Dept. 246 - Operating Instructions

SECTION VI - DETAILS OF OPERATION OF THE STILL OPERATOR

H. #1 & 2 STILL (SEMI-CONTINUOUS DISTILLATION)

1. Synopsis

The distillation systems of #1 & 2 stills are similar to each other and they are both straight takeover. The systems consist of a still pot, condenser, receiver, furnace and vacuum. Crude aroclor in the still pot is circulated through the gas furnace where it is heated to its boiling point. The vapors are pulled by vacuum through the water-cooled condenser where they are liquified and fall into the receiver. ~~#1 still is charged with 30% from a batch blow tank; #2 still is~~ #2 Still is charged with 45% from a batch blow tank. ~~150# of lime is added to tie up impurities such as HCl.~~ When the vapors condense, the liquid is clear as it falls into the receiver.

~~Most of the time,~~ #2 still is used as a "boildown" still. The highboilers are pumped over from #3 & 4 stills into #2 and #2 is boiled down. ~~Usually 5 pump overs are made before bottoms are dropped from #2 still.~~ #2 still is the only still equipped to drop bottoms.

2. Equipment

a.	~~#1 Still~~	~~Item 130~~
b.	~~#1 Still Circulating Pump (Submerged)~~	~~Item 132~~
c.	~~#1 Still Furnace~~	~~Item 131~~
d.	~~#1 Still Condenser~~	~~Item 136~~
e.	~~#1 Still Receiver~~	~~Item 138~~
f.	~~#1 Receiver Pump~~	~~Item 140~~
g.	#2 Still	Item 133
h.	#2 Still Circulating Pump (North)	Item 168
i.	#2 Still Circulating Pump (South)	Item 177
j.	#2 Still Furnace	Item 134
k.	#2 Still Cyclone Separator	Item 200
l.	#2 Still Condenser	Item 137
m.	#2 Still Receiver	Item 139
n.	#2 Receiver Pump	Item 141

3. Equipment Function

a. ~~The function of #1 still is to provide a vessel for holding crude aroclors while it is circulated and heated to its boiling point.~~

b. ~~The function of the still pump is to circulate the crude aroclor through the furnace and keep the lime in suspension. The pump is submerged.~~

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3. Equipment Function (cont'd.)

c. The function of #1 still furnace is to transfer heat to the crude aroclor as it is circulated through a coil inside the furnace. The furnace is gas fired.

d. The function of #1 still condenser is to liquify the aroclor vapors as they pass through it. The vapors are in the tube side; the cooling water is on the shell side.

e. The function of #1 still receiver is to collect the distilled aroclor. When the receiver is full, it is pumped into ~~#1 or #2 blend~~ #1 TODAY tank.

f. The function of #1 receiver pump is to move the distilled aroclor from the receiver to ~~#1 or 2 blend~~ #1 TODAY tank.

g. Same as (a) except it is also used for boiling down and dropping bottoms (#2 still).

h. & i.
Same as (b) except the pumps are external.

j. Same as (c).

k. The function of the cyclone separator is to remove any entrainment (liquids) from the vapors and return them to the still.

l. Same as (d).

m. Same as (e).

n. Same as (f).

4. Control Loops, Interlocks and Alarms

a. Control Loops

1) Level Control (#1 LRC-103, #2 LRC-100)
The function of this loop is to maintain and record the level of material in #1 & 2 still pots.

2) Vacuum Control (#1 PRC-104, #2 PRC-101)
The function of this loop is to control the vacuum on the still pot. Set point is 40 mm (absolute

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4. Control Loops, Interlocks and Alarms

a. Control Loops (cont'd.)

3) Receiver Level Recorder (#1 LR-105, #2 LR-102) The function of this loop is to record the level in #1 & 2 receivers. Both levels record on the same chart in the control room.

b. Interlocks

There are no interlocks on these stills.

c. Alarms

1) If the circulating pump on #1 still kicks off or is shut off, an alarm will sound and telealarm window marked "#1 STILL PUMP OFF" will light.

2) If the circulating pump (or spare pump) for #2 still kicks off or is shut off, an alarm will sound and telealarm window "#2 HORIZONTAL STILL PUMP OFF" will light.

5. Startup Instructions

a. #1 & 2 Stills

1) Close the outlet valve on the still pot (#2 only) and receiver.

2) Align the valves to charge the still for #1 RESIDUE blow tank, bypassing the automatic level control valve.

3) Transfer crude from #1 RESIDUE blow tank to stills as follows:

~~#1 still charge is 30%~~
#2 still charge is 45%

4) Shut off the charging pump and close the bypass valve.

5) Align the valves and start circulating the crude through the furnace coil.

6) NOTE: Check the multi-point recorder for temperatures of coil in and coil out. These two temperatures must run within 10 degrees of each other.

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5. Startup Instructions

a. ~~#1~~ & 2 Stills

7) Apply vacuum to the still (~~per instructions in Section F~~).

~~8) Add 150# of lime to the crude in the still pot. Be "positive" it is circulating first.~~

~~9) Tighten the lid on the lime port securely so it will hold vacuum.~~

10) Set the vacuum controller on 40 mm.

11) When the vacuum is down to 40 mm, put 5 lbs. of gas on each furnace burner.

12) Watch the 12-point recorder for temperatures of coil in and coil out. They must rise together as the crude heats.

13) When the temperature of the coil out reaches about 180°C, turn water on the still condenser.

14) The fires on the burners can be increased if necessary.

15) When the material starts to flow, check the level control set point and the valves around the automatic; be sure the bypass is closed.

16) Turn steam on the receiver ~~for 1248, 1254 & 1260.~~ #7 STORAGE

17) When the receiver fills, pump it into ~~#1 or 2 blend tanks. Each blend tank will hold 2,000 gal. or 2 receivers.~~ HAVE 2ND OPERATOR CHECK FOR VALVE ALIGNMENT

18) ~~After 15,000 gal. of crude has been distilled, add 150# of lime to the still pot as follows:~~

a) Turn the gas off the furnace burners.
b) Close a manual block valve in the feed line to the still.
c) Break the vacuum by opening the vent valve on top of the receiver. When the vacuum is down to zero, open the pressure release valve on top of the still pot. NEVER release the vacuum by using the valve on the still pot; this will cause color problems.

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5. Startup Instructions

b. Pumpover - #1 Still

10) Watch the charts in the control room for #2 still level and #2 still vacuum. The level should raise until #1 still is empty, then #2 still will lose vacuum.

11) Close the valves that were opened to transfer the material from #1 still to #2 still.

12) Close the vacuum release valves on #1 still and receiver.

Recharge using the startup instructions in Section (a).

c. Boildown on #2 Still

~~#2 still can be operated as a semi-continuous still as #1 is, but most of the time it is used as a "boildown" still. Highboilers are pumped into it from #1, 2 stills. One pump-over is normally 45% in #2 still. When a second pump-over is made, the level will be about 90%. It is then boiled down to 40% on the level chart to make room for another pump-over. After 5 pump-overs are made, the still is boiled down and bottoms are dropped into open top drums. (No lime is added to this still when it is used for bottoms.)~~ RESIDUE TANK

1) For the 1st pump-over, close the outlet valves on the bottom of the tank.

2) Apply 40 mm of vacuum to the still (per instructions in Section F).

3) Make the pump-over from the ~~appropriate still~~ RESIDUE TANK.

4) Align the valves to circulate the material through the furnace coil and back into the still.

5) Turn on the circulating pump. Check the following to be certain the material is circulating:

i. Pressure on the circulating line - 45-50 lbs.
ii. Ampmeter - 18-20 amps
iii. The 12-point recorder for temperatures. Coil-in and coil-out should not be more than 8° apart.

(If it isn't circulating, the vacuum may have to be released before it will.)

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H. ~~#1~~ & 2 STILLS

5. Startup Instructions

c. Boildown on #2 Still (cont'd.)

~~6) Check that the vacuum is down to 40 mm.~~

~~7) Apply just enough gas to the furnace to hold the temperature around 200°C on the coil outlet (5# on 1 burner).~~

~~8) The 2nd pumpover, and others, can be made to #2 still without shutting down the circulating pump.~~

~~9) After each pumpover (except the 1st and 5th), boil down #2 still until the level recorder is on 40. (Use 20# of gas on each burner when boiling down between pumpovers.)~~

#2 STORAGE.

10) When #2 receiver fills, pump it into ~~#2 blend tank~~

d. Boildown to Drop Bottoms on #2 Still

Dropping bottoms should be scheduled so another ~~still~~ CHARGE is ready to be pumped over immediately after the bottoms are out. Usually the boildown is started at 10:30 p.m. so bottoms will be ready to drop shortly after 8 a.m. the following day.

1) Circulate the material in #2 still and check the following before applying any heat to the furnace:

   a) Coil-in and coil-out temperature should be within 8° of each other.

   b) Circulating line pressure - 45-50 lbs.

   c) Ampmeter - 18-20 amps

   d) Vacuum - 40 mm

2) Put 10 lb. of gas on all 4 furnace burners.

3) Reduce the pressure on the circulating line by 10 lbs. by pinching back on the discharge valve just above the circulating pump.

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5. Startup Instructions

d. Boildown to Drop Bottoms on #2 Still (cont'd.)

4) Normal boiling temperature is about 240°C on the 12-point recorder. When the vapor temperature raises to about 75°C, turn the water on the still condenser.

5) The boildown should go normal until the level is down to about 40 on the chart. Watch closely the forward flow, the amps, the pressure on the circulating line and the temperature difference between the coil-out and the vapor.

Stop the distillation if any one of the following exist, no matter which comes first:

a) Temperature difference between coil-out and vapor - 45° Max.
b) Amps should not raise more than 2 from starting point.
c) Pressure on the circulating line - 15 lb. Min.
d) Forward flow not reaching the far side of the sight glass.

6) Shut the fires off the furnace (3rd floor).

7) Close the big valve under the still (1st floor).

8) Open the valve on the pump discharge, all the way (1st floor).

9) Start sucking the coil by opening the 1" valve on the suction side of the pump (1st floor).

10) Open the bypass valve, just under the furnace, into the side of the still for 10-15 seconds. Then close valve. (2nd floor)

11) When the coil is sucked clean (2 or 3 minutes), close the 1" valve at the pump. (1st floor)

12) Close the valve just above the circulating pump. This will usually prevent the pump from freezing. (1st floor)

13) Shut off the circulating pump. (1st floor)

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5. Startup Instructions

d. Boildown to Drop Bottoms on #2 Still (cont'd.)

14) Turn steam and water on the fume removal jet for the drum cabinet under the still. (1st floor)

15) Test the thermometer to be sure it will work properly. Blow live steam on it or hold it on a steam line. (1st floor)

16) Put a drum on the two-wheel cart and set it in the cabinet. Close the doors. (1st floor)

17) Close the ram valve all the way. This will break the crust over the valve. (1st floor)

18) Open the ram valve all the way until vacuum sucks up hard into the bottom of the still; then close the ram valve all the way. (1st floor)

19) Break the vacuum by opening the vacuum release valve on top of #2 receiver, slowly but all the way. (2nd floor)

20) Lower the thermometer on top the cabinet so the stem will be in the empty drum. (1st floor)

21) Open the ram valve all the way. This will allow Montar (bottoms) to fall through the line into the drum.

22) Watch the thermometer very closely. When the hand starts moving rapidly, close the ram valve all the way.

23) Remove the thermometer and wipe off the end.

24) Open the cabinet doors, and as one operator pulls the full drum out, the other operator places a lid on it to hold down fumes and prevent spillage.

25) Place the full drum by #3 mix tank.

26) Continue to fill the open top drums until the still is completely empty, usually 18-20 drums.

27) Turn the steam and water off the fume removal jet on the cabinet.

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H. #1 & 2 STILLS

5. Startup Instructions

d. Boildown to Drop Bottoms on #2 Still (cont'd.)

28) Pump over the ~~still~~ RIG 104R that is ready and circulate it as soon as possible. The longer the still cools, the greater the chances of pluggage from Montar.

29) Clean up any spilled Montar as soon as possible.

6. Normal Operations

During normal operations, the following conditions can be expected:

a. #1 Still

1) No more than 18 lbs. of gas on each burner.
2) Amps between 4.5 and 5.5.
3) Receiver filling in 5-6 hours.
4) Water on the condenser.
5) Level on chart on 20.
6) Coil-in and coil-out temperature within 8° of each other.
7) Vapor temperature about 10° below coil-in temp.
8) Vacuum on 40 mm.

b. #2 Still (When Run Like #1)

1) Any amount of gas on each burner.
2) Amps between 18 and 20.
3) Receiver filling in 4.5 to 5.5 hours.
4) Water on condenser.
5) Level on chart on 50.
6) Coil-in and coil-out temp. within 8° of each other.
7) Vapor temp. about 10° below coil-in temp.
8) Vacuum on 40 mm.
9) Circulating line pressure between 45 & 50 lbs.

c. #2 Still (Boiling Down Between Pumpovers)

1) 20# of gas on each burner.
2) Amps between 18 and 20.
3) Water on condenser.
4) Level on chart between 90 and 40.
5) Coil-in and coil-out temp. within 8° of each other.
6) Vapor temp. about 10° below coil-in temp.
7) Vacuum on 40 mm.
8) Circulating line pressure between 45 and 50.

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6. Normal Operations

d. #2 Still (Boiling Down to Drop Bottoms)

1) 10# of gas on each burner.
2) Amps slowly increasing between 18 and 20.
3) Water on condenser.
4) Level on the chart dropping between 50 and 40.
5) Coil-in and vapor temperatures slowly spreading to 45° MAX.
6) Vacuum on 40 mm.
7) Circulating line pressure slowly falling from 35 to 15 lbs. MINIMUM.

7. Shutdown Instructions

a. Emergency

1) #1 & 2 Stills (During Normal Operations)

a) Shut off the gas on the burners.
b) Suck the coil.
c) Close a manual block valve in the feed line.

2) #2 Still (Boiling Down to Drop Bottoms)

a) Shut off the gas to the burners.
b) Suck the coil.

b. & c. Normal - Short Time and Long Time

1) #1 & 2 Stills

a) Shut off the gas to the burners.
b) Close a manual block valve in the feed line.

d. Electrical Failure

1) #1 Still

a) Shut off the gas to the burners.
b) Suck the coil.
c) Close a manual block valve in the feed line.

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H. #1 & 2 STILLS

7. Shutdown Instructions

d. Electrical Failure

1) #1 Still (cont'd.)

When the electrical power is restored, start up as follows:

a) Start circulating through the furnace.
b) Check vacuum for 40 mm.
c) Check the temperature on coil-in and coil-out. Be sure they are together.
d) Put the fire back on the furnace.
e) Open the manual block valve in the feed line when the forward flow starts.

2) #2 Still

a) Shut off the gas to the burners.
b) Suck the coil.

When the electrical power is restored, start up as follows:

a) Start circulating through the furnace.
b) Check vacuum for 40 mm.
c) Check the temperature on coil-in and coil-out. Be sure they are together.
d) Put fire back on the furnace.

8. General Information

a) Apply and release vacuum slowly to still receivers to keep crude from flashing over and causing a color problem.

~~b) High amps (6) on #1 still is an indication of bad bearings on the pump.~~

c) After every run, heat and drain the scrubber.

d) When starting up a still, start out with 5 lbs. or less of gas pressure on the burners. When it is certain the material is circulating properly, the gas pressure can be increased.

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8. General Information (cont'd.)

e. Always suck the furnace coils immediately if the circulating pump kicks off and will not start or it is shut off. The heat in the furnace will bake the aroclor in the coil and it cannot be opened. It must be changed out if this happens.

f. If material in the still will not circulate, break the vacuum. The pump should pick up the liquid before the vacuum is down to 20 mm.

g. If the condenser freezes up, drain out the water and apply steam for 15-30 minutes.

h. The cyclone in the vapor line on #2 still has a sight glass under it. When the still is flowing, there should always be some liquid draining into it and about 1" or less showing in the bottom of it.

If the sight glass is full, the still is overcharged and some material should be pumped out before trying to run the still, or the material will expand and dark crude will run into the receiver. If this happens, the receiver will have to be flushed out 2 times with a 10% level to be sure it is clean. The dark material must go through the still again.

i. Pumpovers can be made to #2 still without shutting off the circulating pump.

j. Before dropping bottoms, lay teflon or cardboard on the floor in the cabinet. Montar will be much easier to clean up.

k. Wear a respirator when dropping bottoms to prevent breathing the aroclor fumes.

l. On occasion, put oil on the ram valve threads to keep them in good working order.

m. Always check the inside of the open top drums before dropping bottoms in them. If they contain any water, dump it out, or it will flash when the hot bottoms hit it. Also, check for holes.

n. #2 still circulating pump has a special packing. It must be pulled up very little at a time (like 1 side of a nut).

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8. General Information (cont'd.)

o. If there is any problem with temperatures or circulation of the material in the stills, always suck the coil and start circulating again to protect the coil (keep it from plugging).

p. If there is an extra large amount of material in the sight glass when starting up #2 still and there is no temperature rise or product in the forward flow, it is possibly due to the condenser being froze up. Shut off the furnace fires and steam out the water side of the condenser.

q. NEVER blow air into a charged still that has vacuum on it. Liquid will carry over and cause color problems.

r. If the vacuum raises to 45 mm, cut the furnace fires immediately until the problem is corrected.

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9. Critical Control Points

CONTROL POINTS	EXPECTED READING	DEVIATION LIMIT & EFFECT	CORRECTION
Vacuum on Still Pot	40 mm	35-55 mm. Lower vac. can cause carryover that would result in color problems. High vac. will raise the boiling point and could cause unstable product.	Low vac. - raise set point. High vac. - see Section 11 on troubleshooting for loss of vac.
Level	~~#1 still chart-20~~ #2 still chart-50	± 10%. Lower level could make the pump cavitate. At higher levels, entrainment (liquids) comes over with the vapors.	Low level - Check the feed to the still; if it is o.k., reduce the gas pressure on the furnace. High level - Check bypasses. Increase the
Temperature	a) ~~#1 furnace coil temp. difference between coil-in & coil-out should be 10°C.~~	a) ~~15°C max. Coil will plug if the circulation is not good.~~	a) ~~Shut off the fires and check the circulation, suck coil both ways and try again.~~
	b) #2 furnace coil temp. difference between coil-in & coil-out should be 8°C.	b) 10°C for normal circulation. 25°C max. when boiling down to drop bottoms.	b) If more than 10°C for normal circulation, shut off the fires, suck the coil & into the bottom of the still and try again. If more than 25° while boil ng down, release vacuum and open up on valve above the pump to increase the circ. rate.

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9. Critical Control Points (Cont'd.)

CONTROL POINTS	EXPECTED READING	DEVIATION LIMIT & EFFECT	CORRECTION
Temperature (cont'd.)	c) #2 furnace coil temp. between vapor and coil-out should not be over 45°C.	c) 45° spread max. Coil will plug easily above this spread.	c) Drop bottoms as usual.

10. Other Control Points

Amps	a) ~~#1 still 4.5 to 5.5 amps.~~	a) 6 amps max. High amps usually indicates pump failure.	a) Suck the coil both ways, check valve lineup and try pump again. Notify supervision.
	b) #2 still 18 to 19 amps.	b) 18-20 amps. Just one amp lower than 18 usually means the material is not circulating. Higher amps probably means pump failure.	b) Low amps - Release vac. at receiver and watch the pressure gauge in the circ. line. When the pressure increases to 40-50#, close the pressure release valve. Also check the packing. High amps - Notify supervision.
Pressure on #2 circ. line	40-50 lbs.	If there is any deviation, the material may not be circulating.	Check the other conditions (amps and temp. on coil-in & coil-out) to determine if there is a problem. If there is, correct for amps or temp.
Color of Crude in th orward Flow	10-20 APHA	20 max. Anything higher could be of specs. on color.	Material will have to be distilled again. N / supervision.

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11. Troubleshooting

SYMPTOM	PROBLEM	CAUSE	POSSIBLE SOLUTION
Temp. on coil-in & coil-out are more than 10° apart.	The material is not circulating.	Line or coil pluggage.	Shut fires off the furnace and suck the coil both ways. If line is open both ways, release vacuum until the material is circulating.
Loss of vacuum	Jet cannot hold 40 mm on still pot.	a) Leaks in system.	a) Find the leaks and have them repaired.
		b) Jet nozzle failure due to pluggage or wear.	b) Change jet. Show will repair nozzles.
		c) Automatic valve on jet for vacuum control is open.	c) Check the controller on manual and automatic. Call instrument man.
		d) Receiver level too high.	d) Pump out receiver.
		e) Condenser is froze up.	e) Apply steam to thaw out condenser.
		f) Receiver pump packing leaking.	f) Pull up on packing. Have repacked if necessary.
		g) Feed tank to the still is empty.	g) Hold the still down until feed is available.
		h) Vacuum line is plugged.	h) Blow or steam out.
		i) Scrubber	i) Drain

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11. Troubleshooting (Cont'd.)

SYMPTOM	PROBLEM	CAUSE	POSSIBLE SOLUTION
Level dropping in the Still Pot	The material is boiling off faster than it can feed into the still.	a) The feed line is plugged.	a) NEVER blow air into the still unless there is no vacuum on it. When possible, block back thru the line; if necessary, have the control valve removed.
		b) The feed line is froze up.	b) Check tracers & traps. Also check for bare spots where the insulation has been removed.
		c) The feed tank is empty.	c) Shut the still down until feed is available.
High Color on Finished Aroclor	Picking up contamination in the process.	Air leaking or being blow into the still pot.	Add 2 bags of lime, reduce rate until color clears. Notify supervision.

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