

Pumped product tank 3 times this shift ~90 gallons!

11/6/90 Swing Shift (ROC & DOP)

Based on PCA read temp to ~~8~~90°F but very little system responce - I think we may have a vessel full of non volatil high M.W. organic tar. The vessel may not have been ~~[illegible]~~ full - indication in #2 was very rapid jump in temp as if T/C was not in liquid.

1800 Heat off - Cooling again shows ~~the~~ signs of heat transfer problems.

DOP
150°
11/7/90

11-7-90 Wed S

Days
Installed new pump and screen, new T/C in #1 Vessel, stuck level in #2 Vessel and tank 141. added 35 gal of water to 141

- T/C is now in liquid as #1 Vessel is ~50% solids this is shorter than old one; about 18" down from top.

- Level in #2 vessel was ~4" from bottom with less than 1" solid. No detectable contamination on stick in #2, seems clean.

- Very thick liquid in strainer but ~~that~~ thin liquid in 141 ~ 4¼" level

Swing
Started up the pump and had a leak on the outlet side of the 3 way valve lost ~ ½ oz. of liquid. Shut down and call for HPT.

The pump worked very well with >22" Hg. vacuum but seemed to stop delivering liquid may be plugged.

11/7/90 Wed Swing (cont.)

on inlet side, Screen or suction pipe.

* → Check the screen when the fitters are here to fix the tubing leak.

1700 Boiling down in #1 vessel to make room for feed. As we now have liquid temp measurement & plan to push the hot oil up to the new limit (490) and see if the boiling (or lack of it) is controlled by vapor pressure or heat transfer.

inspector want drums at satatite accumulation
no sign of Deterioration DSP

PCA DD000-032 Received Procedure change authorization #DD000-032 Raising the high oil procedure limit from 460°F to 495°F (Controller limit is 490 to avoid any possibility of hitting 500+ which would be a spec. violation.) RGC

1900 Reached steady state on Vessel with oil set at 490°F (Data logger reads 466).

Liquid in vessel is 463°F with no significant boiling! This is real tough stuff.

The mole fraction Hexone must be less than 8%.

The heat transfer is excellent for only 1/2 the coil in service. (The bottom 1/2 is covered in crud.)

→ Boiled out ~33 gal from #1 vessel so there is at least that much space

RSP

DAY SHIFT 8 NOV 90

- REPAIRED LEAK on feed pump. The swagelock fitting on the valve was loose. There was also some leakage from valve stem, so we tighted down on the packing (1/2 turn). Cleaned and surveyed the area.

- Could not remove/inspect feed strainer because of high winds.

JDSP 1600 11/8/90 Started system up from essentially cold @ 9:30 AM. Running feed pump batchwise, pumping water out of 141 tank.

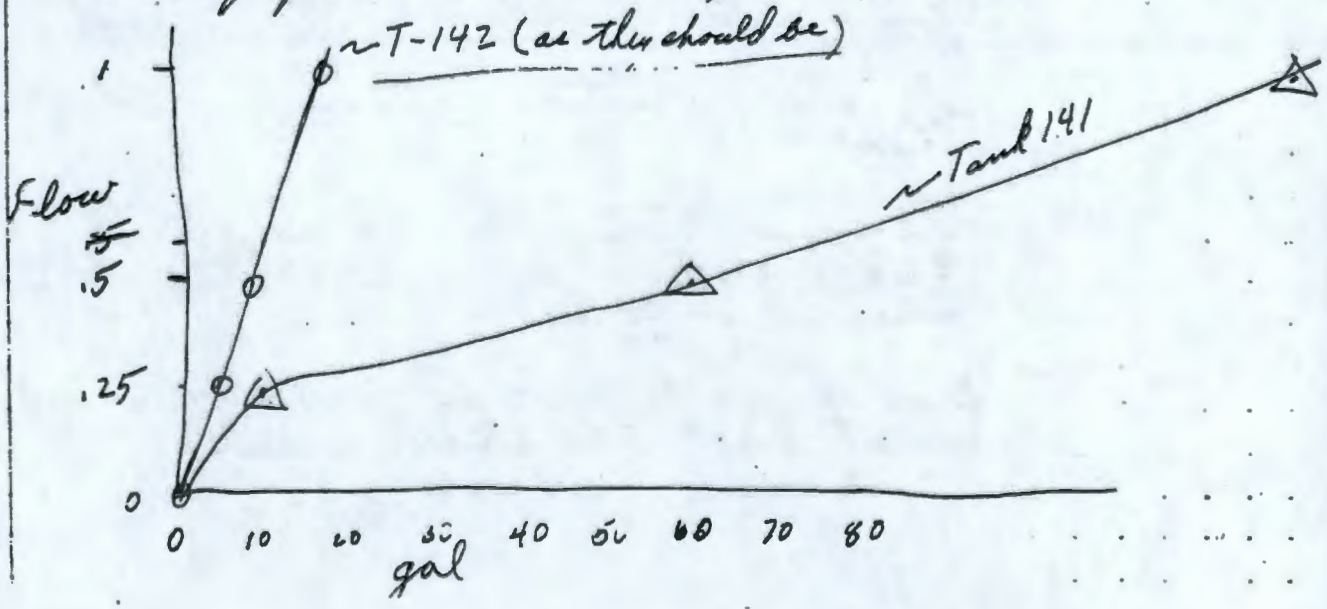
Made 30 gal product transfer to tank car at 3:45 p

Swing

Tried to start and run Tank 141. No good - would pump a little then quit when the pump pulled air. Tried several times always the same. We did get some organic and it looks like we may have taken out more than we put into tank 141, 47 in ~ 110 out.

Note:

Found a problem with the level gage on Tank 141. The tip is plugged and I could not blow it out. Tried twice and even tried to let liquid back up in it, no good, by reducing the gas flow a good reading is possible now but may plug off completely.
Plot of flow vs change in level.



11-8-90

Based on the plot I suggest ~.25 c5h gives a good value of 154 gallons in 141 and 98 gal in 142

2050 Not getting much ~~stu~~ product off now at 350 but getting very poor heat transfer to #1 which is interesting because last night it was very good. The change is at least a factor of 5! Heat off, cooling down

The weight factor level measurement indicate the liquid over the tip of the bubbler is

T-141 = 0.25" { corrected for 0.22
T-142 = 0.75" { Tank pressure.

2080

DAY SHIFT 11-9-90

Started up distillation system and pumped tank 141 three times. Hit air each time. On first transfer #1 level went from 190 → 95. The other two didn't affect the reading significantly.
Took vessel up to 457°F and boiled out 40 gallons to product tank per W.F. Subtracting 8 gallons of water (reading as 10 gal), we recovered 30 gallons of organic from tank 141

Lab ran NPH sample to dryness, evaporating last NPH at 509°F (leaving small amount of charred dry NPH residue).

Heat off at 4:15 pm

Total product now 31 + 8 gal water

AFTERDAY 11/10/90

~~Ab~~ Pumped out 141 one more time level → 136 → 99 added 25 gal H_2O level slowly went up to 133 then pumped to 100

11/11/90

SUNDAY

OPN
Red

Pumped 141 a couple of times after starting up oil system and boiling some of Saturday's pumpings out of distillation vessel #1.
Pumped 141 again later, but the suction piping appears to be plugging up with high suction on the pump.
Boiled liquid at a good rate (0.4 gpm +) until power went out about 3:40 pm.
Turned everything off except power to sump pump, in case power comes back on by itself (which is highly improbable)
Pumped product tank ~~three~~ twice (~ 38 gallons indicated), then re-filled product tank to 25.4 for a total of 71 gal product + 2 gal while pumping = 73 gal indicated
- 52 gal H_2O (indicated) - from 42 gal added this weekend
21 gal organic recovered
Product rate was still 3.4 / 10 minutes when power went off.

MONDAY

Reset 276-J breaker
Installed new dip leg 3½ inches above bottom of 141 with 2½ bell reducer. Added 113 gal water & distillation system is at temperature

MONDAY SWING-YARD 3:30 mon afternoon till 02:30 Tues morn.

23:00 We are still boiling down the second batch from tank 141. Running high #1 vessel pressures (about 30 inch). and high demister d.p. (mid - upper 20's) Very little is getting over to the product tank. (about ½ gallon in 10 minutes.

What were results of NPH boil down test that was to be completed this morning?

23:25 Finally made our first product tank pump out, (filled from 25.5 gallons, to a 40.1 → 14.6 gallon of product in 6 hr. I think its cold enough outside that we ~~are~~ were running a reflux return in our demister filter. OR that when we first started up the demister filter was cool enough that it got soaked thru with condensate and we are having difficulties "drying" it out.

TUESDAY
00:45

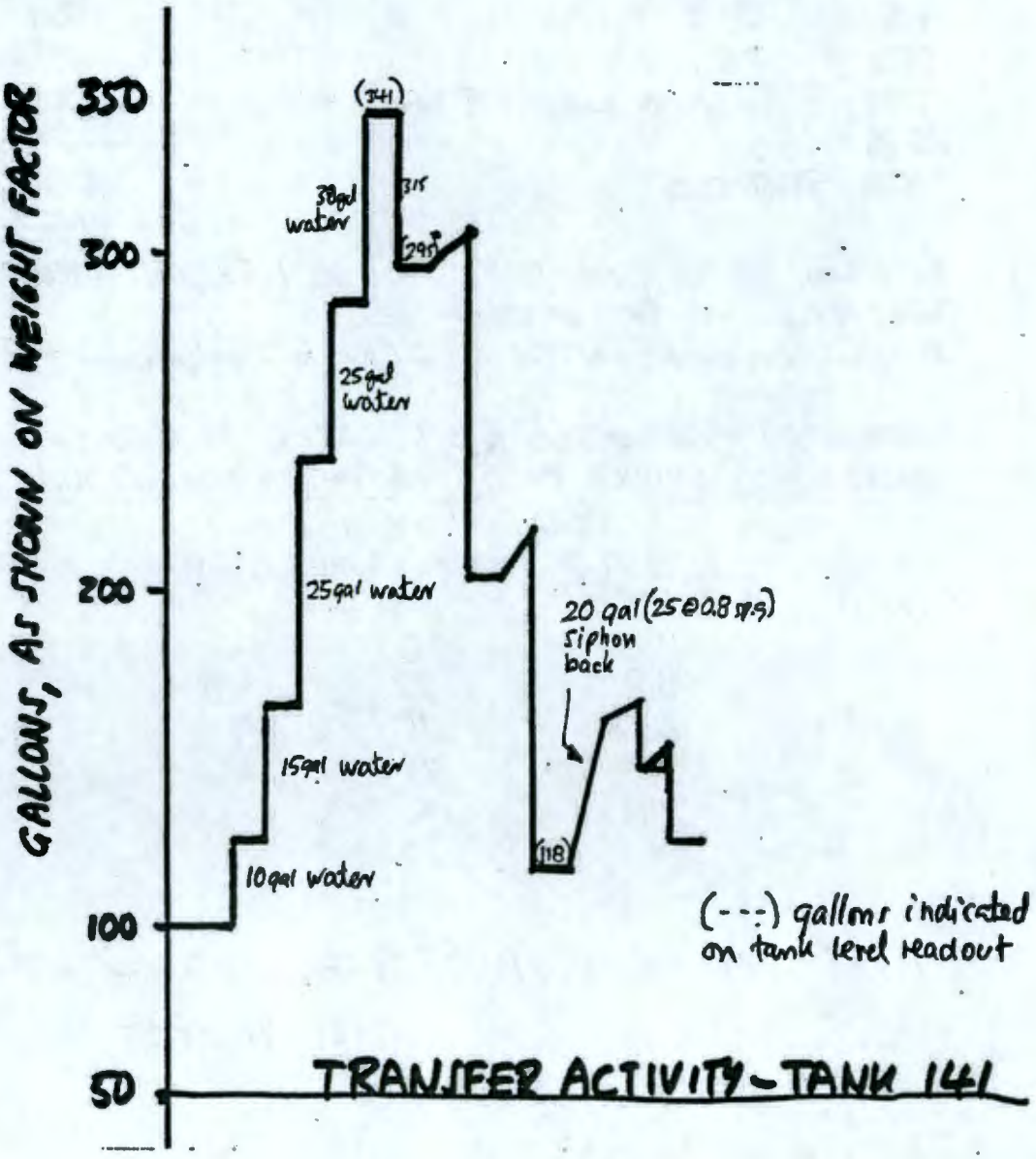
PUMPED OUT BATCH #3. started to suck air, you could hear it at the check valve then we lost inlet suction. Shut time valve and pump immediately. To check our pressures I opened top (red) weir valve, assuming the vessel was not full. Wrong, vessel must have been above weir because we did get some siphoning out overflow.

I would recommend calling Roving HPT (3-2731 or 3-2557) PHONE (or see pg 29 of first log), and then lowering the feed line down further in tank. We could then pump out remaining pumpable liquid. Remember, we want to pump what is possible from tanks.

GRAVEYARD 11-16-90

Pumped 141 tank briefly. Used a lot of water to prime pump - the previous transfer had pretty much sucked the line dry - total 119 gal water. Added to 113, water total since Monday morning is 124 gallons.

Did not pump very long, as we were getting high pump vacuum (~ 20" Hg at full recirculation), only dropped 141 tank by gallons (indicated on weight factor). This suggests screen may be



plugged. Did not want to risk pump under these conditions.
Decided to boil system down to prevent the possibility of siphoning organic out of top of distillation vessel #1
Operated until 7:20 am, when power went out again. At this point we were at ~~#~~ 320°F in vessel #1.

PRODUCT RECOVERY

WATER ADDED MONDAY & TUESDAY : 124 GALLONS, READING 155 GAL @ 0.8 sp.g.

STARTING VOLUME IN PRODUCT TANK : 25.4 GALLONS

TIME	VOLUME		ADDITION WHILE PUMPING	TOTAL	GALLONS TO PRODUCT
23:27	27.9	+	0.5	(28.4 − 13.4 = 15.0)	15.0
2:17	29.0	+	0.8		29.8
4:57	28.5	+	1.6		30.1
6:10	35.4	+	5.0		40.4
7:25	ready to pump full batch @ 41.0				~~(27.0)~~ 30.1
20:19					~~27.6~~
TOTAL RECOVERED					~~[illegible]~~ ~~[illegible]~~ GALLONS @ 0.8 sp.g

ACCORDING TO THIS, WE ARE STILL 10.7 GALLONS SHORT OF RECOVERING ALL THE WATER ADDED.
ADDING INVENTORY IN 141 : 50 GAL ÷ 2.7 wf/gal/gal water = 18.52

RECOVERED + INVENTORY = 162.8 GALLONS @ 0.8 sp.g.
VESSEL #1 SHOULD BE AT A FAIRLY LOW LIQUID LEVEL

CALIBRATION IN 141

	W.F. RDG	WATER ADDED	TOTAL	ΔW.F	ΔW.F./GAL WATER
START	100				
	126	10	10	26	2.6
	168	15	25	42	2.8
	239	25	50	71	2.84
	285	25	75	46	1.84
	341	38	113	56	1.47

AFTER 19 GAL FLUSH : TOTAL WATER = 143 = 178.75 @ 0.8 sp.g

TOTAL RECOVERY = 179.0
+ 0.25

ADDITIONAL LL IN

TANK 141 CALIBRATION

DIP STICK READING	TANK CALIBRATION	W.F. READING MV	Δ MV	WATER ADDITION	MV TO INCHES ΔMV/10.67	LEVEL "
4.25"	205 GALLONS	.4093		~~10 GAL~~		
		.4127	3.4	10 GAL	0.32"	4.57
	264	.4182	5.5	25	0.80"	5.05
		.4267	8.4	50	1.63	5.88
		.4317	2.4	75	2.10	6.35
	415	.4374	28.1	113	2.67	6.88

CONCLUSION: TANK 141 CONTAINS ~~102~~ 98 GALLONS OF SLUDGE ABOVE THE 4.25" LEVEL OR 47% SLUDGE TO 6.88" LEVEL

THE RANGE 4.25" TO 5.05" CONTAINS 58% SLUDGE (34/59)

THE DEPTH BELOW 4.25" ESTIMATED:

0-1"	100% SLUDGE	= 23 GAL =	23
1-2"	90% "	= 0.9 × 42 =	38
2-3	80% "	= 0.8 × 56 =	45
3-4"	70% "	= 0.7 × 65 =	46
4-4.25"	60% SLUDGE	= 0.6 × 19 =	11
			163 gallons sludge

LIQUID BELOW 4.25" = 205 - 163 = 42 gallons of water + solvent

* FOR 2 TANKS: 84 GALLONS

NPH DISTILLATION TEST

Scott Donaldson reported results on his latest NPH distillation test. All NPH is gone by 190°C = 374°F

TV CAMERA - Aaron Schate called, would like to help with TV camera support. J-3142 ← PAV

Electricians re-set breaker in 276-5 about 1:15 pm. Resumed operation and pumped 141. Plan to flush & pump a couple

more time tonight, boil down, then switch to 142 Wednesday morning.

Fitters are making up a new suction pipe attachment to replace the bell:

This is intended for sideways liquid draw to pull in the thin organic layer. Will be installed tomorrow when suction is changed to 142

Talked to Purex about loading out & shipping NPH here. Requested delivery Wednesday, but not confirmed.

[illegible] SHIFT DAVE HENDERSON

Pumped from 141 again, and are boiling down. Not much water pumped, took about an hour. Set the controller at 65% manual control and let it raise slowly. This is to assure we don't reach 100% capacity of the heater and blow the breaker. Sort of an "anti-cowboy" insurance. No sign of any boil-off after the water. ~~Vap~~ Vessel pressures stayed less than 5 in and Vessel #1 temp now up to 410°F

10:25 Hot oil temp (at controller) is 485°, #1 Vessel temp is 442° and no sign of any evaporation (i.e. no vessel pressures and no product) We shut off the heaters.

Pumped out product tank once on shift and accumulated 5 to 6 gallons in it after we were finished boiling water. Whatever is in the #1 vessel it sure doesn't boil easy. When we are down we should stick the vessel to make sure we haven't just filled it with crud.

DAY SHIFT 11-14-90 OTTR + RED

Pumped 141 added 10 gallons & pumped again with dipleg @ 3½" level USING THE NEW SUCTION INTAKE (see above). Pump ran rough at first, then suddenly quieted down — believe we pumped water and then organic.
Boiled off material pumped into vessel #1, then dropped dip leg 1". Pumped out, then dropped dip leg to ½" off the bottom = 1¾" suction level.
Moved suction leg to 142 tank, set at 3½" off bottom

11/14/90 Wed. Swing RGC, DDP

1600 Continuing boil off of the material pumped in on days. The #1 Demister ΔP is higher than should be expected and may indicate some plugging. Power ~ 60%

1900 The Power has been increased slowly to 67% and will not be set higher to avoid blowing out the seal loop or tripping the transformer

2230 Just finishing boiling off water from #2 wheel. Oil up to 295 #2 vapor cooling off and ΔP #2 Demister dropping.

Total product this shift ~44 gal by volume thus at least that much space should be available in #1 for flush of 142.

Note: No boil off of organic was indicated. I suspect we put in water and take out water.

Added 25 gal to #142. Pump and line are primed and ready to pump. Water increased readout by 54 gal. This is less than 1"

DAY SHIFT Started heating up distillation system at 7:15, and pumped 142 shortly thereafter. Added 6 gallons water to 142 after the pumping.
Boiling out all morning.
Got new supply of measuring sticks from Carpenter Shop.
Pumped product tank - 28 gallons, indicated
Pumped 142 after lunch, then lowered dip leg 1", pumped again.
Lowered dipleg to bottom & raised ½ inch. Did not pump as we wanted to boil out the #1 vessel first.
Boilermakers removed all but 1 bolt from old distillation vessel 18" flange in preparation for pumping out that vessel tomorrow morning.
Also plan to flush overhead line on tank cars tomorrow

and disconnect last 3 tank cars for agitation & sampling.

SWING SHIFT DAV & ~~CARRY~~ CARRY DIERALL.

Vessel was filled at start of shift. We are boiling down.

16:55 Gradually ~~losses~~ loosing pressures and temp in #1 vessel rising. We are thru the water phase and will take vessel up to 250°

19:00 TOOK #1 VESSEL TO 252° without indication of any ~~no~~ boiling other than water. Vessel temp never lingered, there was no increase in vessel pressures. We let things cool down and will retry the pump.

19:45 RESTARTED PUMP. PUMPED 142 DOWN TO 41.5 GALLONS (con'level). PUMP INLET VARIED FROM 10 TO 20 INCHES FINALLY IT SETTLED AT -15 AND 142 LEVEL QUIT GOING DOWN (IN FACT IT WENT UP A GALLON OR TWO) SO WE SHUT THE PUMP DOWN. SOME STUFF CAME INTO THE VESSELS ENOUGH TO DEPRESS THE TEMP & PRESSURES BUT NOTHING DRASTIC. WHEN WE SHUT THE PUMP OFF, THE SUCTION PRESSURE HELD AT -15 FOR ~~ABOUT 5~~ A FEW MINUTES AFTER PUMP WAS OFF INICATING THAT THE FEED INLET IS PROBABLY NOW SEALED WITH CRUD. LOOKS AS IF WE ARE AS LOW AS WE CAN GO! WILL RAISE VESSEL TEMPS UP AND BOIL OFF WHATSOEVER WE CAN.

19:55 VESSEL #1 BOILING AT 211°. WHAT WE PUMPED OUT WAS A FEW GALLONS OF WATER. (INDICATED VALUE WENT FROM 64 TO 41 = 23 gallon). THAT WAS AT THE BOTTOM OF THE TANK FROM PREVIOUS WATER ADDITIONS

11:05 Vessel #1 was taken up to 290° BEFORE HAVING TO SHUT DOWN. AS ALWAYS THERE WAS NO POINT WHERE THERE WAS A BOILING POINT BUT THERE IS STILL VAPOR COMING OFF, SMALL BUT POSITIVE VESSEL PRESSURES AND VAPOR OUT TEMPS OVER 100°. I RECOMMEND THAT EVEN THO WE WILL NOT BE PUMPING, WE SHOULD TAKE THE VESSELS UP TO MAX TEMP FOR SEVERAL SHIFTS TO BOIL OFF ANY ORGANIC WE CAN. I KNOW SOME CHOOSE TO IGNORE THIS---BUT WE STILL HAVE TO DISPOSE OF THE DISTILLATION ~~BOTTOMS~~ AND THAT REQUIRES AN EXTRACTION TEST TO PROVE NO RESIDUAL HEXONE IN THE BOTTOMS. LETS SAVE SOME GRIEF LATER AND BOIL THE ------ OUT OF IT NOW.

DAYSHIFT Rodded vessel #1 — Liquid level was ~12" below level of weir, and solids level ~ 2' below weir

Rodded old vessel #1 thru 18" opening — sludge level was ~ 4' 1" below 18" flange and SOLID [redacted] with a center depression

Connected 1/2 [illegible] hose to [illegible]

out old vessel #1. We have about a cup of liquid left, most of which drained out of the pipeline when it was drained. During the pumping operation the 142 valve was opened and also some water flow was on most of the time to prevent excessive vacuum and a pump self-destruct. Used ~ 40 gallons of water. Vessel #1 is at 32.8" pressure

Plan was to drain vessel down to 15" pressure via feed line, then boil vessel down and pump 142 tank again. In preparation we filled the suction line (tank side) with a 4-gallon flush to clear the intake.

(Note: We changed the screen out - it appeared plugged)

Tried to start system but tripped 276-S breaker twice. It turns out the problem is in Hydrotherm heater contactors & wire. Replaced wires (2) and lug, and started up system after Ted & Jim Gore scrounged up parts (MIRACLE).

Removed water line for primer system.

Now heating up distillation system.

17:30 : Inspected waste drums, in good condition no signs of deterioration

17:50 : WE ARE BOILING IN VESSEL #1 (HAVE BEEN SINCE 15:30) HOWEVER NOTHING IS GETTING OVER TO #2. WE HAVE A #1 VAPOR OUT TEMP OF 196°. BUT #2 VAPOR OUT IS AMBIENT WITH NO D.P. AND NO PRODUCT. WE ARE RUNNING AT AS HIGH OF TEMPERATURES THAT WE CAN IN AN EFFORT TO HEAT UP THE SYSTEM.

19:00 FINALLY GETTING VAPORS OUT OF #2 VESSEL. (D.P. .4, Vapor out 180° Vessel 235)

22:15 Pumped 142 from an indicated 168 to 73 gallons (95 gallons) pump was still pumping good at -20 inches when we started getting pressure at the weir. Shut it down then but we still have more that can be pumped. We will heat vessel back up to see if any water is present then pump again. Vessel #1 was cooled down quickly to 145°. Also there was no need to use the pump recirculation valve, there may be air in it that could cause an air lock in the pump.

22:50 YES, WE HAVE WATER IN THE VESSEL. WE WILL BOIL OFF THE WATER FIRST BEFORE PUMPING MORE ORGANIC INTO THE VESSEL. SINCE THE ORGANIC WOULD FLOAT TO TOP IT COULD CREATE A "BUMPING" SITUATION.

00:25 17 NOV 90 WE'RE DONE!
Pumped out 142 from 77 gallon to 46. The check valve was chattering, no pump outlet pressure & only -5 inlet pressure. Ran it for a minute or so to pump out the line. Will now set the heater on manual and run up the temperature to boil off whatever will boil off!

01:00 PUT A NEW DATA DISK IN THE COMPUTER. WE WERE GETTING "OVERLOAD" WARNINGS ON THE DISPLAY

SATURDAY GRAVEYARD OLAF 1 gal. Boiled off all NIGHT. Ran oil @ 70% power until it reached 490°, then switched to auto temp control. Pumped product tank out twice. Boiloff rate dropped to 0.5 gal/10 min until vessel #1 reached into the 440-460 range, at which time the rate went up to 0.9 gal/10 min for some time. Reached 460°F by which time rate was already dropping off. Turned off power about 7:20 am

MONDAY BLEW OUT TANK CAR PRODUCT HEADER INTO EACH OF THE PRODUCT CARS 1 AT A TIME.

2' 9" TO SOLID (IN VESSEL #1)
17" INCHES LIQUID

BSP

TUESDAY RIGGERS CAME OVER & DROPPED OFF SCAFFOLDING FOR DISCONNECTING THE LAST 3 TANK CARS. WE WILL NEED HPT SUPPORT TOMORROW — IF & WHEN RIGGERS RETURN CONTACT CHUCK WARREN

TESTED 141 AND 142 TANKS WITH COMBUSTIBLE GAS ANALYZER USING AN AIR BLEED TO THE INSTRUMENT. FOUND TANKS ARE IN THE EXPLOSIVE RANGE WITH AIR. THIS MEANS WE'LL HAVE TO DO AN NPH FLUSH. TESTED NPH — NO INDICATION AT ALL.

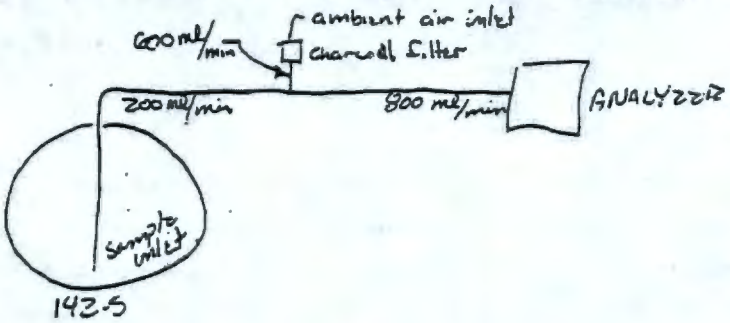
11/21/90

Added 100 gal of NPH & Primed the feed pump to 1412. ~23 gal to #1 vessel and 77 to T142 level 86 → 175 gal. Should be ready to go

PSP

DAYSHIFT 26 NOV 90 PAU & OLAF

- PPE came over and sampled the 142 tank vapor space. Near the bottom of the tank the results were 15% O_2 and 63% of the LEL. The LEL is based on a Hexane calibration and the the sample was diluted 3:1 with ambient air which would require a correction factor of 4X.



When we used this method last week before the NPH addition we went off-scale. So there is a definite reduction in the explosive gas concentration in the tank. However, it is still explosive.

- We disconnected the flex lines ~~between~~ to the last three rail cars. This required scaffolding. Considered removing all piping but we decided we weren't ready.

- Pulled feed inlet filter it was filled with black ooze, a little more watery than usual. Replaced it with a 60-mesh. (removed one was 80-mesh)

DAYSHIFT 27 NOV 90 PAU, OLAF & GEORGE

Started heating up the system @ 08:30 after Cowan walked us thru how to re format an old disc for reuse. We used the previous data disc since there was no operation in that time frame. When formatting it took 2 tries, first try indicated a bad disc (ERROR: "MEDIUM")

At 10:10 The ~~sw~~ data logger locked up after scanning. We backed off of hot oil set point to 200°F to assure no process upset. And are getting some new discs and trying to reach Cowan.

WE RESTARTED HEATING AT NOON.

13:30 ~~01:30~~ As we heated up we did get some overflow from vessel #1 Overflow temp gradually climbed then peaked at 122° @ 13:15

14:30 Vessel #1 is at 394 °F. and no vapor pressure. Both N_2 valves were open. Closed off the red bypass valve.

1700 Added ~20 gal of NPH to #1 vessel and it still will not boil like it should – I doubt the boiling point info from the lab. Wish I had gotten a sample and run the B.P. myself!

1800 Raised suction to ~ ~~2~~ 2" from bottom of 142 and primed the pump using NPH. Opened the vent to top from

2100 Tried to get boiling several times. Filled the vessel and up into the demister section (~18" ΔP on #1 demister) then dump the hot material back to the tank. Did get some boiling as vapor did go above 300°F but this was short ~~lasts~~ time. The vent on the #1 Vessel overflow is plugged and the system will continue to syphon over when you open the valve on the ~~bot~~ pressure sensing line to the top of the vessel. Had to open up N_2 purge to stop the syphon.

→ Need to Disconnect and blow out the vent line when we are down.

Could not run in continous mode due to syphon problem.

The amount of vapor and liquid returned to the tank was enough that the feed from the tank was ~120°F

11-28-90 Wed. Swing RGC + RGH

1920 Heated the material from Tank 142 reaching 460°F Vapor off 346°F, highest Vapor temp on record!

The NPH Boiling point was remeasured by WOG in our lab and found to be much higher then reported earlier by Scott Donaldson. I have no idea how the boiling point of NPH could be missed by this much. ~~WOG~~ Wilber reported

First liquid off condenser at 225°C or 437°F

Boiling temp for bulk of liquid 280°C 536°F

These values are consistant with a mixture of $C_{13} \rightarrow C_{16}$ which is much heavier than expected. We can not boil of just NPH and must add some lower boiling material to process it.

2115 Vapor Temp droped to 200°F, Heat off Distilled ~10 gallons this shift

not at 200°F were not

LOOK LIKE YOU WERE STILL BOILING OFF WHEN YOU SHUT DOWN! WHY NOT RUN TILL THE END OF THE SHIFT?
PAV

12-4-90 Tuesday Dayshift

Heated up, pumped from 141, and blew out the level tube, The Level Seems to be OK. ~90gal now

The plan is to heat this up to ~460°F on swing shift.

12/4/90

cont

The tank vapor question seems to be resolved!

The Hexone is low as expected ~385 ppm per the sample run on the GC but the methane is off scale. This gas does not indicate on the VOC analyzer but ~~still~~ gives high readings on the E.G. Meter when calibration is for Hexane. Thus we seem to be happy with all the measurements now. The 1213 ppm Hexone by VOC must be multiplied by 3-4 to get actual value then 385 per GC is right in there. The Drager tubes seems to have overreported Hexone by factor of 2.

The tank vapor is definately above the LEL and the carbon train can not remove the explosive gas then treat the vent as a potentally flamable gas.

FYI 88P RSP

SWINGSHIFT 4DEC90 PAV & HENDERSON.

- RAISING THE TEMP UP TO MAX. SAW SOME VAPORS OFF AT 450° HOT OIL & 408° VESSEL TEMP.

- RECEIVED A CALL FROM D&D ENGINEERING. THEY HAVE A QUART OF HEXONE IN THEIR CHEMICAL INVENTORY AT 224-B, AND ASKED IF WE COULD INCLUDE IT WITH OUR STUFF TO BE TREATED. IT'S NON-RAD REAGENT GRADE. DAVE ENCKE IS THE PERSON WHO IS GOING TO VERIFY ITS CONDITION AND MAY CALL. I SEE NO PROBLEM WITH THIS WE CAN ADD IT AT THE FEED PUMP, THE WAY WE ADD THE NPH.

- WE NEED TO GET THE N2 DEWAR CHANGED OUT. PROBABLY SHOULD HAVE DONE THAT TODAY. WE MAY NOT HAVE ENOUGH TO RESTART. OUR PROCEDURE STATES THAT WE HAVE AT LEAST A 1/2 DEWAR AVAILABLE TO START. (MY FAULT FOR NOT CALLING IT IN WHEN ON DAYSHIFT YESTERDAY)

- WE ARE GETTING SOME BOIL OFF IN #1 AT VESSEL TEMP OF 450°

1805 PRODUCT TANK PUMPED OUT. WE'VE GOTTEN 16 GALLONS SO FAR THIS SHIFT. VESSEL TEMP IS 458 VAPOR OUT AT 102

#1 Vapor out temps are fluctuating between 100° & 130°. and we are still getting product out.

19:15 #1 Vapor out temp finally dropping down to 79°. VESSEL press & d.p. lower. We were vaporizing for over 2 hrs at a vapor temp of about 100°F

19:30 #1 vapor out dropped like a rock to 56° #2 vapor out is shooting up like a rocket ~~86~~ → ~~316~~ 323 in a minute! then started to die off

20:45 #1 vapor out temp = ambient #2 dropped below 130° started shut down. Assuming tommorrow we will pumpout of 141 again and be up again. After talking to Cowan ~~I~~ We will be back out on swing shift give me a call if this changes 88-509-248-8737 (on FTS). I cannot work Thursday swing tho (Eye doctor exam)

12-5-90 DAYS

13.30 Started repacking of waste drums. Repacked 1 for 1 drum Hex 90-001 & Hex ~~90002~~ 90-002.
Drums 90-003 & 90-004 consolidation started. Both drums currently partial -003 ~ 3/4 full -004 ~ 1/3 full.
Fillins of drums will be completed from 3 drums not yet numbered. [illegible]

DRAINED #1 VESSEL BACK TO TANK 141 (~ 3.5")
AND PUMPED 141 TANK INTO VESSEL WHICH DROPPED 141 TANK LEVEL TO 78 GALLONS (AFTER W.F. DIP TUBE BLOW OUT), HEATED UP SYSTEM — GOT BOILING ABOUT 225°F. HAD TO CUT BACK OIL TEMP TWICE TO KEEP FROM PRESSURIZING VESSEL #1. RAISED SYSTEM TEMP TO 450°F (VESSEL TEMP) BUT WE APPEAR TO HAVE MOST OF BOILING OCCUR IN THE 225 – 250°F RANGE.
6 GALLONS TO PRODUCT TANK (SIGH).
STARTED COOLING AT 12:30 PM.

TOTAL CONDENSATE PRODUCTION SINCE 120 GAL NPH ADDITION: 73 GALLONS

PHOTOS SCHEDULED FOR THURSDAY PM, TANK CAR SAMPLING NEXT WEEK.

Rkd.

SWING SHIFT PAV / HENDERSON

17:30 we tried twice to pump from 141. The pump would pull 15-20 inches for awhile (minute) then pull upto 30" and get noisy. We would turn it off immediatly at that point It is acting like we are plugging the line. We were getting just enough into the vessel to get some pressurization of the system (both vessels & product tank) and the vapor out temps would shoot up We did not overflow either time

18:30 - ATTEMPT #3 at pumping! Yes the line is plugged. The pump inlet goes directly to 30 in. section. This means we were unable to pump anything. Plans for tommorrow should include cleaning of (or change) the inlet filter. I would not plan on back flushing any lines until AFTER WE TAKE PHOTOS.

- We also need to have pipefitters blow out the 141 liquid level tubes.

- The photos are important!!!! Do we have a JSA for this job? has anyone talked to Sue Glover in the last few days before scheduling?

FRIDAY 7 DEC 90 "REMEMBER PEARL HARBOR." PAV / DAYSHIFT

(001 & 002)

SHIPPED THE TWO FULL WASTE DRUMS TO CENTRAL WASTE. SOME QUESTION ON THE PAPERWORK WHEN THEY GOT THERE BUT JOHN BILLINS STRAIGHTENED IT OUT.

TOOK IN TANK PHOTOS IN 142 & 141 (in that order) Removed the new feed line from 141 and placed in 142, but did not connect.

Took Liquid levels in the tanks - - - - - - - - TANK 141 was 4½ inch from bottom of tank, 142 was 2¾ inch. Looks like we may still have to clean the filter and pump from 141 again. BUT we should not do anything ~~without~~ before the pictures are back which should be monday. Note this corresponds to 223 gallon in 141
107 gallon in 142

We used a mirror (to reflect sunlight) to see inside the tanks. Don (the pipefitter) said he saw a mist inside the tanks. This may impact photo clarity.

Oz concentrations varied 3½% in 141 and 4½% in 142

12-14-90 FRI 14 DEC 90.

We pulled the colowasc samples from tank cars 3684 and 3687.
The liquid level in 3684 is 5'-9"
The liquid level in 3687 is 5'-8"
The aqueous level in '87 is approximately 5".
We could not determine an aqueous level in -84

12-18-90 TUESDAY 12-18-90

SAMPLED TANK CARS 3688 and 3685
LIQUID LEVELS : 3688 5'4" consisting of 3'3" organic
3685 5'3" " " 3'11" organic
~~7286~~ 5972 is correct OTK
SAMPLED TANK CAR 3802, SAMPLE #E~~5972~~ OTK RESULT: Total Activity less than 50pCi/ml
SNIFFED VAPOR SPACE FOR COMBUSTIBLE GASES - 0 %

12-20-90 OTK ATTEMPTED TO DRAIN TANK CAR 10K-3802 BUT BY NOW EVERYTHING IS FROZEN SOLID. WAS UNABLE TO OPEN DRAIN VALVE

12-14-90 OTK DRAINED TANK CAR 3802, SUSPECT TANK CAR STILL HAS A SIZABLE AMOUNT OF ICE, SINCE DIP STICK COMES UP DRY. DID A SIZEABLE FLOW OUT THE DRAIN FOR A FEW MINUTES.

1-12-91

0900 SHIPPED 6 DRUMS OF WASTE HEX-90-003 thru 008 to CWC 200W AREA.

SUMP PUMP WILL NOT TRANSFER. SUSPECT PIPING IS STILL FROZEN. WILL TRY AGAIN TOMORROW

0900 6 waste drums RSR 36712
HWM 11 91221 - Drums 3 [illegible] 008

1-15-91
~~12-18-90~~ OTK Tried to pump sump drum with drum pump, but pump quit.

Removed sump pump and found check valve cracked.

Replaced check valve and pumped drum & base. Removed sand from pan outlets, but still were not able to get all pans to drain empty. There is likely more sand in the drain hose that leaves a 1/2"-1" of liquid in some pans. The pump switch in "ON" position

1-22-91 Noticed that EPA WASTE STICKERS HAD BEEN PUT ON DRUMS CONTAINING Removed Demister units. Called Cowan to see if this was correct. He said that the units were not waste but used equipment for which there was still a possible use. Therefore, the WASTE STICKERS should not be on the drums. As these stickers have hard to remove in cold weather I voided and initialed stickers, will check to determine proper method of voiding stickers.

John Bigli

1-24-91 Switched N_2 DEWARS 0822

1-29-91 Called off planned sump piping repairs account no HPT available. Turned sump pump power "off".
OPR Re-scheduled for 1-30-91 tentatively

2-8-91 Straightened out track pans & replaced the worst of the "flammable" tags on the tank cars

2-21-91 Checked out hexone system. Track pans are dry and looking O.K.
OPR Still have sump pump switched off to prevent freeze damage. Dewar #1 is full. Dewar #2 is disconnected but not surveyed. Will re-fill later. Lab reports they have raw data (scintillation scans) on tank car samples, but Oak Ridge is still calibrating data before actual numbers can be assigned
Filed surveillance data sheets

3-5-91 Checked out hexone system. Track pans are mostly dry and looking
OPR O.K. Sump drum is ready to pump, but am continuing to leave power to pump off, as temperatures in 20s are forecasted tonite. One dewar is disconnected & empty. Other dewar is ~ ½ full. Continuing to await tank car sample results. Promised for 3-6-91, but there is room for scepticism.
Filed surveillance data sheets.

3-8-91 Checked hexone system. Everything looks O.K. Still a very
OPR small amount of rain water in a couple of pans. Pump is off. Put empty tag on DEWAR. HPT will survey after lunch & Red

3-12-91 Checked data logger on account of comments on surveillance data sheet. RJC suggested pushing "Run" button to clear "error" message and data logger is working fine again. RJC thinks data logger locked up due to low nitrogen pressure when Dewar ran out. O.K. now.
OTR

Checked pans – all dry, except ~ 1 gallon water in one shaded pan. Pump remains off.

3-25-91 Turned on sump pump and checked piping. Everything looks to be working O.K. Leaving sump pump in "ON" position as the chance of freeze damage should be very low by this time of the year.
OTR

Red had turned on demand system last week, but turned off, as there is adequate N_2 flow via rotometers to keep system inert (but not pressurized significantly).

3-27-91 Inspected system. Looks O.K. for now, but will initiate some cleanup/maintenance work in April.
OTR

4-10-91 Inspected system. RWP is being re-issued to get going on maintenance work as soon as possible later this week.
OTR

4-18-91 New RWP's were issued and are posted on the cork board.
OTR

swing shift Removed suction hose from feed pump &

* Drained liquid (1/2 cup) into small green 5 gal drum and placed in waste accumulation drum
* removed short piece of tubing and blanked suction on pump
* cleaned tar from pan – no radioactivity
* removed paper and installed new spiral kraft paper.
* Sampled rain water in tank car 3802 (3802-1)
* labelled 2 drums "Spare Demin Resin"

4-27-91 8:15p GRR

Received sample of rain water from tank car 10H-3802 (rain water). Results below detection limit. Total Activity less than @ 5 pCi/ml. Drained car.

Inspected site. One dewar is empty. The other is very low, but still shows ~ 100psi. Will arrange disconnecting empty dewar on Monday for pick up.

2-18-92 Vessel #2 Stabilization

- ☐ Disconnect oil lines and instrument lines and weir
- ☐ Remove insulation blanket
- ☐ Remove demister set and set on ground on it's side
- ☐ Check inside of vessel – photo
- ☐ Pump out liquid –or– remove vessel

Flat Car Satellite Drum

2 Funnel
1 Screen w/ 2 tblspoons tar
1 pair gloves
rags
1 pair blue
1 gasket
piping
1 flex hose

Date Received: 1-8-93 NS

INFORMATION RELEASE REQUEST

Reference: WHC-CM-3-4

Complete for all Types of Release

Purpose

- [] Speech or Presentation (Check only one suffix)
  - [] Full Paper
  - [] Summary
  - [] Abstract
  - [] Visual Aid
- [] Speakers Bureau
- [] Poster Session
- [] Videotape
- [] Reference
- [] Technical Report
- [] Thesis or Dissertation
- [] Manual
- [] Brochure/Flier
- [] Software/Database
- [] Controlled Document
- [] Other

ID Number (include revision, volume, etc.): Letter # 9207414R1

List attachments.

Date Release Required: 1-8-93

Title: HEXONE REMEDIATION

Unclassified Category:

Impact Level:

New or novel (patentable) subject matter? [X] No [] Yes
If "Yes", has disclosure been submitted by WHC or other company?
[] No [] Yes Disclosure No(s).

Information received from others in confidence, such as proprietary data, trade secrets, and/or inventions?
[X] No [] Yes (Identify)

Copyrights? [X] No [] Yes
If "Yes", has written permission been granted?
[] No [] Yes (Attach Permission)

Trademarks?
[X] No [] Yes (Identify)

Complete for Speech or Presentation

Title of Conference or Meeting: N.A.

Group or Society Sponsoring:

Date(s) of Conference or Meeting:

City/State:

Will proceedings be published? [] Yes [] No
Will material be handed out? [] Yes [] No

Title of Journal:

CHECKLIST FOR SIGNATORIES

Review Required per WHC-CM-3-4	Yes	No	Reviewer - Signature Indicates Approval: Name (printed)	Signature	Date
Classification/Unclassified Controlled Nuclear Information	[]	[X]			
Patent - General Counsel	[X]	[]	S.W. BERGLIN		1/11/93
Legal - General Counsel	[X]	[]			
Applied Technology/Export Controlled Information or International Program	[]	[X]			
WHC Program/Project	[X]	[]	G.T. BERLIN		1/6/93
Communications	[]	[X]			
RL Program/Project	[]	[X]			
Publication Services	[]	[X]			
Other Program/Project	[]	[X]			

Information conforms to all applicable requirements. The above information is certified to be correct.

	Yes	No
References Available to Intended Audience	[]	[] N.A.
Transmit to DOE-HQ/Office of Scientific and Technical Information	[]	[X]

Author/Requestor (Printed/Signature): O. R. RASMUSSEN
Date: 1-6-93

Intended Audience
[] Internal [] Sponsor [X] External

Responsible Manager (Printed/Signature) Date

INFORMATION RELEASE ADMINISTRATION APPROVAL STAMP

Stamp is required before release. Release is contingent upon resolution of mandatory comments.

APPROVED FOR RELEASE
NS
1-11-92

Date Cancelled

Date Disapproved

ENCLOSURE 2

Hexone Distillation System Rinse Summary

HEXONE DISTILLATION SYSTEM RINSE SUMMARY

RINSE STRATEGY: TANK CARS RINSED WITH GLOBAL SPRAY HEAD AND ALLOWED TO AIR DRY. CONDENSERS, PRODUCT TANK, PRODUCT PUMP AND OVERHEAD LINE FLUSHED TO TRIPLE RINSE STANDARDS.

	TANK CAR 3684	TANK CAR 3687	TANK CAR 3688	TANK CAR 3685	CONDEN-SER #1 (3)	CONDEN-SER #2 (4)	30 GAL PRODUCT TANK & PUMP (5)	1" X 200' OVERHEAD TRANSFER PIPE (6)
SOAP, GAL (1)	10(2)	10(2)	10(2)	25(2)	30	30	30	60
WATER, SOAP FLUSH& RINSE, GAL	195	195	196	130	25	55	65	100
TOTAL GAL	205	205	206	155	55	85	95	160*
ORGANIC VAPOR SAMPLE	LESS THAN 0.1 ppm VAPOR SPACE	LESS THAN 0.1 ppm VAPOR SPACE	LESS THAN 0.1 ppm VAPOR SPACE	LESS THAN 0.1 ppm VAPOR SPACE			LESS THAN 0.1 ppm VAPOR SPACE	
TOTAL ACTIVITY					LESS THAN 50 pCi/ml			
TRUCK SHIPMENT	616 GALLONS (TRAILER LOAD #7)			315 GALLONS (TRAILER LOAD #8)				

TOTAL RINSATE VOLUME: 931 GALLONS

NOTES:

(1) 17% PACE T-10-X, 83% WATER FOR TANK CARS 3684, 3687, 3688. 7% PACE T-10-X FOR OTHER FLUSHES
(2) INJECTED INTO WATER STREAM TO PRODUCE 3% SOAP SPRAY
(3) CONDENSER #1 RINSED VIA OVERHEAD LINE TO TRAILER
(4) CONDENSER #2 RINSED VIA PRODUCT TANK, PUMP, AND OVERHEAD LINE TO TRAILER
(5) PRODUCT TANK RINSED VIA PUMP AND OVERHEAD LINE TO TRAILER
(6) OVERHEAD LINE RINSED WITH 150 GAL CONDENSER/PUMP TANK RINSE
FINAL 10-GAL RINSE THRU OVERHEAD LINE ONLY.
(SINGLE RINSE AT 0.0018 GAL/SQ. INCH REQUIRES 4.3 GALLONS, TRIPLE RINSE 13.2 GALLONS).

ENCLOSURE 3

Manifests for Trailer Loads #7 and #8 to DSSI Incinerator

Please print or type. (Form designed for use on elite (12-pitch) typewriter.)

Form Approved. OMB No. 2050-0039. Expires 9-30-92

UNIFORM HAZARDOUS WASTE MANIFEST

1. Generator's US EPA ID No. WA7890008967 Manifest Document No. A2163

2. Page 1 of 1

Information in the shaded areas is not required by Federal law.

3. Generator's Name and Mailing Address
DEPARTMENT OF ENERGY, RICHLAND OPERATIONS
P.O. BOX 550, 2355 STEVENS DR., HB-11, RICHLAND, WA 99352

4. Generator's Phone (509) 376-4900(w)/967-2335(h) att: MR ROMSOS

A. State Manifest Document Number

B. State Generator's ID

5. Transporter 1 Company Name
KINDRICK TRUCKING CO.

6. US EPA ID Number IND987766078

C. State Transporter's ID

D. Transporter's Phone (435) 882-0457

7. Transporter 2 Company Name
NONE

8. US EPA ID Number

E. State Transporter's ID

F. Transporter's Phone

9. Designated Facility Name and Site Address
DIVERSIFIED SCIENTIFIC SERVICES, INC., ATT: J CRIDER
ROUT 3, GALLAGER ROAD, PO BOX 963
KINGSTON, TENNESSEE 37763

10. US EPA ID Number IND982109142

G. State Facility's ID

H. Facility's Phone (615) 376-0084

11. US DOT Description (Including Proper Shipping Name, Hazard Class and ID Number)	HM	12. Containers No.	Type	13. Total Quantity	14. Unit Wt/Vol	I. Waste No.
a. RQ Waste Radioactive Material, low specific activity, n.o.s., UN2912 (contains methyl isobutyl ketone)		1	TT	1310	G	F002, F003 F005, D001 D018, WC01
b. T(other forms) Liquid, Organic .004 Ci Total U(depleted) .36 grams Total water 25.0% acid esters <1.0%						WP01, WT02
c. hexone 33.0% tributyl phosphate <1.0% kerosene 38.0% misc. solvents 2.0% acetone <1.0%						
d.						

J. Additional Descriptions for Materials Listed Above
- Land disposal notification attached.
- In Emergency contact (509) 373-3800.
- Exclusive Use Shipment
  Drivers Instructions Attached

K. Handling Codes for Wastes Listed Above

15. Special Handling Instructions and Additional Information
RETURN TO GENERATOR IS NOT DELIVERED
TRUCK PLACARDED: "RADIOACTIVE-2912 & FLAMMABLE-1993
CARRIER POSSESSES DOT 1990 P5800.5 ERG HANDBOOK_____(driver initials).

16. GENERATOR'S CERTIFICATION: I hereby declare that the contents of this consignment are fully and accurately described above by proper shipping name and are classified, packed, marked, and labeled, and are in all respects in proper condition for transport by highway according to applicable international and national government regulations.

If I am a large quantity generator, I certify that I have a program in place to reduce the volume and toxicity of waste generated to the degree I have determined to be economically practicable and that I have selected the practicable method of treatment, storage, or disposal currently available to me which minimizes the present and future threat to human health and the environment; OR, if I am a small quantity generator, I have made a good faith effort to minimize my waste generation and select the best waste management method that is available to me and that I can afford. on behalf of DOE-RL

Printed/Typed Name MR ROMSOS, TA ANDERSON JM FRAIN, OR RASMUSSEN, GO ROMESS | Signature | Month Day Year

TRANSPORTER

17. Transporter 1 Acknowledgement of Receipt of Materials
Printed/Typed Name NONE | Signature NA | Month Day Year

18. Transporter 2 Acknowledgement of Receipt of Materials
Printed/Typed Name | Signature | Month Day Year

FACILITY

19. Discrepancy Indication Space

20. Facility Owner or Operator: Certification of receipt of hazardous materials covered by this manifest except as noted in Item 19.
Printed/Typed Name | Signature | Month Day Year

Please print or type. (Form designed for use on elite (12-pitch) typewriter.)

Form Approved. OMB No. 2050-0039. Expires 9-30-

UNIFORM HAZARDOUS WASTE MANIFEST

1. Generator's US EPA ID No. W A 7 8 9 0 0 0 8 9 6 7 — Manifest Document No. A 2 0 8 5

2. Page 1 of 1

Information in the shaded areas is not required by Federal law.

3. Generator's Name and Mailing Address
DEPARTMENT OF ENERGY, RICHLAND OPERATIONS
P.O. BOX 550, 2355 STEVENS DR., MS-11, RICHLAND, WA 99352

4. Generator's Phone (509) 376-4900(w)/967-2335(h) att: MR ROMSOS

A. State Manifest Document Number

B. State Generator's ID

5. Transporter 1 Company Name
KINDRICK TRUCKING CO.

6. US EPA ID Number T N D 9 8 7 7 6 6 0 7 8

C. State Transporter's ID

D. Transporter's Phone (615) 882-0457

7. Transporter 2 Company Name
NONE

8. US EPA ID Number

E. State Transporter's ID

F. Transporter's Phone

9. Designated Facility Name and Site Address
DIVERSIFIED SCIENTIFIC SERVICES, INC., ATT: J CRIDER
ROUTE 3, GALLAGER ROAD, PO BOX 863
KINGSTON, TENNESSEE 37763

10. US EPA ID Number T N D 9 8 2 1 0 9 1 4 2

G. State Facility's ID

H. Facility's Phone (615) 376-0084

11. US DOT Description (Including Proper Shipping Name, Hazard Class and ID Number)	HM	12. Containers No.	Type	13. Total Quantity	14. Unit Wt/Vol	I. Waste No.
a. RQ Waste Radioactive Material, low specific activity, n.o.s.; UN2912 (contains methyl isobutyl ketone)		1	TT	4775	G	F003,D001 WC02,WT02
b. T(other forms) Liquid, Organic .13 Cl Total water 13.90% misc. solvents .05%						
c. hexone 82.10% acid esters .05% kerosene 4.46% tributyl phosphate .05% acetone .28%						
d.						

J. Additional Descriptions for Materials Listed Above
- Lead disposal notification attached.
- In Emergency contact (509) 373-3800
- Exclusive Use Shipment
- Drivers Instructions Attached

K. Handling Codes for Wastes Listed Above

15. Special Handling Instructions and Additional Information
RETURN TO GENERATOR IF NOT DELIVERED
TRUCK PLACARDED: "RADIOACTIVE-2912 & FLAMMABLE-1993"
CARRIER POSSESSES DOT 1990 P5800.5 ERG HANDBOOK (driver's initials).

16. GENERATOR'S CERTIFICATION: I hereby declare that the contents of this consignment are fully and accurately described above by proper shipping name and are classified, packed, marked, and labeled, and are in all respects in proper condition for transport by highway according to applicable international and national government regulations.

If I am a large quantity generator, I certify that I have a program in place to reduce the volume and toxicity of waste generated to the degree I have determined to be economically practicable and that I have selected the practicable method of treatment, storage, or disposal currently available to me which minimizes the present and future threat to human health and the environment; OR, if I am a small quantity generator, I have made a good faith effort to minimize my waste generation and select the best waste management method that is available to me and that I can afford.

GENERATOR

Printed/Typed Name: K.L. CLAWSON OR RASMUSSEN, J EIDE, TA ANDERSON — Signature — Month Day Year 0 3 2 6 9 2

TRANSPORTER

17. Transporter 1 Acknowledgement of Receipt of Materials

Printed/Typed Name — Signature — Month Day Year 0 3 2 6 9 2

18. Transporter 2 Acknowledgement of Receipt of Materials

Printed/Typed Name: NONE — Signature: NA — Month Day Year

FACILITY

19. Discrepancy Indication Space

20. Facility Owner or Operator: Certification of receipt of hazardous materials covered by this manifest except as noted in Item 19.

Printed/Typed Name — Signature — Month Day Year

ENCLOSURE 4

Organic Vapor Monitoring Data for Tank Cars

and Associated Instrument Calibration Log

WHC ENVIRONMENTAL INDUSTRIAL HYGIENE FIELD MONITORING

Page ___ of ___

1. Work Package No.:

2. Contact: OLAF RASMUSSEN

3. Building/Facility: HEXONE RR. CARS + TKS.

4. Area: 2-W

5. Monitoring Date: 10-7-92

6. Room/Location/Site No.

7. Monitoring By: JM Sjostrom

8. PPE (Resp. Prot. & Prot. Clothing)

SWP. WHERE CALLED FOR.

9. Engineering Controls/Work Practices

10. Instrument Type	11. Instrument ID Nos.	12. Calibration Data (Date, Time)
580-B O/M	36944-254	10-7-92 0650
TMX - 2/10	92-9-2	10-7-92 0650

13. Monitoring Type	14. Time	15. Location	16. Activity	17. Results
LEL / O2 / ORGANIC	0830	RR CAR HO-10H 3802	CHECKING FOR ORG.S + LEL	0 / 20.9 / <1PPM
"	0840	HEXONE DISTILLATION SYS. PRODUCT TANK ON RR. HO-10A-3608	"	0 / 20.9 / <1PPM
"	0900	RR CAR HO-10H-3684	RE CHECK FOR LEL + ORGANICS	0 / 20.9 / <1PPM
"	0900	HO-10H-3687	"	0 / 20.9 / <1PPM
"	0900	HO-10H-3688	"	0 / 20.9 / <1PPM
"	0900	HO-10H-3685	"	0 / 20.9 / <1PPM

OPERATOR CALIBRATE
DATA SHEET

TE	EQUIPMENT	ID/SERIAL #	STANDARD	STANDARD CREDENTIALS	PROCEDURE#	COMMENTS/DISCREPANCY			USER
/92	OVM 580-B	36944-254	SPAN #2	0721 of L	[illegible]	AMP 10.6	TIME 0650	CALIB. 102.2	[illegible]
/92	"	"	"	"	"	"	0620	102.6	[illegible]
/92	"	"	"	"	"	"	0700	102.0	[illegible]
/92	"	95 3-7	"	"	"	"	1515	102.3	[illegible]
2/92	"	4-254	"	"	"	"	0610	102.8	[illegible]
/92	"	"	"	"	"	"	0700	102.3	[illegible]
/92	"	"	"	"	"	"	0620	102.1	[illegible]
/92	"	"	"	"	"	"	0615	102.4	[illegible]
/92	"	"	"	"	"	"	0610	102.6	[illegible]
/92	"	"	"	"	"	"	0640	102.4	[illegible]
/92	"	"	"	"	"	"	0600	102.1	[illegible]
/92	"	"	"	"	"	"	0615	102.3	[illegible]
/92	"	"	"	"	"	"	0605	102.5	[illegible]
.92	"	"	"	"	"	"	0603	102.2	[illegible]
/92	"	"	"	"	"	"	0610	102.8	[illegible]
28.92							0555	102.7	[illegible]
9/92	"	"	"	"	"	"	0700	102.3	[illegible]
/92	"	"	"	"	"	"	0615	102.4	[illegible]
/92	"	"	"	"	"	"	0700	102.3	[illegible]
/92	"	"	"	"	"	"	0610	102.1	[illegible]
/92	"	"	"	"	"	"	0615	101.5	[illegible]
/92	"	"	"	"	"	"	0710	102.3	[illegible]
/92	"	"	"	"	"	"	0710	102.1	[illegible]
/92	"	"	"	"	"	"	0600	102.5	[illegible]
/92	"	"	"	"	"	"	0610	102.1	[illegible]
/92	"	"	"	"	"	"	0610	102.3	[illegible]

OPERATOR CALIBRATE
DATA SHEET

DATE	EQUIPMENT	ID/SERIAL #	STANDARD	STANDARD CREDENTIALS	PROCEDURE#	TIME	COMMENTS/DISCREPANCY	USER
9-16-92	TMX 410	92-9-2	50% PENT	BYANE	TMX 410	0620	Pump OK	[illegible]
9-16-92	"	"	"	"	"	2305	"	[illegible]
9-30-92	"	"	"	"	"	0630	"	[illegible]
10/1/92	"	"	"	"	"	0640	"	[illegible]
10/5/92	"	"	"	"	"	0625	"	[illegible]
10/6/92	"	"	"	"	"	0635	"	[illegible]
10/7/92	"	"	"	"	"	0650	"	[illegible]
10/8/92	"	"	"	"	"	0620	"	[illegible]
10/9/92	"	"	"	"	"	0700	"	[illegible]
10/12/92	"	92-9-10	"	"	"	1515	"	[illegible]
10/12/92	"	92-9-2	"	"	"	0610	"	[illegible]
10/13/92	"	"	"	"	"	0700	"	[illegible]
10/14/92	"	"	"	"	"	0620	"	[illegible]
10/15/92	"	"	"	"	"	0615	"	[illegible]
10/19/92	"	"	"	"	"	0610	"	[illegible]
10/20/92	"	"	"	"	"	0640	"	[illegible]
10/21/92	"	"	"	"	"	0600	"	[illegible]
10/22/92	"	"	"	"	"	0615	"	[illegible]
10/23/92	"	"	"	"	"	0605	"	[illegible]
10/26/92	"	"	"	"	"	0605	"	[illegible]
10/27/92	"	"	"	"	"	0610	"	[illegible]
10/28/92	"	"	"	"	"	0550	"	[illegible]
10/29/92	"	"	"	"	"	0700	"	[illegible]
11/2/92	"	"	"	"	"	0615	"	[illegible]
11/3/92	"	"	"	"	"	0700	"	[illegible]
11/4/92	"	"	"	"	"	0610	"	[illegible]
11/[illegible]								

ENCLOSURE 5

Total (Radio-) Activity Analysis for Rinsate from Condenser #1

SAMPLE STATUS REPORT FOR [illegible]
DISPATCHED: 6/ 8/92 15: 5 SAMPLE HAS NOT BEEN SLURPED
RECEIVED: 6/ 9/92 7:56

EXT.	DETER.	RESULTS OR STATUS	OUT OF RANGE?	GOOD ANS?	CHARGE CODE
****	********	**********	***	***	******
4271	TOT-ACT	< 5.00000E 01 PICI/G	N	Y	YLBB1

END OF REPORT

ENCLOSURE 6

Job Safety Analysis for Loading Tank Trailer from Tank Cars

JOB SAFETY ANALYSIS	Job Description: Top-Unloading of Hexone Tank Cars to Tank Trailer	JSA Number: HEX-T-1
	Component: Hexone Tank Cars	Building/Area: 276-S
Reviewed By: INDUSTRIAL SAFETY AND FIRE PROTECTION	Prepared By: O. R. Rasmussen OR Rasmussen	Date: 10-22-91
Date: 11-7-91 Initials: LLH	Review Dates	

SAFETY EQUIPMENT REQUIRED	TOOLS AND EQUIPMENT REQUIRED	JOB PREPARATION
-Full Face Masks with GMH-C Cartridge -SWP Clothing as required by HPT -Gloves, insulated -Gloves, Chemical -Oxygen Meter -Chemical Suits (CNES)	-Spark Proof Wrenches -Ground cables with alligator clips	-Set up nitrogen hose from Dewar to manifold rack. -Install quick disconnect fittings on top fill and vent lines of tank cars, or use unions. ORR -Pre-job Safety Mtg. Review MSDS'S, RWP, ISA, CNES Work Instruction

HAZARDOUS MATERIALS	MSDS	RELATED REQUIREMENTS
Hexone	1343	Radiation Work Procedure: Yes [X] No [ ]
Kerosene	1284	Nuclear Safety Specification: Yes [ ] No [X]
Tri Butyl Phosphate	1739	
Nitrogen (Inert Gas)		

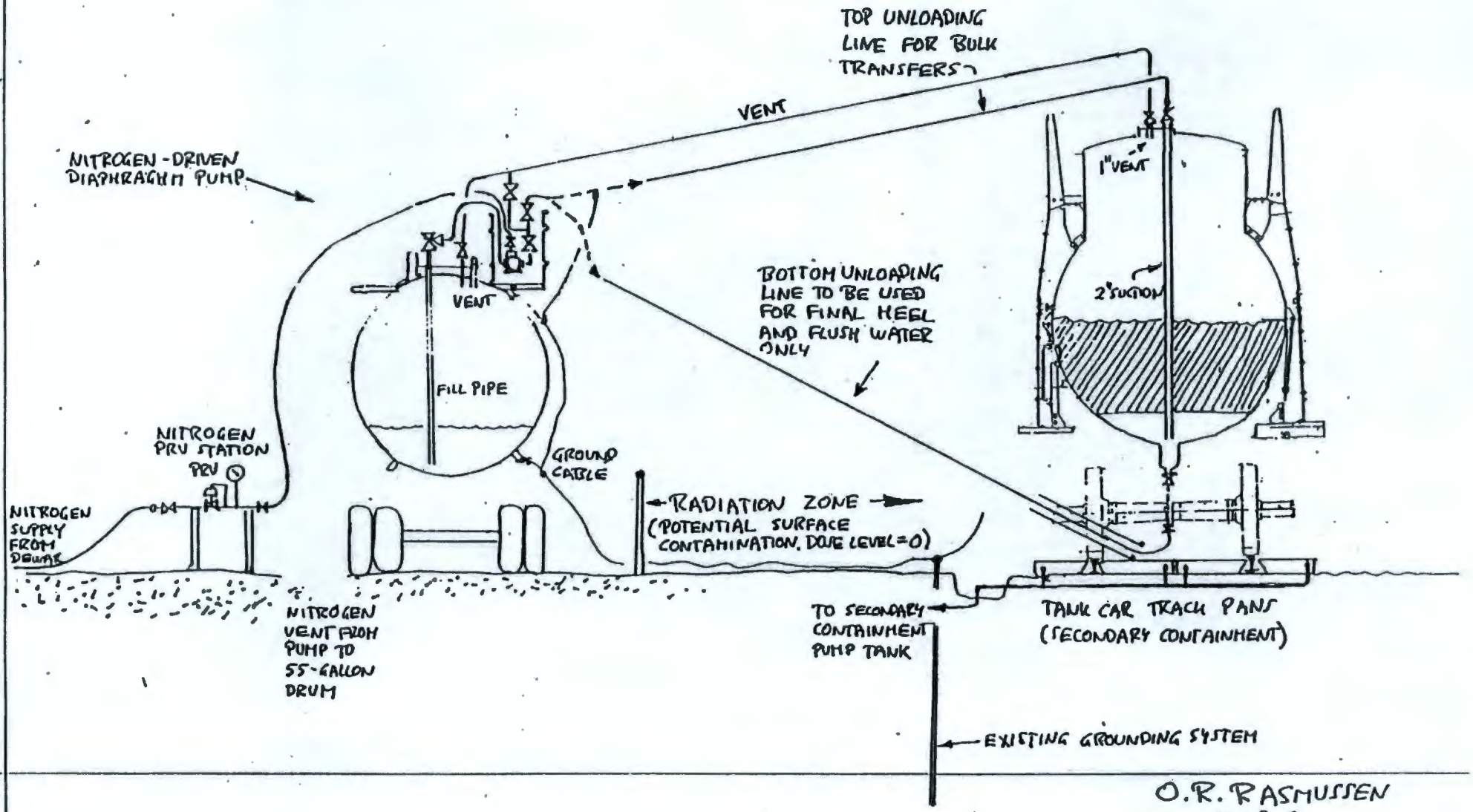
JOB STEP	HAZARD	HAZARD CONTROL AND PROTECTIVE EQUIPMENT
1. Preliminary		
A. Weigh truck and trailer on truck scales at 1100 Area		
B. Survey truck and trailer for radioactivity	Falling From Trailer (This applies to all subsequent work on tank trailer.)	Use handrails and ladder where provided. Take remote swabs where falling hazards are a concern
C. Fill trailer tank with nitrogen gas to inert vapor space		
* Connect hose from 1-inch nitrogen line at 337 Bldg (HTSF) N_2 supply to tank truck fill line (dip leg)		
* Vent trailer tank to atmosphere		
* Verify that 30-50 psi of nitrogen pressure is available		
* Purge approximately 1,000 cubic feet of	Breathing inert gas and residual fumes (if any)	Keep off trailer tank while purging is in progress.

JOB STEP	HAZARD	HAZARD CONTROL AND PROTECTIVE EQUIPMENT
nitrogen thru trailer tank.	-Freeze injuries	Wear gloves (leather, fabric or insulated) when operating nitrogen valves
* Verify oxygen level in trailer tank is less than 10% using oxygen meter.		
* Disconnect nitrogen supply and close valves on tank trailer.		
2. Pumping Preparations		
-Install quick disconnect fittings to vent and unloading lines on tank cars.		Use spark proof wrenches (Applies to all following steps where tools are used on trailer, tank cars and transfer fittings.)
-Set up nitrogen supply manifold. Set pressure regulator to 40 psi.		
-Set up pump and hose connections on truck (by CNES)	-Sparks, fire	Use spark proof tools for making hose and piping connections.
(See transfer diagram)		Shut down engine on truck/tractor at all times except when required to move trailer.
		NO SMOKING AT ANY TIME WITHIN 100 FT OF TRUCK OR TANK CARS
		Connect tractor, tank trailer, pump, vent drum, valve manifold, and aluminum support pipe to ground cable grid with additional ground cables.
-Make hose connection to tank car with quick disconnect fittings	-Breathing fumes -Contact with hexone	Wear full face mask with GMH-C Cartridge. Truck/Pump area - Chemical Suit (Applie to all follow steps. Tank Car - Chemical Gloves
3. Pump tank car to tank truck (top unload)	Sparks, fire	Use spark proof wrenches to open and close plug valves on tank car.
-Open tank car Vent valve	Minor chemical fumes	Wear full face mask with GMH-C cartridge (Applies to all following steps where potential for fumes exists)
-Open tank car Unloading valve		
-CNES will control transfer	Truck overflow due to pumping or siphoning	. Monitor transfer volume with flow totalizer (CNES)
-Close unloading valve on request by CNES or on any indication of a leak.		. CNES verify trailer volume with dip stick . Vent pump out line whenever transfer is stopped to break siphon (CNES).

JOB STEP	HAZARD	HAZARD CONTROL AND PROTECTIVE EQUIPMENT
4. Disconnect transfer lines as follows:		
A. Verify that "CNES" has shut down pump vented unloading line, and allowed enough time for unloading line to drain to the trailer and tank-car.		
B. Close 1" and 2" valves on top of tank car.		
C. Disconnect fittings and seal fittings with blank connectors.	Fumes	Wear full face mask with GMH-C cartridge
	Liquid drips	Wear chemical gloves and catch any drips in a metal can
5. Repeat steps 2 and 3 as required to draw liquid from other tank car(s).		

PROPOSED

TANK CAR TO TANK TRUCK TRANSFER ARRANGEMENT



O.R. RASMUSSEN
10-8-91

ENCLOSURE 7

Job Safety Analysis for Rinsing Equipment Into Tank Trailer

JOB SAFETY ANALYSIS	RINSE TANK CARS WITH SOAPY WATER	HEX-T-3
	Component: HEXONE TANK CARS	Building/Area: 276-S
Reviewed By: INDUSTRIAL SAFETY AND FIRE PROTECTION	Prepared By: O. R. RASMUSSEN	Date: 3-24-92
Date: 3/24/92 Initials: [illegible]	Review Dates: 6-9-92	

SAFETY EQUIPMENT REQUIRED	TOOLS AND EQUIPMENT REQUIRED	JOB PREPARATION
- FULL FACE MASK WITH GMH-C CARTRIDGE - SWP CLOTHING AS REQUIRED BY HPT - GLOVES, CHEMICAL - OXYGEN METER	- SPARK PROOF WRENCHES - GROUND CABLES WITH ALLIGATOR CLIPS	1. CHECK TRAILER O_2 - 10% MAX 2. SET UP RINSE HOSE FOR SOAPY WATER ADDITION 3. PRE-JOB SAFETY MTG. REVIEW MSDS'S, RWP, JSA

HAZARDOUS MATERIALS	MSDS	RELATED REQUIREMENTS				
HEXONE	1343	Radiation Work Procedure	Yes	☒	No	☐
KEROSENE	1284					
TBP	1739	Nuclear Safety Specification	Yes	☐	No	☐
NITROGEN - INERT GAS	1390					

JOB STEP	HAZARD	HAZARD CONTROL AND PROTECTIVE EQUIPMENT
1. CONNECT TRAILER & PUMP SYSTEM TO TANK CAR 87 PER JSA HEX-T-1	SEE JSA HEX-T-1	SEE JSA HEX-T-1
2. PUMP OUT TANK CAR 87 TO TRUCK (786 GALLONS) FOR THIS CAR		
3. MOVE RINSE WAND TO TOP OF TANK CAR	FALLING. THIS APPLIES TO ALL WORK ON TANK CARS	USE CAUTION WHEN WORKING ON TANK CAR PLATFORM. USE 3 PEOPLE, IF REQUIRED TO WORK SAFELY WITH RINSE WAND
4. OPEN TANK CAR LID. CHECK TEMP. AND O_2 CONTENT. VERIFY TEMPERATURE IS 60°F OR LESS, AND OXYGEN LEVEL IS 10% OR LESS. IF OXYGEN LEVEL IS ABOVE 10%, REDUCE WITH N_2 PURGE TO 10%	HEXONE FUMES. THIS APPLIES TO ALL WORK ON OR AT TANK CARS WHEN TANK CARS ARE OPEN	WEAR MASK WITH GMH-C CARTRIDGE ON TANK CAR PLATFORM IF PURGE IS REQUIRED, LEAVE TANK CAR PLATFORM WHILE PURGE IS IN PROGRESS.

JOB STEP	HAZARD	HAZARD CONTROL AND PROTECTIVE EQUIPMENT
5. INSERT FLUSH WAND. COVER TANK CAR OPENING WITH PLYWOOD COVER TO MINIMIZE AIR INTRUSION. CONNECT HOSES.	FIRE	FOLLOW PREVENTION LIMITS: - TANK CAR TEMP ~~OR~~ BELOW 60°F - OXYGEN CONTENT BELOW 10% O_2
6. FLUSH 60 GAL(MIN.) INTO CAR (55 GAL FROM HYDRANT, 5 GAL FROM SOAP SOLUTION (17% PACE-T-10X) DRUM. PUMP OUT FLUSH SOLUTION INTO TANK TRUCK. MOVE WAND INTO OTHER END OF TANK CAR AND REPEAT FLUSH/PUMPING	FORCING SOAP SOLUTION INTO WATER LINE	1. OPEN WATER AT TOTALIZER TO PRESS. HOS. 2. OPEN WATER VALVE ON TK CAR 3. START SOAP FLOW 4. STOP SOAP FLOW 5. CLOSE WATER VALVE ON TK CAR 6. CLOSE WATER VALVE AT TOTALIZER
7. REMOVE COVER, PULL OUT FLUSH WAND, AND SECURE TANK CAR LID.		
8. REPEAT SEQUENCE FOR OTHER 3 CARS EXCEPT 60°F TEMPERATURE LIMIT DOES NOT APPLY TO TANK CARS 88 AND 85 WHICH CONTAIN HIGH FLASH POINT LIQUID. FOR THESE CARS TEMPERATURE LIMIT IS 90°F.		

JOB STEP	HAZARD	HAZARD CONTROL AND PROTECTIVE EQUIPMENT
9. CHECK BOTTOM DRAIN OF EMPTY TANK CARS BY	FUMES	WEAR GMH-C MASK
CAREFULLY REMOVING DRAIN PIPE CAP WITH 5-GAL BUCKET UNDER DRAIN. CATCH ANY LIQUID IN BUCKET. OPEN DRAIN VALVE AND CATCH ANY LIQUID (LIQUID, IF ANY SHOULD BE WATER, BUT ASSUME ORGANIC COULD DRAIN). DISPOSE OF ANY LIQUID IN TANK TRUCK.	CONTACT WITH SOLVENTS	WEAR CHEMICAL GLOVES USE CAUTION WHEN MOVING 5-GAL BUCKET, SEAL BUCKET WHEN MOVING TO TANK TRAILER.
10. SET UP TO RINSE DISTILLATION SYSTEM CONDENSERS AND OVERHEAD TRANSFER PIPING (PRODUCT). SEE FIG. 2.	(NOTE: THIS STEP ADDED 6-9-92 BY O.R. RASMUSSEN AND APPROVED BY TELECON BY S.S. GLOVER	
A. PUMP OUT PREVIOUSLY COLLECTED RINSATE (IN DRUMS & OTHER CONTAINERS) INTO TANK TRUCK		
B. FLUSH #1 CONDENSER INTO DRUM USING 7% PACE-T-10X (30 GAL) FOLLOWED BY WATER (25 GAL). FLUSH #2 COND., PROD. TANK & PROD. PUMP WITH 30 GAL. SOAP SOLN.		WEAR GLOVES TO AVOID EXCESSIVE CONTACT WITH STRONG DETERGENT

JOB STEP	HAZARD	HAZARD CONTROL AND PROTECTIVE EQUIPMENT
C. DRAIN PIPING AS WELL AS POSSIBLE INTO TRAILER		
D. FOLLOW SOAP FLUSHES WITH A WATER FLUSH. THE COMBINED SOAP AND WATER FLUSH THRU THE TRANSFER PIPING SHOULD BE A MINIMUM OF 50 GALLONS. USE AT LEAST 30 GALLONS OF 7% PACE-T-10X, AND 20 GAL. WATER.		
E. PIPING MAY BE DRAINED INTO A DRUM. IF IT IS NOT POSSIBLE TO ACHIEVE COMPLETE DRAINAGE INTO THE TRAILER, DRAIN DRUM INTO TRAILER.	SPILLING RINSATE	SEAL DRUM WHEN HOISTING TO TOP OF TRAILER.

* EMPTY PACE-T-10X CONTAINER MAY BE USED

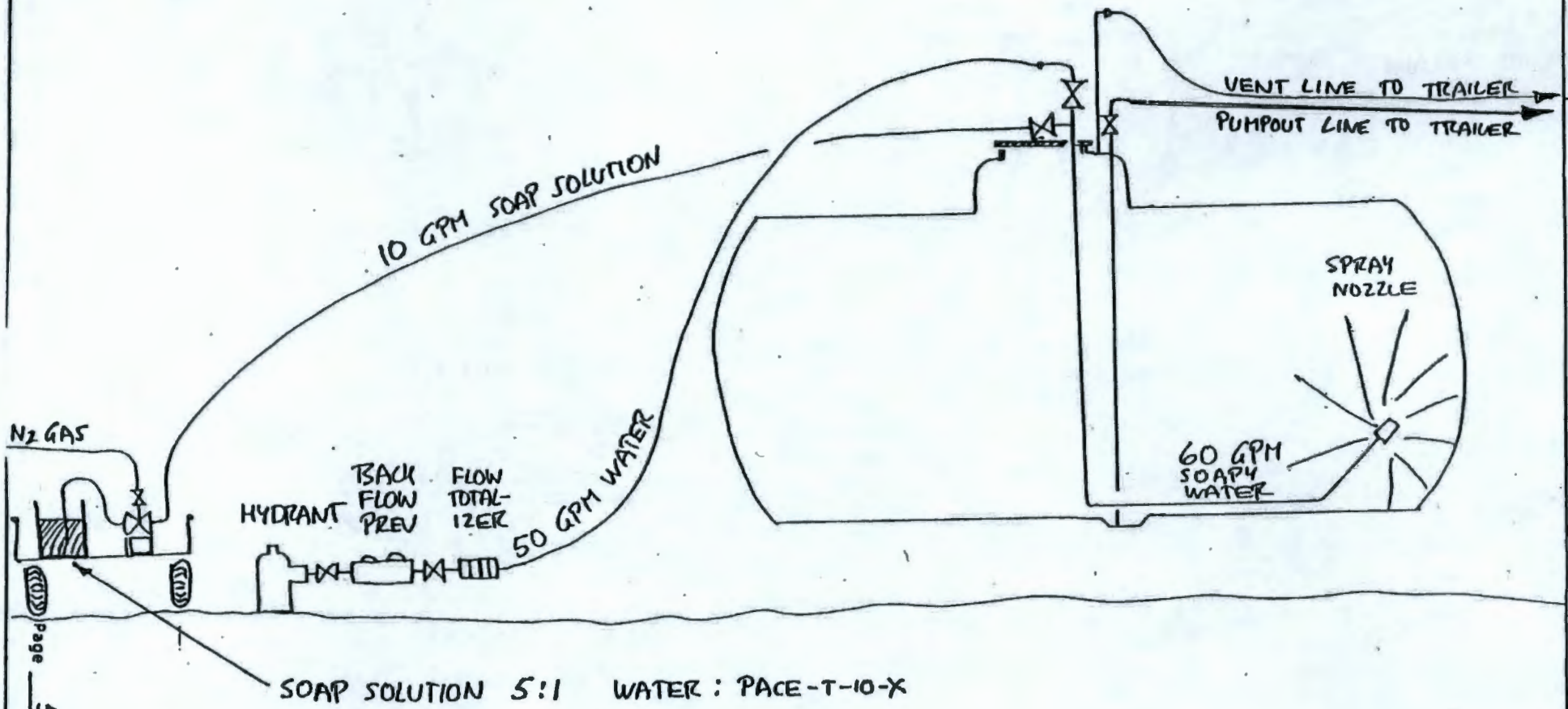
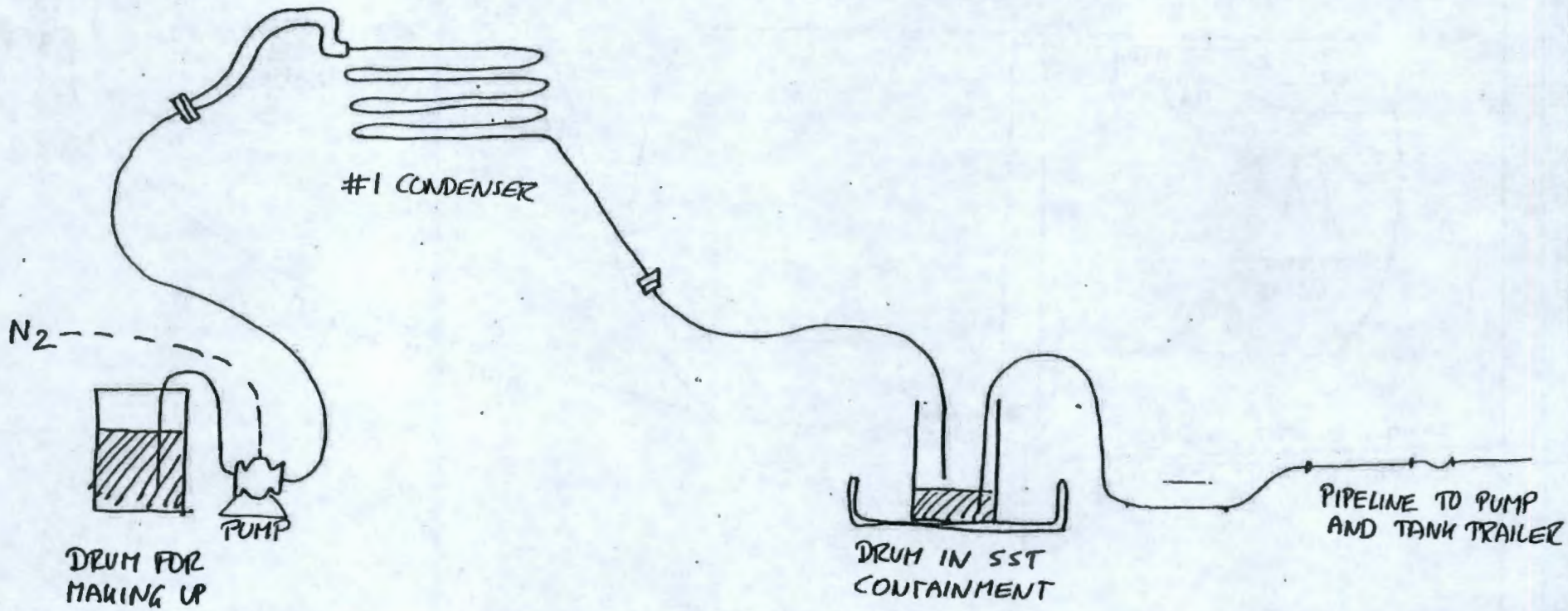


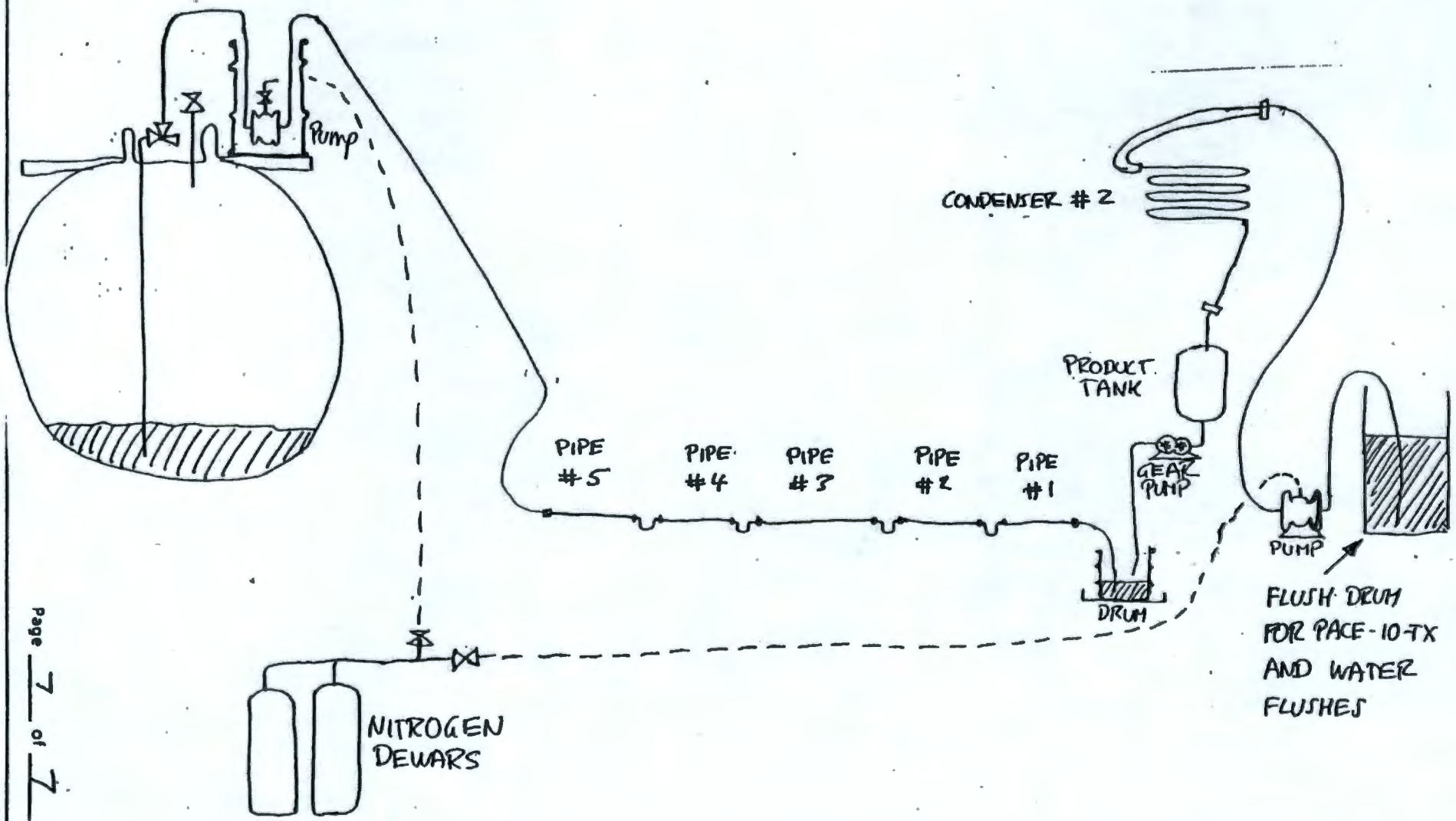
FIG. 1

JOB STEP	HAZARD	HAZARD CONTROL AND PROTECTIVE EQUIPMENT

ARRANGEMENT FOR RINSING #1 CONDENSER INTO DRUM



ARRANGEMENT FOR FLUSHING #2 CONDENSER AND PRODUCT PIPING



ENCLOSURE 8

APPROVAL FOR TRIPLE RINSE PROCEDURE

9103435

RECEIVED
JUL 29 1991
T.R. VENEZIANO

STATE OF WASHINGTON

DEPARTMENT OF ECOLOGY

Mail Stop PV-11 • Olympia, Washington 98504-8711 • (206) 459-6000

RECEIVED
AUG 01 1991
CORRESPONDENCE CONTROL

July 24, 1991

Mr. Steven H. Wisness
Hanford Project Manager
United States Department of Energy
P.O. Box 550
Richland, Washington 99352

Re: Equipment Decontamination Procedures

Dear Mr. Wisness:

We have reviewed the equipment decontamination procedures submitted by E.A. Bracken and R.E. Lerch in response to a request by Toby Michelena of my staff. This response came in a letter received by us on June 5, 1990. We find this procedure acceptable.

If you have any further questions about this matter please call Gary Anderson on my staff at (206) 438-7558.

Sincerely,

Timothy L. Nord
Hanford Project Manager
Department of Ecology

TLN:ga

cc: Dan Duncan, EPA
Dave Nylander, Kennewick
T. Veneziano, Westinghouse
S.L. Trine, DOE-RL
B.G. Erlandson, DOE-RL

9102456

Department of Energy
Richland Operations Office
P.O. Box 550
Richland, Washington 99352

91-EAB-123

JUN 5 1991

Mr. Timothy L. Nord
Hanford Project Unit Manager
State of Washington
Department of Ecology
Mail Stop PV-11
Olympia, Washington 98504-8711

Dear Mr. Nord:

EQUIPMENT DECONTAMINATION

References: (1) Meeting Minutes, "Dangerous Waste Tank System Negotiations," dated September 14, 1990.

(2) Letter, R. D. Izatt, DOE-RL, and R. E. Larch, WHC, to T. L. Nord, Ecology, "Solid Waste," dated April 25, 1990.

Reference 2 documents how the U.S. Department of Energy, Richland Operations Office (DOE-RL) has been managing solid wastes which might be considered "derived from" forms of listed waste since June 1, 1990. This reference stated "Equipment that has been flushed, rinsed, or otherwise decontaminated will be managed as low-level radioactive waste."

During a Dangerous Waste Tank System Negotiation meeting on September 14, 1990, (Reference 1), Mr. T. M. Michelena of your staff requested to review decontamination procedures before they are implemented. Enclosure 1 is a guideline for the decontamination of equipment that DOE-RL and the Westinghouse Hanford Company (WHC) proposes to implement if you concur. The guideline decontamination procedure has been developed such that it meets the "triple rinsing" criteria established for containers that have previously held acutely hazardous waste. Enclosure 2 describes why we believe this procedure meets the "triple rinsing" criteria as defined in the Washington Administrative Code 173-303-160.

Each piece of equipment has unique characteristics which will require the development of a specific decontamination procedure. The guideline decontamination procedure will be the basis used to develop equipment specific procedures. Equipment removed from service and decontaminated according to an equipment specific procedure will be handled and disposed of as non-mixed radioactive waste in accordance with WHCs radioactive solid waste acceptance criteria.

A monitoring program will be established to evaluate the effectiveness of specific decontamination procedures. Information obtained will be used to improve the decontamination process for other equipment.

91-EAB-123

If you concur with the guideline decontamination procedure, WHC will begin to utilize it as the basis for development of all equipment-specific decontamination procedures. No equipment-specific decontamination procedures will be used unless they are judged to meet the guidelines described in this letter. A typical decontamination technique for a large transfer pump that meets these guidelines is provided in Enclosure 3.

If you would like to address this issue in further detail, please contact Ms. S. L. Trine, DOE-RL, on (509) 376-6943, or Mr. B. G. Erlandson, WHC, on (509) 376-5969.

Sincerely,

for EAB

E. A. Bracken, Director
Environmental Restoration Division
Richland Operations Office

ERD:SLT

R E Lerch

R. E. Lerch, Manager
Environmental Division
Westinghouse Hanford Company

Enclosures:
1. Guideline Decontamination Procedure
2. Establishment of compliance with WAC 173-303-160
3. Typical Tank Transfer Pump Removal/ Decontamination Technique

cc w/o encls:
R. E. Lerch, WHC

Guideline Decontamination Procedure

1. Estimate inner surface area in square inches of equipment that has been in direct contact with waste listed under WAC 173-303-080 through 083. Multiply this value by 0.0018 (see Enclosure 2) to obtain the total gallons required for one flush that equals one "rinse" of the "triple rinse" required. The resulting value will be the minimum volume of flush required for one rinse of the inner surfaces. This value will be known as the minimum volume internal-flush (MVI).

2. Estimate exterior surface area in square inches of equipment assembly that has been in direct contact with listed wastes. Multiply this amount by 0.0018 to obtain the total gallons required for one flush that equals one "rinse" of the "triple rinse" required. The resulting value will be the minimum volume of flush required for one rinse of the exterior surfaces. This value will be known as the minimum volume external-flush (MVE).

3. Determine the maximum backflush rate (MBR) through jammed, blocked, or otherwise non-functioning equipment if the equipment could restrict flow during the backflush (i.e., pumps).

4. Examine equipment to determine the preferred method to perform the decontamination. For instance, the decontamination will either be a rinse and empty process with the MVI amount of water three times (i.e., tanks), or it will be a continuous flow of water to equate a "triple rinse" that allows the rinsate to fall away (or be kept away) from the component being decontaminated (i.e., pumps). Select appropriate method as applicable.

5. Examine the potential for waste to re-enter the equipment internals. If waste can and will re-enter the equipment after a backflush, provide adequate controls to prevent this before final backflushing occurs, and if necessary backflush at least one internal equipment volume of clean water through the equipment prior to the final "triple rinse" volume to assure that no tank waste can remain in the component after the "triple rinse." (The triple rinse volume calculated may not be of sufficient volume to displace the internal waste. This is possible on some pump types.)

6. For equipment to be removed from a service location that allows exterior decontamination, determine the removal rate as follows. Determine nominal flow rate of designed exterior wash system. Calculate the minimum time required (MTR) for a total flow of three times the MVE to pass through this wash system. Divide the contaminated equipment length by the MTR to establish the "quickest" permissible removal rate (PRR). (This step is applicable to pumps, thermocouple trees, etc.)

Note: Steps 7 - 16 must be completed within a determined time span (typically one work shift) to assure the flush purpose is fulfilled if the equipment is in direct contact with the waste. (i.e., pumps) It may be necessary to remove the equipment prior to conducting either internal or external flushes, therefore, the succession of these eight steps may vary depending on the type of equipment being decontaminated.

7. All rinsate that is generated during decontamination must be managed as listed, mixed waste. Therefore all rinsate must either be returned to the Double Shell Tanks or otherwise collected and stored according to the Washington Administrative Code 173-303.

8. Backflush the equipment with an amount of water at an appropriate temperature and if applicable the MBR nominal rate that will displace the waste from any piping or other equipment that will need to be disconnected. This backflush is to provide maximum flush coverage of the area that has contacted the waste.

9. Prepare the equipment to be removed by removing connections, jumpers, etc.

10. Implement controls to prevent waste from re-entering equipment internals.

11. Backflush the equipment a second time (with a minimum of three times the MVI volume or more depending on the information determined in step #5) with clean water at an appropriate temperature and if applicable the MBR nominal rate. This backflush is to be performed as determined in step #4. The backflush will be of an appropriate water temperature, flow rate and distribution which will be determined prior to the decontamination to ensure the contaminated area receives the triple rinse and the volume of water required. Appropriate controls will be implemented as necessary to meet these requirements.

12. If external contamination exists, turn on the external wash supply. This wash will be of an appropriate temperature, impact force and spray pattern providing maximum contaminated external surface coverage with a minimum of three times the (MVE) volume of continuous clean water flow.

13. Begin controlled removal of equipment at or below established PRR.

14. If applicable, stop removal when the equipment is no longer in contact with the waste. Allow an appropriate time interval for the equipment internals to drain back into the waste tank or other appropriate container.

15. Handle and/or dispose of the equipment as non-dangerous waste.

16. Record pertinent information that can be used to improve future decontamination. (i.e., Tank ID #, date, component removed - description and size, radiation readings - note shielding and distance, decontamination process used, visual data - caking, etc.)

Establishment of Compliance with WAC 173-303-160

The U.S. Department of Energy, Richland Operations Office/Westinghouse Hanford Company will utilize, with the State of Washington Department of Ecology (Ecology) concurrence, a guideline decontamination procedure for equipment removed from service. When removed and decontaminated according to the guideline, the equipment will be handled and disposed of as Ecology non-regulated empty containers. This enclosure references the applicable regulations and the technical basis for compliance with these regulations.

Pieces of equipment will be considered empty containers upon removal from service. The WAC-173-303-160 and the manual WHC-CM-5-16, "Hazardous Waste Management," requires "triple rinsing" of containers previously contacting acutely hazardous wastes in order to classify these containers as non-regulated.

The WAC-173-303-160 specifies the "triple rinse" requirements, but these requirements seem to be based on a standard shape container. Some pieces of equipment have a small volume capacity with a large contact surface area, causing existing triple rinse requirements to be inadequate as specified. A different criteria will be determined via scaling from the established 10 percent container volume rule as described in the "triple rinse" regulation.

Containers must be rinsed with a volume sufficient to allow the "acutely hazardous" component to go into solution and be carried away from the container. To establish an equivalent rinse amount, obtain the ratio of total rinse volume to contaminated surface area via scaling from the standard test case 55 gallon drum guidelines. The inner surface area of a drum is (top, bottom, and walls) approximately 3065 square inches. Ten percent of 55 gallons is 5.5 gallons. The resulting ratio of 0.0018 gallons solvent to one square inch of the contaminated surface area determines the critical ratio necessary to determine the volume of one equivalent rinse regardless of container geometry.

As defined above, one "rinse" will be an amount of solvent (determined by estimating the contaminated surface area to be flushed and multiplying it by the critical ratio above to obtain total volume.) flushed over the contaminated surfaces. Three individual "rinses" would be required to meet "triple rinse" guidelines. The rinsing may also be performed continuously until a minimum number of three "rinses" has occurred.. Due to the waste acceptance criteria at Tank Farms the solvent of choice will usually be water.

Some equipment removed from service will have an inner and an outer surface to be "triple rinsed." For the external equipment surfaces, controlled removal rate in combination with the correctly applied volume of water meets the intent of "triple rinsing" as described in Washington State regulations. To multiply the rinse effect, the external equipment surfaces will be washed with water of an appropriate impact force and temperature to maximize removal of adhered solids from the equipment body.

The inner surfaces will be backflushed with at least the "triple rinse" volume of water. The backflush will be of an appropriate temperature, rate, and distribution through the equipment to maximize contaminant removal. Certain equipment designs and conditions would allow the return of tank waste into the equipment body after backflush completion. This will be evaluated and controlled on a case-by-case basis.

Typical Tank Transfer Pump Removal/Decontamination Technique

1. Remove the transfer pump ratchet device. This will allow reverse rotation of the pump internal mechanisms.

2. Internally backflush the transfer pump and jumpers from Flush Pit 241-SY-B. The transfer pump backflush must flow a minimum volume to clean the jumper lines.

3. Internally flush the water injection line on the pump with water from Flush Pit 241-SY-A. This flush must flow a minimum volume to clean the attached lines.

4. Turn off all flush water. All rinsate is returned to the tank.

5. Open the three-way valve on the pump discharge jumper allowing fluid to drain into the tank through the recirculation line.

6. Prepare the pump to be removed by removing the jumpers.

The following steps (7 - 18) must be completed within one shift to prevent natural mixing of the clean backflush water with the tank waste.

7. Install the correct size connector with an attached valve on the pump discharge. Connect the valve to a hot water (160 degree F typical) source. Internally backflush the pump body again at an appropriate flow rate for a minimum of 75 gallons. (During the pump run in test, the pump will be locked in place and backflushed to verify what rate internal backflush is possible. The pump will be locked in place for this test to simulate a jammed or otherwise non-functioning pump. When this rate is determined, it will be used as the appropriate flow rate for the internal backflush).

8. Close the valve attached to the connector after a minimum of 75 gallons have flushed through the pump body, but before the flush source is shut off.

9. Turn off the flush water source.

10. Install the correct size connector with an attached valve on the pump water injection line. Connect the valve to a hot water source. Internally flush the injection line again at an appropriate flow rate for a minimum of 20 gallons.

11. Close the valve attached to the connector after a minimum of 20 gallons have flushed through the injection line, but before the flush source is shut off.

12. Turn off the flush water source. All rinsate is returned to the tank.

13. Bleed off flush line pressure. Remove flush line hoses. The connectors with their attached valves will be disposed of with the pump.

14. Prepare plastic wrap for use. Connect the crane. Connect the riser spray ring to a warm water (80 degree F typical) source.

15. Turn on the riser spray ring supply immediately prior to removal. Maintain spray ring water source pressure between 75 and 100 pound-force per square inch (psi). Rinse water will drain into the tank.

16. Begin pump removal. Remove pump at or below the quickest removal rate of 5.83 feet per minute.

17. Record pertinent information for use in establishing better decontamination processes in the future.

18. Lift pump suction approximately to the spray ring elevation. Stop removal. Turn off the riser spray. Wait ten minutes for pump body and injection line to drain into tank.

19. Continue standard procedure. Swing pump away from pit. Tie the plastic wrap bottom, etc.

20. Dispose of the equipment as non-mixed radioactive waste.