APPENDIX B: RUN CHRONOLOGY

The run chronology covering the period 23 April 1991 to 21 May 1991 is included in this section.

Time on stream (TOS) is assigned 0 hours at 08:00 on 1 May. This is when catalyst reduction had been completed and syngas was introduced to the reactor.

The activation run and material balance periods cover the following times:

Run #	Sta Date	rt of Pe	riod TOS	End Date	d of Per Time	iod TOS
	Date	111176	100	Date	Time	103
AF-A1	4/29	18:30		5/1	05:00	
AF-R1.2	5/3	03:00	43	5/5	13:00	101
AF-R1.1	5/5	21:00	108	5/6	17:00	129
AF-R1.3	5/6	21:00	133	5/7	06:00	142
AF-R2.1	5/9	00:00	184	5/9	17:00	201
AF-R2.2	5/10	09:00	217	5/11	12:00	244
AF-R2.3	5/11	16:00	248	5/12	16:00	272
AF-R2.4	5/12	20:00	276	5/13	04:00	284
AF-R3.1	5/14	00:00	304	5/14	16:00	320
AF-R3.2	5/15	06:00	334	5/16	08:00	360
AF-R3.3	5/16	12:00	364	5/17	09:00	385

References to flow rates, gas compositions, and space velocities reflect real-time values and have not been corrected. This has been done to retain a correspondence with the operations, start-up, and process log books and data sheets.

DATE	TIME	TOS	DESCRIPTION
4/23/91			Blinds on feedgas swung into position
4/25			First NDG calibration with nitrogen.
4/26	16:00		Introduced syngas to begin carbonyl burnout. Operation is once-through. Samples taken to measure concentration of carbonyls, sulfides, and chlorides.
4/28	11:40		Began recycling syngas
	16:30		Syngas backed-out; begin bringing nitrogen into the plant.
4/29	08:00		Charged 129 gallons of oil to Prep Tank.
	11:00		Added 489 lbs of BASF S3-86 to preheated oil in Prep Tank.
	14:00		Transferred slurry from Prep Tank to Reactor.
	15:30		Flushed Prep Tank with 97 gallons of oil and transferred to Reactor.
	18:30		Reduction gas composition lined-up at 1.2% H2, 1.8% CO, 0.5% CO2 (remainder nitrogen). Reduction begins: Reactor at 220 F and 100 psig. Reduction gas flow is 15,000 SCFH.
	18:55		Cut flow momentarily to take an NDG scan. Level at 99" on the tape.
	19:35		Reduction gas composition 1.4% H2, 1.8% CO and flow is 15,000 SCFH. This is fairly typical reactor feed composition. Reactor temperature is 221.4 F.
	21:00	•	Reactor up to 230 F
4/30	11:00		Reactor temperature at 390.5 F. Temperature ramp averaged 11 F per hour over the last 14 hours. Beginning the temperature hold.
	22:55		Reactor temperature at 391.4 F. Have maintained 391 F for 12 hours with negligible uptake of syngas. End of reduction data collection period. Beginning the ramp to 464 F.
5/1	04:00		Temperature at 464 F. Temperature ramp averaged 15 F per hour. Begin 1 hour temperature hold.

DATE	TIME	<u>IOS</u>	DESCRIPTION
•	05:00		Temperature hold period over. Begin reactor cooldown
	06:35		Reactor at 437 F, 100 psig.
	07:34		Started the feed/recycle compressor. Begin pressurizing plant. Nuke reading indicating 80" (gassed).
	07:45		Zero-flow NDG scan indicates liquid level at 69" on the tape. T=439 F, P=419 psig.
	07:50		Pressure up to 450 psig. Temperature profile in reactor ranges from 399-434F (average = 425F).
	08:00	0:00	Time zero mark. Beginning regular operations - syngas into plant
	08:03	0:03	Pressure = 690 psig. Temp profile is 407-450F (avg = 483.4F).
	08:07	0:07	PV-201 opened to 100%.
	08:18	0:18	Pressure = 760 psig. Temp profile is 409.8-468F (avg = 451F).
	08:42	0:42	Effluent sample from GC shows: H2=26.9 mole%, CO=54.8, N2=12.9, CH4=0.07, CO2=5.87, MeOH=0.37.
	08:43	0:43	NDG scan shows level at 84." FQ241 initial reading is 34819.2, FT126A=18258.1 SCFH, FT 187A=15961.8 SCFH.
	09:00	1:00	Transfer started from the 27.12 back to the reactor.
	09:07	1:07	Pump discharge pressure at the 27.12 increased to raise reactor level.
	09:20	1:20	Level detected in the 22.10. Reactor level is 90." Target is 140."
	09:30	1:30	Pressure = 752 psig. Temp profile is 393.3-463.1F (avg = 439F).
	10:02	2:02	Reactor Feed is 39% H2, 56% CO, 5% CO2. Reactor feed is FT187B=20125 SCFH. Tavg=448 F, P=751 psig

DATE	TIME	TOS	DESCRIPTION
	10:30	2:30	Oil drained from flare KO pot = 46 lbs. A small amount of clumpy catalyst was removed but oil was relatively clear.
	10:35	2:35	Reactor effluent is 23.5% H2, 61.3% CO, 7.8% CO2, 10.8% MeOH
	10:40	2:40	Pressure = 748 psig. Temp range is 409.2-468.8F (avg = 449.8F). Level is at 108". FT126A=17543, FT187A = 4066, FT187B=19400.
	10:53	2:53	Level is at 112."
	11:58	3:58	Level is at 131."
	12:10	4:10	Recycle flow started. Nuke indicating a level of 140."
	12:17	4:17	FT126A = 20139, FT187A=40421, FT187B=40129. Level = 168."
	12:40	4:40	Oil addition stopped.
	13:27	5:27	Nuke on automatic control, level at 180." Temp range is 444.1-478.9F (avg=478F).
	15:15	7:15	Started raising nuke level to 213."
	16:59	8:59	Started to transfer product from 22.15 to day tank. FQ241 not registering. 22.15 level is at bottom of 10th bolt, day tank starting point is 19.5."
	18:38	10:38	Slurry sample taken off reactor.
	22:30	14:30	The 8.5% MeOH standard was suspected of partial condensation in the cylinder. The 3.8% std will now be used for the GC calibration. John Wallace correction factors: GC1: 0.9562 for MeOH > 8% GC2: 0.9375 for MeOH > 8%
5/2	00:00	16:00	Increased recycle to reactor.
	00:15	16:15	Transfer from 22.16 to 28.10. 22.16 readings: start=43.5," end=19.5."

DATE	TIME	TOS	DESCRIPTION
	04:00	20:00	Plant and GCs have been running steady. Reactor flow =63,000 SCFH Inlet Comp = 38% H2, 48% CO, 13% CO2 Effluent = 24% H2, 48% CO, 16% CO2, 10% MeOH Reactor T = 480 F, P = 751 psig
	09:00	25:00	Oil addition stopped to let level drop.
	12:20	28:20	Oil sample taken off the 27.14, MeOH sample taken off the 22.11 (96.7 wt% MeOH).
	12:45	28:45	Flow to the reactor was increased to 85,000 SCFH to bring SV to 9000. Will maintain this condition to track activity reduction and eventually enter the AF-R1.2 data period. With the increase in flow, the bottom four TI's in the reactor tightened to within 2F. Previously, these TI's ranged up to 10F. Better mixing is noted. Reactor level is above nuke, recycle is reduced to bring level down.
	13:35	29:35	Reactor TI's started diverging again. Flow had been dropped to only 83,000 SCFH.
	14:20	30:20	Level is 211." Temp profile varies 4.5 F between Tl262-2 and Tl626-5.
	18:30	34:30	First transferred from day tank to tanker (approx. 315 gallons)
	23:20	38:40	The iron carbonyl level in reactor feed was measured with GCs to be 2-3 ppb.
	23:45	39:40	MeOH in reactor effluent is consistent and around 10.7 mole%.
5/3	03:00	43:00	Start of Data Collection Period for AF-R1.2
	03:10	43:10	Began transfer from day tank to trailer.
	05:40	45:40	End of transfer (770 gallons)
	10:30	50:30	Methanol composition in effluent continues to fluctuate. Blew out sample pots.
	12:00	52:00	Transferred from day tank to trailer. Unloaded about 1480 gals, current production is about 3000 gals. New tubing installed which made transfer go quickly.

DATE	TIME	TOS	DESCRIPTION
ı	12:00	52:00	Oil sample taken off the 27.14, MeOH sample taken off the 22.11 (96.8 wt% MeOH).
	14:00	54:00	Problem found with LIC 292 on 22.15. The level in the sight glass must be taken into account when day tank readings are taken. Began to fill the 21.80 evaporator with CO2.
	16:00	56:00	Mass balances between 0600-1600 5/3 indicate productivity to be: 33.2 gmole/kg-hr by GC and flows 28.3 gmole/kg-hr by day tank.
	23:40	63:40	DEC computer did not update from now until 5/4, 0300.
5/4	00:30	64:30	Mass balances between 5/3 1600-5/4 0030 show productivity to be: 32.6 gmole/kg-hr by GC and flows 27.6 gmole/kg-hr by day tank.
	11:25	75:25	Transferred from day tank to trailer (1710 gal).
	12:00	76:00	Oil sample taken off the 27.14, MeOH sample taken off the 22.11 (96.7 wt% MeOH).
	18:00	82:00	Needle gauge on CO2 supply was found to be off. CO2 is used with discretion.
5/5	10:35	98:35	Transferred from day tank to trailer (1700 gal).
	12:00	100:00	Oil sample taken off the 27.14, MeOH sample taken off the 22.11 (96.7 wt% MeOH).
	13:00	101:00	End of AF-R1.2 Data Period. Reactor flow reduced to 46,780 SCFH, SV is nominally 5000.
	20:00	108:00	Plant as lined-out sufficiently to begin AF-R1.1 Data Period.
5/6	00:00	112:00	Begin to purge-out the DME recovery section.
	11:00	123:00	Charged the prep tank with estimated 110 gallons of oil. Will begin heating to a target temperature of 180 F.
	11:15	123:15	Transferred from day tank to trailer(1340 gal). Oscillations in measured methanol composition of reactor effluent are fairly substantial - time average composition is stable.

DATE	TIME	TOS	DESCRIPTION
	12:00	124:00	Oil sample taken off the 27.14, MeOH sample taken off the 22.11 (96.1 wt% MeOH).
	18:00	130:00	Mark end of AF-R1.1 Data Period. Begin to increase rates to the original SV=9000 conditions (AF-R1.2). Will discontinue oil pump-back from 27.14 to force the slurry to thicken. Current level at 211" on the tape.
	21:30	133:30	Added 32 lbs of alumina to oil in the prep tank and continue heating. The objective is to heat mix in prep tank at 250 F for 12 hours to drive off water. Prep tank is under N2 Purge.
5/7	0:00	136:00	Prep tank contents at 256 F. Pressurized then depressurized three times to aid in getting the water into the N2 purge.
	01:10	137:10	Down to approx. 157" on tape slurry level. Effluent MeOH composition has returned to AF-R1.2 levels, as expected.
	02:45	138:45	Level control for reactor put back on automatic to hold slurry level at 149" on the tape.
	07:30	143:30	Level set point had been reduced further, level now at 140". Gas-to-reactor is temporarily cut-out and slurry allowed to degas and settle. After settling, level was 67" on the tape.
	09:15	145:15	Syngas backed-out and replace with nitrogen (+2% H2) - End of AF-R1 series. Will begin cooling the reactor in preparation for the slurry transfer operation.
	11:00	147:00	Drained MeOH out of 22.10 and 22.15 into the day tank.
	11:40	147:40	Dropped plant pressure to 150 psig.
	12:25	148:25	Reactor at 250 F, prep tank at 265 F. Drained 78 lbs of reactor slurry to drum S1.
	13:00	149:00	Transferred 1/3 of reactor contents to prep tank, withdrew a slurry sample from reactor, then transferred the rest of the reactor contents to prep tank. Begin 2 hour hold in prep tank under agitation to blend alumina with MeOH catalyst.
	14:50	150:50	Begin transfer of prep tank contents to reactor. To be followed by flush of prep tank with 35 gallons of oil.

DATE	TIME	TOS	DESCRIPTION
	15:30	151:30	Transfer prep tank rinse to reactor and bring-in N2 purge flow (+2% H2) to reactor and begin heat-up.
	17:00	153:00	Drained 27.12 and 27.14 to bottom of their respective sight glasses.
	20:50	156:50	Reactor at 430 F. The new slurry level is too high in the reactor (above the span of the NDG). Will have to carefully boil-off oil with nitrogen flow. Continue increasing reactor temperature towards 450 F.
5/8	05:15	165:15	Emptied day tank into trailer.
	08:20	168:20	Cut-out N2 flow to measure degassed level. Level is 137" degassed (70 psig, 450 F) - sufficient to proceed with regular operations.
	08:45	168:45	Begin to raise reactor pressure under nitrogen.
	09:45	169:45	Reactor at 756 psig, recycle flow started.
	10:28	170:28	Flow stabilized at 40,000 SCFH. Reactor at 737 psig, 421 F.
	11:00	171:00	Begin to bring-in syngas.
	12:41	172:41	Reactor at 744 psig, 443 F. Reactor flow is approx. 44,000 SCFH. Reactor effluent composition is 21%H2, 41% CO, 8% CO2, 25% N2, 4% MeOH and 2% DME. The reaction is hot! Will ramp the temperature up slowly.
	16:05	176:05	Reactor at 756 psig, 470 F. Reactor flow is approx. 48,200 SCFH. Reactor effluent composition is 22%H2, 49% CO, 16% CO2, 3% N2, 5% MeOH and 5% DME.
	17:30	177:30	Levels are building in 22.10 and 22.15
	20:20	180:20	22.10 and 22.15 operating on level control - liquid being transferred to day tank. Back-end is cooled, 22.18 is at 10 bolts - begin to transfer liquid from 22.14 to 22.18.
5/9	00:00	184:00	Begin data collection period for run AF-R2.1 (SV=5500). Reactor at 750 psig, 481 F. Reactor feed flow is 40,000 SCFH at 36%H2, 50.5% CO, 1% N2, 11% CO2, 1% DME. Effluent composition is 22% H2, 46% CO, 1% N2, 19% CO2, 5.8% DME, 6.3% MeOH.

DATE	TIME	TOS	DESCRIPTION
	06:30	190:30	Alumina catalyst appears to have initial hyperactivity. DME concentration in reactor effluent has dropped to 5.7%.
	08:30	192:30	22.11 liquid sample taken at 03:45 contained 9% DME and degassed considerable. Will attempt to reduce DME in liquid by increasing 22.10 temperature. To achieve this, cooling water flow to 21.30 is reduced.
	11:00	195:00	Cooling water temperature exit 21.30 is too hot (150 F) and has had no effect on 22.10 temperature (21.30 is oversized and pinched). Cooling water flow is returned to normal.
	12:15	196:15	DME in effluent continues to decline, now at 5.6%. MeOH in effluent remains fairly stable. Liquid has been accumulating in the 22.18 (level at 1 bolt).
	14:00	198:00	Begin to thicken slurry - stop returning oil flow from 27.14.
	15:00	199:00	Completed construction of a special sample bomb for 22.11 liquid. Sample will be taken at 22.11 pressure, weighed, blown-down to atmospheric pressure, then weighed again. The weight loss is the quantity of degassed vapor.
	17:00	201:00	End of AF-R2.1 data period. Final DME concentration in effluent is 5.4%. Will begin to increase rates and continue to boil-off oil. Target reactor feed gas flow is 74,500 SCFH.
5/10	03:30	211:30	Reactor feed flow up to 64,500 SCFH. Level continues to build slowly in 22.18 (now at 3 bolts).
	09:00	217:00	Up to desired rates. Begin data period for run AF-R2.2 (SV=9000). Reactor at 481 F, 750 psig. Reactor feed flow is 75,600 SCFH at 36% H2, 51% CO, ½% N2, 12% CO2, 1% DME. Reactor effluent is 24% H2, 48% CO, ½% N2, 17% CO2, 3.7% DME, 6.7% MeOH. Level in 22.18 is now at 5.5 bolts.
	12:00	220:00	Liquid sample from 22.11 shows 3.5% DME, 93.5% MeOH.
	21:20	229:20	Lost FT187A from 20:45 (loose wire - short), now back on line.

DATE	TIME	TOS	DESCRIPTION
5/11	0:00	232:00	Plant has been running steady. No change in MeOH or DME concentration in reactor effluent. 22.18 level up to 15 bolts.
	07:15	239:15	22.18 is at 21.5 bolts will dump liquid to 28.10 storage tank.
	08:30	240:30	Transfer complete, level is 2 bolts. Sample from 22.18 indicates 86% MeOH, 9% DME, 3% water.
	12:00	244:00	End of data period for run AF-R2.2. Begin to reduce rates to for next condition, target flow is 47,000 SCFH.
	16:00	248:00	Plant responded quickly to condition change. Begin data period for run AF-R2.3 (SV=5400). Reactor at 482 F, 750 psig. Reactor feed flow is 46,900 SCFH at 35% H2, 52% CO, ½% N2, 12% CO2, 1% DME. Reactor effluent is 21% H2, 48% CO, ½% N2, 19% CO2, 5.1% DME, 6.5% MeOH. Level in 22.18 is now at 6 bolts.
5/12	04:30	260:30	Plant operation very steady. 22.18 at 10 bolts.
	10:15	266:15	Alumina prep for next campaign underway. Approx. 62 gallons of oil added to prep tank, followed by 67.5 lb of alumina, followed by another 25 gallons of oil. Begin heating prep tank under nitrogen.
	14:15	270:15	Prep tank temperature is at 246 F.
	16:00	272:00	End of data period for AF-R2.3. Increase rates (target is 76,000 SCFH) and begin to thicken slurry (stop oil return from 27.14).
	20:00	276:00	Are at new conditions and stable. Begin supplemental data period (run AF-R2.4, SV=9000). Reactor at 481 F, 750 psig. Reactor feed flow is 76,400 SCFH at 36% H2, 51% CO, ½% N2, 11% CO2, 1% DME. Reactor effluent is 23% H2, 50% CO, ½% N2, 15% CO2, 3.6% DME, 6.9% MeOH.
	21:30	277:30	Prep tank at 293 F.
5/13	02:10	282:10	Transferred day tank to 28.10.
	04:00	284:00	Operation has been steady and quite similar to run AF-R2.2. Thickening of the slurry has been going slowly so will begin to increase rates further - end of data period for run AF-R2.4.

DATE	TIME	TOS	DESCRIPTION
	05:30	285:30	22.18 liquid transferred to 28.10. Prep tank holding at 290 F.
	06:30	286:30	Feed flow to reactor at 93,700 SCFH.
	08:40	288:40	Begin reactor cooldown. Target temperature 250 F
	09:15	289:15	Reactor at 430 F. Back-out syngas and bring in N2 (+2% H2).
	11:40	291:40	Reactor at 301 F and 504 psig. Temporarily cut-out N2 flow to measure degassed slurry level (54") - OK to proceed.
,	12:05	292:05	Drained 150 lb of slurry into a drum and took a sample. Transferred the remainder to prep tank. Will agitate in prep tank with the fresh alumina until 14:00.
	14:15	294:15	Transferred prep tank contents to reactor.
	14:30	294:30	Rinsed prep tank with 24 gallons of oil and transferred to reactor. Reactor at 272 F, 42.5 psig - level is 116". Begin heating slurry in reactor under nitrogen.
	18:00	298:00	Reactor at 400 F, 750 psig. Bring in syngas and operate once-through to purge N2.
	19:00	299:00	Reactor at 430 F. Reactor flow is 36,000 SCFH (operating with partial recycle).
	20:30	300:30	Reactor at 460 F. Reactor flow is 48,000 SCFH. Reactor effluent composition is 24% H2, 48% CO, 8% N2, 12% CO2, 5% DME, 3% MeOH. Once again, the catalyst mix is extremely active.
	21:15	301:15	Reactor up to 480 F.
5/14	00:00	304:00	Plant is pretty well lined-out. Begin data period for run AF-R3.1 (SV=5400). Reactor at 481 F, 750 psig. Feed flow is 49,400 SCFH at 36% H2, 51% CO, 1.5 N2, 10.5% CO2, 1% DME. Reactor effluent composition is 23% H2, 46% CO, 1.5% N2, 19.5% CO2, 7.6% DME, 2.4% MeOH.
	04:40	308:40	Levels in 22.10 and 22.15 lined-up - beginning to transfer to day tank. Levels in 22.14 lined-up; level in 22.18 at 15 bolts.

DATE	TIME	TOS	DESCRIPTION
	06:30	310:30	Reactor effluent composition is 23% H2, 46% CO, 1% N2, 20% CO2, 7.6% DME, 2.4% MeOH.
	12:00	316:00	Operation still steady - effluent is 7.5% DME, 2.4% MeOH. Sample from 2218 shows 8% DME, 89% MeOH. Level in 22.18 still 15 bolts.
	12:45	316:45	Begin draining 22.18 to the 28.10.
	14:00	318:00	End of data period for run AF-R3.1. Begin to thicken slurry in preparation for higher rates.
	16:30	320:30	22.18 drained down to 1 bolt. Begin to increase flow rate to reactor.
	18:00	322:00	Reactor flow at 55,400 SCFH, will stay here for a while and continue thickening. Have increased pressure of 22.18 to 170 psig - will now begin draining 22.18 into 28.40.
5/15	00:30	328:30	Reactor flow up to 59,800 SCFH.
	05:30	333:30	Reactor flow at 74,000 SCFH.
	06:00	334:00	Plant is now lined-out. Begin data period for run AF-R3.2 (SV=9000). Reactor at 482 F, 750 psig. Feed flow is 74,900 SCFH at 36% H2, 50.5% CO, ½% N2, 12% CO2, 1.5% DME. Reactor effluent composition is 27% H2, 47% CO, ½% N2, 17% CO2, 5% DME, 2.9% MeOH. Blocked 22.18 discharge to allow level in 22.18 to rise as a means of estimating rates.
	08:35	336:35	Level in 22.18 is up to 16 bolts. Open valve to allow liquid to continue to drain into 28.40 (28.40 is currently 25% of max. level).
	18:30	346:30	Level in 28.40 at 46%. Operation is pretty steady with some small variations in H2 concentration in the loop.
5/16	01:45	353:45	Level in 28.40 is 70%. Block-in 22.18 discharge line and drop 22.18 pressure back to 23 psig. Plant operation largely unaffected.
	08:00	360:00	End of data period for run AF-R3.2. Begin reducing rates.
	09:30	361:30	Rates are at design flow of 46,400 SCFH.

DATE	TIME	TOS	DESCRIPTION
	12:00	364:00	Temperature oscillating somewhat which is showing up as +/- 0.2% in DME concentration in reactor effluent. Currently returning controllers. Average production is not affected so will mark this as beginning of run AF-R3.3 data period. Reactor at 480 F, 750 psig. Reactor feed flow is 46,800 SCFH at 36% H2, 51% CO, ½% N2, 11% CO2, 1% DME. Reactor effluent is 25% H2, 47% CO, ½% N2, 15% CO2, 6.9% DME, 2.9% MeOH.
	18:00	370:00	Reactor temperature back under tight control - DME composition in effluent has stabilized.
5/17	00:30	376:30	Plant running steady.
	09:00	385:00	End of data period for run AF-R3.3. Have been operating at reduced level. Begin to pump oil from 27.14 to reactor in order to increase level to 100%.
	10:00	386:00	Level up to 100% in reactor. Will take data for 6 hours to see if productivity/selectivity change.
	13:40	389:40	There is no significant change in productivity but may be a slight increase in DME selectivity.
	14:00	390:00	Regular operations end. Begin reactor/plant cooldown. Syngas out, N2 in
	17:30	393:30	Compressor shut down.
	17:50	393:50	Reactor at 200 F. Shutdown data computer. Begin draining liquid from reactor. Drain into drums D1-D5.
5/20			Plant has been down under nitrogen purge over the weekend
	07:00		NDG calibration performed under nitrogen at 198, 55, and 6.5 psig.
			Reactor top and bottom heads removed for vessel inspection. Top head is very clean, internal heat exchanger piping has 1/8" of crusted catalyst on horizontal runs, reactor walls show slight "film" but can clearly see metal surface. Bottom head and 4" liquid slurry line are a different story.

DATE TIME TOS DESCRIPTION	DATE	TIME	TOS	DESCRIPTION
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5/21

Bottom head is filled with slurry up to about 1" below the bottom of the sparger. Scooped-out 74 lbs of mixed oil and catalyst. Most of this material was black with intermixed brown and red. Some material was very dry and crumbled when held.

The 4" line between reactor head and block valve was also filled - 14 lbs removed.

Bottom plate was removed from 27.14. Inside was clean with only 3/4" of slurry covering the 12" plate.

The inlet head was removed from the 22.10 exchanger to inspect tubes. Very clean inside tubes with a little bit of catalyst on the tubesheet.