

Plt - Springfield
Dept - Opalon Suspension Resin

SDP 92-634.0

Title - Process Details For Opalon 610 And 630 Resins
Via The A-6 Process

634.00 SUMMARY

This process described herein is designed to produce Opalon 610 and 630 type resins using A-6 catalyst in 3700-gallon glass-lined reactors.

~~Summary of polymerization conditions is attached.~~

These resins are used in making Vyram bottle compounds in 85 bldg.

634.01 SUMMARY OF POLYMERIZATION CONDITIONS*

		610	630
A.	<u>Formulation and Measuring Technique</u>		
1.	VCM Meter	11,000 lbs.	11,000 lbs.
2.	Water - kettle charge Meter	1,850 gal.	1,850 gal.
	Water - B-2 charge Vol. & Meter	50 gal.	50 gal.
	Water - B-2 flush Meter	80 gal.	80 gal.
3.	B-2 Weigh	5 lbs. 10 oz.	5 lbs. 8 oz.
4.	C-1 Weigh	30 lbs.	34 lbs.
5.	TCE Weigh	185 lbs.	65 lbs.
6.	A-6 Weigh	2 lbs. 8 oz.	2 lbs. 8 oz.
7.	A-6 Solvent Weigh	7 lbs.	7 lbs.
8.	Antifoam Volume	Hallco CPH-35N	Hallco CPH-35N
		3 qts. max.	3 qts. max.
9.	Ionol Volume	100 gms.	
B.	<u>Water Temperature During Purge</u>	58 - 61° C.	58 - 61° C.
C.	<u>Vacuum Purge, Minutes @ Boil</u>	10	10
D.	<u>BATCH Temp. @ Catalyst Addition</u>	56 - 59° C.	55 - 58° C.
E.	<u>Reaction Temperature</u>	58° C.	57° C.
F.	<u>Maximum Heat Kick</u>		
1.	Full Cold Water	@ 1° above REACT. Temp. (Same)	@ 1° above REACT. Temp. (Same)
2.	Water Inj., 250 gal.	@ 2°	@ 2° (AS 610)
	250 gal. addition @ second	@ 2°	@ 2°
G.	<u>Final Vent</u>	50 psig or 20 min. after 75 psig	50 psig or 20 min. after 75 psig
H.	<u>Agitator Speed</u>	107-110 rpm.	107-110 rpm.
*	The exact batch formulation and reaction temperature for each kettle are specified daily by the Operating Supervisor, on a daily instruction card.		

634.02

634.02 - SUMMARY OF EMERGENCY PROCEDURE

630 and 610

~~660 and 650~~

1. Heat kick control at 1°C above reaction temp.
at 2°C " " "

at 63°C batch temp.

at 66°C " " and
appears it will not
turn over in the next
2°C

FCW
250 gal. water inj. twice if
necessary for a total of 500 gal.

Vent to gasholder and/or stack.

Add 1 gal. of styrene and continue
venting.
2. Note: Although the above procedure is based on batch temperatures, add
1 gal. of styrene at any point if the pressure reaches 140 psig and
is still rising..
3. Note: After catalyst injection, there will on occasion be as much as a 4°C
overshoot. This is not a heat kick and requires no action. However,
it should be watched carefully until it shows it will come back down.

RSV0028628

634.10 RAW MATERIALS

634.11 Description and Hazards of Raw Materials

The raw materials normally encountered in the polymerization area are listed below with a brief description of the hazards. A more complete definition is written in Section 1160, Safety.

1. TCE + high heat phosgene
 (open flame) (highly toxic)
2. TCE + caustic explosive mixture
 (dichloroacetylene)
- ~~3. L_2O_2 + glycerine combustible material~~
- ~~4. Calcium Hydroxide (Lime) Heat and decomposition of
 + L_2O L_2O + possible fire~~
- ~~5. PDR-1 Doesn't normally support combustion, will
 burn under application of heat and oxygen.~~
3. Caustic Causes extremely bad chemical burns,
 exothermic (gives off heat) when dis-
 solved in water.
4. VCM Extremely flammable and explosive
- ~~8. VAC Flammable liquid and vapors present explosion
 hazard--vaporization is slower than VCM.~~
5. DAM Not highly combustible.
- ~~10. Glycerine Will burn in presence of oxidizing agent.~~
- ~~11. CNS Not toxic or flammable.~~
16. TCE Not flammable--store in cool dry place.
17. L_2O_2 Extremely flammable--store in cool dry place.
8. B-1 & B-2 No fire but dust explosion hazard.
9. C-1 No fire or explosion hazard.

634.10 RAW MATERIALS (Cont'd.)

634.11 Description and Hazards of Raw Materials (Cont'd.)

16. ~~Calcium Hydroxide~~ ~~Not flammable, will irritate eyes, nose, etc.~~
17. ~~Pytol~~ ~~Slightly flammable~~
18. ~~Nopeco NVZ~~ ~~No fire, slipping hazard.~~
19. ~~Sterox 110~~ ~~No fire, slipping hazard.~~
20. Halloo CPH-35N No fire, slipping hazard,
21. Tetrahydrofuran-Highly flammable and explosive, irritates (Solvent) eyes, nose, etc. Must be routinely inhibited to prevent formation of peroxides.
22. A-6 Catalyst Unstable material, decomposes violently at temperatures above 5-8°C.
23. DEM (A-6 Solvent) Slightly flammable; irritating odor; can cause irritation to skin.
24. A-6 Solution Unstable material; decomposes violently at temperatures above 40°C; slightly flammable; irritating odor; can cause irritation to skin.

634.12 Raw material Charge Measurement

A polymerization log sheet, Form MS-⁵⁸⁵⁰~~2444~~, is to be completely filled out with all readings recorded. A sample of the form is attached.

Summary (#1, #2, #3 Tanks)

Water, Kettle Charge	1850 gal.
B-2 Charge	50 gal.
B-2 Flush	80 gal.

639.10 RAW MATERIALS (Cont'd.)

639.12 Raw Material Charge Measurement (Cont'd.)

639.121 Vinyl Chloride Monomer Charge Quantity

Due to density changes with temperature, it is necessary to correct the VCM volume charge for VCM temperature as shown in the following table.

<u>VCM Temp., °C</u>	<u>VCM Vol., Gal.</u>
0	1391
1	1393
2	1396
3	1399
4	1401
5	1404
6	1406
7	1409
8	1412
9	1415
10	1418
11	1420
12	1423
13	1426
14	1429
15	1432
16	1434
17	1437
18	1440
19	1443
20	1445
21	1448
22	1451
23	1454
24	1457
25	1459
26	1462
27	1465
28	1468
29	1471
30	1474
31	1477
32	1480
33	1483
34	1486
35	1489

634.10 RAW MATERIALS (Cont'd.)

634.12 Raw Material Charge Measurement (Cont'd.)

634.122 Vinyl Chloride Meter Calibration

Accurate VCM charging is essential. Hence, VCM meters are calibrated on a weekly basis, ~~using the procedures defined in Section 620.30.~~

634.123 Process Water Charge Quantities

Process water is divided between (a) the initial kettle water charge and (b) the water used for dissolving and flushing B-2.

(a) Kettle Charge

The table below summarizes the dipstick vs. volume charges for 88 Bldg. and 92 Bldg. kettles.

<u>Dipstick</u> <u>inches</u>	<u>81-86 Kettles</u> <u>gallons</u>	<u>87 Kettle</u> <u>gallons</u>	<u>92 Bldg. Kettles</u> <u>gallons</u>
70	2025		1785
71	1995		1756
72	1965	2025	1727
73	1935	1995	1698
74	1905	1965	1669
75	1875	1935	1640
76	1844	1905	1609
77	1813	1875	1578
78	1782	1844	1547
79	1751	1813	1516
80	1720	1782	1485
81	1689	1751	1454
82	1658	1720	1423
83	1627	1689	
84		1658	
85		1627	

(b) Water Charge - 1st Floor Premix Tank

A total of 130 gallons of water are charged with the B-2 to the reactor. The first 50 gallons is used to dissolve the B-2 while an additional 80 gallons (metered) is used to flush the B-2 into the reactor.

8.

634.124 B-2 Charge

The exact amount of B-2 is weighed into a container and the net weight is printed on the back of the batch log sheet. Since B-2 absorbs moisture from the atmosphere, it is important that the surplus supply of B-2 be kept in polyethylene lined drums and the drums be covered at all times when not in use. Use only the specified lot of material.

634.125 C-1 Charge

The exact amount of C-1 is weighed into a container and the net weight is printed on the back of the batch log sheet. The C-1 viscosity increases rapidly as the temperature drops and, therefore, it is necessary to keep all C-1 55-gallon drums in a warm storage area. Be sure to use only the specified lot of C-1.

634.126 TCE Charge

The TCE weight tank system is used to accurately charge the specified amount of TCE. The weight tank is filled, and the red pointer set at the weight to be delivered. The starting weight (tare weight) and discharge weight are printed on the back of the log sheet.

634.10 RAW MATERIALS (Cont'd.)

634.12 Raw Material Charge Measurement (Cont'd.)

634.127 DEM Charge

The exact amount of DEM is weighed directly into the A-6 dissolving tank. Prior to the weighing, the scale poise should be moved until the empty tank is tared to zero scale reading.

634.128 A-6 Charge

The exact amount of A-6 is transferred directly from the 10# trays into the DEM in the dissolving tank. During this operation it will be necessary to have the 10# trays in contact with dry ice. With an ice pick the cake of A-6 can be gently broken up into small pieces and these can be transferred with a plastic scoop. The scale reading will indicate both the DEM and A-6 weight.

634.13 Raw Material Preparation

634.131 Dissolving and Filtering B-2 in 1st Floor Premix Tanks

At least two hours before kettle is to be charged, start the B-2 makeup. As B-2 solution can be held indefinitely, prepare the next batch as soon as the tank is empty.

- a. Weigh the specified quantity of B-2 into a container and position near the tank. Record lot number on log sheet.
- b. Add approximately 20 gallons 2 to 3 inches above bottom impeller of hot water--65 to 80°C on tank thermometer.
- c. Start tank agitator.
- d. Slowly add the B-2 to the hot water over a 1-2 minute period.
- e. After the B-2 is thoroughly wetted, 1-2 minutes, add cold water until tank is half full. Temperature should be below 30°C.

634.10 RAW MATERIALS (Cont'd.)

634.13 Raw Material Preparation (Cont'd.)

634.131 Dissolving and Filtering B-2 in 1st Floor Premix Tanks (Cont'd.)

- f. Record time and solution temperature on log sheet.
- g. Allow B-2 to mix and dissolve for two hours before filtering. After two hours the agitator can be shut off to avoid spillage.

634.132 Preparation of A-6 Solution

- a. A-6 solution can be kept indefinitely under refrigeration in the kettle hold tanks. However, for simplicity of control in case of an emergency keep A-6 solution inventory in hold tanks down to 4 batches.

634.10 RAW MATERIALS (Cont'd.)

634.13 Raw Material Preparation (Cont'd.)

634.132 Preparation of A-6 Solution (Cont'd.)

- b. Operator must wear gloves and goggles (or face shield) in the dissolving room
- c. At least once per shift, add sufficient dry ice to all Transport Boxes containing A-6.
- d. Place the dissolving tank on the scale and adjust poise to zero scale reading.
- e. Turn DEM spigot over tank and carefully add 7 lbs. DEM.
- f. Check the temperature of the DEM. Add sufficient dry ice, if necessary, to lower the temperature to 15-20°C (60-70°F).
- g. Tare scale again; check scale accuracy once per shift by using the 5 lb. weight.
- h. Remove a tray of A-6 from Transport Box and place in the refrigerated holder.
- i. Remove cover from A-6 tray and gently break A-6 into small pieces using an ice pick.
- j. With a scoop, transfer the A-6 into the dissolving tank. Add the specified amount.
- k. Replace cover on A-6 tray and return to the Transport Box. A-6 solid should not be left outside of the Transport Box for more than 5 minutes.
- l. Carefully move dissolving tank to the agitator station.
- m. Slowly lower agitator into tank by steadying the agitator with the left hand and slowly turning the 2-way valve handle.
- n. Start agitator. Small needle valve on motor base controls RPM; use slowest speed.
- o. Dissolve A-6 for 8 minutes. The solution temperature will drop quickly as the A-6 dissolves. Follow the temperature closely.
 - (a) At the start the temperature should be 15-20°C (60-70°F).

634.10 RAW MATERIALS (Cont'd.)

634.13 Raw Material Preparation (Cont'd.)

634.132 Preparation of A-6 Solution (Cont'd.)

(b) After 3 minutes of agitation, the temperature should be less than 5°C (40°F).

(c) After 8 minutes of agitation, the temperature should be less than -6°C (20°F).

If solution temperature goes higher, stop agitation and add sufficient dry ice to the material to cool it.

p. After dissolving period, shut off main air valve to stop agitator. Slowly raise agitator steadying the system with the left hand while turning 2-way valve with the right hand.

q. Close cover of tank and immediately transport this material to the desired kettle hold tank--never delay.

r. At no time leave the dissolving room area until all is completed.

634.133 Emergency Procedure in Dissolving Room

a. If you are in need of dry ice to restore inventory level--contact Foreman immediately.

b. Never place an object on top of Transport Boxes; CO₂ gases must be allowed to vent.

c. If DEM spills occur:

Large spills should be soaked up with Vermiculite and swept up. Place in the disposal can. Wash down area with water.

Small spills should be washed down with water.

d. If solid A-6 spills occur:

Spills should be swept up with Vermiculite and placed in a flat open tray. The sweeps must not be thrown into a drum or with other trash. Remove the sweeps to the area north of 92 Bldg. ~~Switch Room~~ outside of the building spread on the ground. Notify the foreman.

→ catalyst disposal area.

634.10 RAW MATERIALS (Cont'd.)

634.13 Raw Material Preparation (Cont'd.)

634.133 Emergency Procedure in Dissolving Room (Cont'd.)

- e. If A-6/DEM spills occur:

Large spill should be soaked up with Vermiculite. Do not allow pools to exist. Handle the same way as large spills of A-6; that is, remove sweeping to the ~~outside catalyst disposal area~~ ^{catalyst} the building and have transferred to the dump. Small spills should be washed down with water.

~~All equipment handling A-6 solution must be decontaminated with water wash and potassium hydroxide solution before removed from the area for maintenance. Handling of potassium hydroxide solution must be supervised. Rubber gloves and goggles are necessary to handle this caustic. See Section 633.80.~~

634.134 Transfer of A-6 Solution

- a. The A-6 solution must be transferred to the refrigerated charging tank immediately after completion of the dissolving operation.
- b. Preparation for the transfer:
- (a) Position dissolving tank in support ring, but do not open drain valves.
 - (b) Loosen cover on the transfer tank.
 - (c) Be sure 2" vent valve is open on the kettle hold tank.
 - (d) Drain any liquid from the kettle hold tank into a pail containing some dry ice. If more than a few drops, drain, remove to disposal area after completing the transfer.
 - (e) Check kettle hold tank charge valve and drain valve.
 - (f) Check "A-6 Solution" temperature gauge--should be below 8°C.
 - (g) Refrigeration must be operating and "brine temperature out" must be below 2°C.

634.10 RAW MATERIALS (Cont'd.)

634.13 Raw Material Preparation (Cont'd.)

634.134 Transfer of A-6 Solution (Cont'd.)

- c. Transfer the solution by making up the quick-coupling and then opening the (a) kettle hold tank transfer valve first, and (b) then opening the dissolving tank drain valve.
- d. When the A-6 solution is completely drained, first close the dissolving tank drain and then the kettle hold tank valve. A-6 solution is now ready for the charging operation.

634.135 Emergency Procedure

If any abnormal condition, such as listed below, arises drain the solution into a pail containing dry ice. Dispose of this material by spreading Vermiculite ~~in the ground~~ *cata/4,5* ~~outside of the Switch Room~~ and pouring the solution on top. In this manner, gradual decomposition will occur.

Abnormal Conditions

- a. Equipment maintenance is required.
- b. Solution temperature rises either due to decomposition or lack of refrigeration.
 - (1) Normal A-6 Solution Temp. 0 to 5°C
 - (2) Warning Temperature 10°C
 - (3) Drain A-6 Solution 15°C
 - (4) Allow A-6 Solution to decompose in hold tank with 2" vent valve open 30°C or higher

634.14 Charging Water, B-2, C-1,

IT IS NECESSARY TO USE WATER CHARGES AS FOLLOWS:

634.10 RAW MATERIALS (Cont'd.)

634.14 Charging Water, B-2, C-1, (Cont'd.)

Use #1, #2,
#3 B-2 Tank

Process Water Charge	1850 gal.
B-2 Make-Up Water.	50 gal.
B-2 Water Flush	80 gal.
Total	1980 gal.

1. The instrument settings to be used during the water charge are as follows:

Kettle Batch Temp. Recorder and Controller (BTRC)
..... on Manual, set point @ reaction temp.

Jacket Temp. Recorder and Controller (JTRC)
..... on Automatic, set point @ 62°C.

Charge Water Temperature Controller
..... Set point at 58°C.

2. When the kettle is ready for polymerization (kettle has been spaced, VCM is available, etc.) start addition of process water charge.

- a. At the start of the water charge--after the T on bottom of kettle has been filled with water--close the diaphragm valve.

- b. Check pipe downstream of the Durco plug valve for indication of leakage. If pipe is hot, and both valves shut, stop charging sequence and have valves repaired before proceeding.

2. When the water charge is started, charge B-2 solution.

- a. Be sure all sewer valves are closed.

- b. Line up valve from tank to pump through filter and into the kettle.

- c. Start pump and drain tank completely.

634.10 RAW MATERIALS (Cont'd.)

634.14 Charging Water, B-2, C-1 (Cont'd.)

- 3 When using 1st floor pre-mix tanks:
 - a. When ready to filter B-2 pressurize system between B-2 tank bottom valve and kettle valve with water to 30 psig to be sure of no leaks in the system.
 - b. Pump B-2 solution to kettle.
 - c. Add 80 gallons (metered) of cold water to the tank to flush out the system. Pump out flush water while adding to tank.
- 4 During the process water addition and the B-2 addition charge the C-1 to the reactor via the manhole.
- 5 Before closing manhole cover, check the catalyst water flush. Open the rotameter valve wide to be sure line is clear and then adjust flow to 9-10 gph. If line is not clear, repair before proceeding any further. The water flush is to be kept on at all times (except during kettle entry for cleaning.)

Dipstick batch level and
- 6 As soon as the B-2, and C-1 have been added, close and dog down manhole cover.
- 7 As soon as the manhole cover is secured, start vacuum purge operation.

634.15 Vacuum Purging

1. Check to be sure all kettle valves are closed, ~~except~~
~~water charge valve if still being~~

634.10 RAW MATERIALS (Cont'd.)

634.15 Vacuum Purging (Cont'd.)

2. Determine and record maximum vacuum controller level.
 - a. Do not proceed unless water is flowing to barometric condenser, and
 - b. the system vacuum is below 3.5 inches absolute.
3. Open vacuum valve on kettle.
4. After adding B-2 water and process water, the batch temperature should be in the 54-61°C range and approaching a constant temperature.
 - a. If it is necessary to adjust batch temperature to the desired range, hold up vacuum on kettle until temperature is within range.
5. Maintain a 5-minute vacuum boil.
 - a. If the water is boiling at the completion of the water charges, close water valve and purge for an additional 5 minutes.
 - b. If the water is not yet boiling at the completion of the water charges, close water valve. Continue to purge until a boil is reached, and hold vacuum for an additional 5 minutes.
6. The batch water is boiling when the vacuum controller is reading less than the theoretical boiling pressure listed below, and the actual pressure is not dropping faster than 0.2 inches per minute.

<u>Batch Temp.</u>	<u>Theoretical Boiling Pressure</u>
53.0°C	4.22 inches of Mercury
53.5	4.32
54.0	4.43
54.5	4.52
55.0	4.61
55.5	4.73
56.0	4.86
56.5	4.98
57.0	5.10
57.5	5.20

634.10 RAW MATERIALS (Cont'd.)634.15 Vacuum Purging (Cont'd.)

<u>Batch Temp. (Cont'd.)</u>	<u>Theoretical Boiling Pressure (Cont'd.)</u>
58.0°C	5.40 inches of Mercury
58.5	5.50
59.0	5.60
59.5	5.75
60.0	5.90
60.5	6.00
61.0	6.15
61.5	6.30
62.0	6.45

7. Notes

a. ~~Accurate water charging is very important. Therefore, always charge water with the man hole open and dipstick the water ~~the~~ charge carefully.~~

~~a. Accurate water charging is important and therefore two water meters are used in series when the meters are more than 50 gallons apart at end of water charge:~~

- ~~(1) Shut off vacuum.~~
- ~~(2) Break vacuum through water charge line.~~
- ~~(3) Open manhole cover and dipstick.~~
- ~~(4) For undercharge of water, add proper amount.~~
- ~~(5) For overcharge of more than 3 inches on dipstick drain kettle contents to sewer and recharge.~~

~~b. If only one water meter is operable, dipstick kettle above water charges before proceeding to close and purge kettle.~~

~~c. ~~Close~~ vacuum purge valve.~~

634.16 Charging TCE

1. At the end of the vacuum purge, charge the specified amount of TCE from the weigh tank.
2. Print the discharge weight on the log sheet.

634.10. RAW MATERIALS (Cont'd.)

634.17 Charging VCM

1. After completion of the vacuum purge and TCE charge, charge all the VCM to the kettle. ~~Do not use any recovered VCM.~~
2. As the VCM is added, the batch temperature will drop. Try to keep batch temperature as close as possible to reaction temp.
 - a. BTRC is maintained on manual control.
 - b. JTRC is maintained on automatic control.
3. Make adjustments as necessary to jacket temperature set point to attain 56-59°C. batch temperature by the end of the VCM add; adjust BTRC set point to reaction temperature.
4. During the VCM charge, read the VCM thermometer and determine the proper gallonage figure from the chart.
5. When the VCM charge is completed, close the valve at the VCM meter first, then the valve at the kettle to allow the liquid in the vertical line at the kettle to drain. This will lessen pressure buildup problem caused by thermal expansion of VCM.

634.18 Charging of A-6 Solution

1. After the VCM charge is completed and the reaction temperature is between 56-59°C., prepare the A-6 solution charging.
2. At the kettle panel, open the high pressure flush water valve to 9-10 gph rotameter setting. (Note that catalyst flush water must be flowing before starting the VCM charge.)
3. Check to be sure the water flowing through the catalyst charge line is cool to the touch. Do not charge if water is hot.

634.10 RAW MATERIALS (Cont'd.)

634.18 Charging of A-6 Solution (Cont'd.)

4. Check to be sure the A-6 solution temperature is below 10°C.
5. Check the kettle hold tank drain valve--must be closed.
6. Close transfer valve to charge tank.
7. Drop the Reactor Jacket Water Setpoint to 50°C.
8. Close 1/4" needle valve on control panel--finger tight.
9. On A-6 Instrument panel push "Close Button" which automatically closes the contromatic vent line valve. (Valves are equipped with a timer that re-opens valve at the end of an 8-minute period. If A-6 charge time is extended beyond 8 minutes, contromatic vent valve can be reset for another 8-minute period by pushing "close" button a second time.)
10. Slowly open 1/4" nitrogen needle valve and pressurize tank to 325-350 psig. Then, close needle valve.
11. When jacket temperature has dropped below 55°C., start A-6 solution addition.
12. Slowly open 1/2" plug charge valve below tank. As the solution flows from the tank to the reactor the pressure in the tank will drop. Adjust charge valve so that pressure drop from 325/350 to 150 psig. takes 1 minute. This is necessary to permit complete drainage of the tank.
13. When the tank pressure reaches 150 psig., close the charge valve. Repressurize to 325 psig. and slowly charge to kettle again.
14. When the tank pressure reaches 150 psig., close the charge valve and de-pressurize the charge tank by opening the 1/4" needle vent valve at the control panel.
15. When hold tank pressure drops below 5 psig., open the 2" vent valve by pushing the "open button."
16. Place a pail of Vermiculite beneath the charge tank drain valve and open valve. Report to supervision if more than a few drops of liquid drain from the tank. *and dump the contents immediately into the catalyst disposal area*
NOTE: Gloves and goggles must be worn during this operation.
17. After the 1-minute high water flush period, adjust the flow rate to 6 gph and maintain. Do not shut off unless kettle goes down for cleaning.

STANDARD PROCEDURE.

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Department

Sh is Page21.....

TITLE633.0 PROCESS DETAILS FOR OPALON 650 AND 660 RESINS VIA
THE A-6 PROCESS.....634.20 REACTION TEMPERATURE CONTROL634.21 Heat-up Control1. Instrument Settings

BTRC	on Manual
Proportional Band	18-20
Reset	10
Derivative	10
Set point	at reaction temp.
JTRC	on Automatic
Proportional Band	10
% Pneumatic	50

2. Between 0-10 minutes after the A-6 catalyst solution has been added to the kettle, switch BTRC to automatic control.
3. Normally the settings above will automatically control the batch temperature. However, in the evidence of a batch temperature over-shoot or cycling proceed as follows:

Batch Over-ShootAction

55-57°C
58°C

None, normal over-shoot
Switch BTRC to manual and drop jacket set to zero. Put FCW on jacket.

59°C

Inject 200 gallons of water

~~60°C~~~~Vent to gasholder~~~~61°C~~

Vent to gasholder and/or stack

~~62°C~~

Inject 1 gallon styrene

After batch has turned over and started to return to reaction temperature, level off system under normal control. Switch to automatic control when reaction temperature is under good control, $\pm 1^\circ\text{C}$.

Any batch that must be ~~vented to the gasholder or stack~~ ^{STYRENE INJECTED OR GOES ABOVE 68°C} in order to control reaction temperature will be down-graded to Technical Code - 6.

ISSUED 3-26-65
STANDARDS DEPARTMENT

BY *A.O. Ericksberg*

STANDARD PROCEDURE:

Plant SpringfieldNumber SDP 88-633.0Department Sh ts 22 Page 22TITLE 633.0 PROCESS DETAILS FOR OPALON 650 AND 660 RESINS VIA THE A-6 PROCESS633.20 REACTION TEMPERATURE CONTROL (Cont'd.)633.22 Heat Kick ControlHeat Kick Control

AT 12° Above Reaction Temp. F C W

AT 2° " " " 250 GALL. WATER INJECTION. Twice if NECESSARY FOR A TOTAL OF 500 GALL.

AT 63° Batch Temp. Vent To recubder and/or stack.

AT 66° Batch Temp. and

APPEARS IT WILL NOT TURN

OVER IN THE NEXT 2° Add 1 GALL. OF STYRENE and continue VENTING.

N.T.C.: Although The above procedure is based on batch Temperature Add 1 GALL. OF STYRENE AT ANY POINT IF THE BATCH PRESSURE REACHES 140 PSIG. and is still rising.

N.T.C.: AFTER CATALYST INJECTION, THERE WILL ON OCCASION BE AS MUCH A 4° OVERSHOOT. THIS IS NOT A heat kick and REQUIRES NO ACTION. HOWEVER IT SHOULD BE WATCHED CAREFULLY UNTILL IT SHOWS IT WILL come back down.

ISSUED 3-26-65
STANDARDS DEPARTMENT

BY A.O. Erickson

634.30 FINAL VENT

1. Start the final vent at 50 psig or 20 minutes after batch pressure drops below 75 psig, whichever comes first.
2. Prior to starting the final vent, add 3 quarts of Hallico anti-foam to the anti-foam bomb, AND START THE VENT SCRUBBER SYSTEM. (See SDP 438.4)
3. Pressurize the anti-foam bomb to 75-80 psig with N_2 . Close nitrogen valve.
4. One minute before starting the final vent, open anti-foam valve to kettle for continuous addition during the vent.
5. If foaming occurs, slow down the rate of venting.
6. After the final vent is complete, drop jacket temperature water to $50^{\circ}C$.

634.40 EMERGENCY ACTION

If for any specific reason there is danger of a set-up batch, add one gallon of styrene monomer to the kettle. Specifically, styrene injection should be employed for the following conditions:

1. Power Failure

See Section 637.0 Emergency Procedure; (add 1/2 gallon of styrene monomer to each batch.)

2. Water Failure

Same as above.

3. Mechanical Failure of Equipment Causing Loss of Agitation

If for any reason (such as seal leak, spark, generation, motor overload, excessive vibration at both high and low speed agitation, reducer failure, belt breakage) it becomes necessary to stop agitation immediately, add 1/2 gallon of styrene to the batch.

If repairs cannot be effected in 30 minutes, vent off remaining VCM with batch temperature reduced to 35-50°C. If repairs can be completed, start up agitation (by jogging agitator on-off) and complete batch polymerization.

4. Mechanical Failure of Equipment Causing Partial or Complete Loss of Cooling Water.

Basically, the emergency is very similar to "Water Failure Emergency Procedure--Section 637.0". Add 1/2 gallon of styrene if necessary to maintain batch temperature below 70°C and/or batch pressure below 135 psi.

5. Suspending Agent Omission or Failure

If the B-2 charge is accidentally omitted (or only partially charged) or if the B-2 suspending agent is destroyed by an impurity--such as caustic--batch set-up will occur. An early indication of this problem is a sudden drop in the jacket water temperature after only 3 to 4 hours of polymerization. A heat kick will follow immediately.

634.4C EMERGENCY ACTION

5. Suspending Agent Omission or Failure (Cont'd.)

With these symptoms, add 1/2 gallon of styrene to the batch.

Discretionary Action

If for some reason batch temperature and batch pressure show abnormal performance, contact kettle foreman. If he cannot be reached and it is your opinion that a set up batch may occur, add 1/2 gal. of styrene to short-stop the reaction.

STANDARD PROCEDURE.

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TITLE 633.0 PROCESS DETAILS FOR OPALON 650 AND 660 RESINS VIA
THE A-6 PROCESS

634.50 BATCH CLASSIFICATION

634.51 Reclassification of Batches in Kettle

Downgrade to Code 6 (Technical Grade) for the following reasons:

- (a) Batch is mischarged.
- (b) Agitator is off more than 15 minutes after A-6 charge has been started.
- (c) RVCN is used.
- (d) ~~Heat~~ ~~exceeds 640°~~ STYRENE is Added OR Temp. exceeds 682
- (e) Particle size is obviously too coarse.
- (f) Excessive temperature cycling occurs.

Cycle time will generally vary on batches using RVCN. If cycle time, as measured from start of catalyst to start of vent, is 5.0 hrs. or less on any batch using RVCN, notify Supervision for segregating instructions before dumping. Also, hold for dumping instructions if there are any other abnormalities such as poor temp. control, high heat back, etc.

634.52 Segregation of Batches

1. Complete wash-out of lines and slurry tanks is required between all product changes, i.e., going from ~~650-1~~ to ~~660-1~~ and vice versa. 630-1
2. No clean-out is necessary between product changes of Technical Grade material.
3. Code 1 and Code 6 are kept completely segregated. If it becomes necessary to dump a Code 6 batch into a tank that has not been washed out, then the entire contents are immediately downgraded to Technical Grade.

The tank is maintained as a Technical Grade--regardless of the number of Code 1 batches dumped--until the tank is washed out

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BY A.A. Erickson

639.60 DUMPING OF BATCHES

1. Take required samples from each batch before dumping.
2. Dump slurry in tank with adequate space for batch-- avoid spillage!
3. When kettle has been dumped, lower the jacket temperature ~~set~~ point to zero so that kettle can be inspected (without steam vapors) during subsequent flushing and inspection operation.
4. Flush dump lines to and from the pumps for 2-3 minutes with water to prevent solids settling and plug-ups.

~~At the end of the water flush, the pump is shut off and the kettle dump valves are lined up to the sewer (in preparation for the Seller's flushing.)~~

5. If normal dumping is not possible (i.e., plugged dump hole) use "Siphon Unloading Technique". (Section 639.70)

639.70 Kettle Flushing

6. Fill kettle with water via the Full Kettle Water Flush system. Be sure manhole is closed with 4 dose. The Kettle is Full when a pressure spike is observed on the batch chart.
7. Agitate for 10 minutes
8. If no other kettle on the common dump line is dumping, proceed to dump the flush water to the sewer.
9. For a detailed description of the Full Kettle Flush system, see S.O.P. 639.1.

STANDARD PROCEDURE.

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TITLE 633.0 PROCESS DETAILS FOR OPALON 650 AND 660 RESINS VIA
THE A-6 PROCESS

634.70 KETTLE FLUSHING

1. Considerable attention must be given to cleaning out all loose adhering polymer before proceeding with the water charging of the next batch. Residual polymer will result in high, out-of-spec fisheye level.
2. While the batch is being dumped, gently wash down kettle walls with low pressure water.
3. After batch is dumped, close kettle drain valve.
4. Simultaneously ~~add 1000 gallons of cold water to kettle~~ *fill kettle with water via the full kettle flush system. Be sure manhole is plugged. The kettle is full when a "pressure spike" is observed on the chart.*
5. ~~When 1000 gallons of water have been added, shut off water and start agitator.~~ *Agitate for 10 minutes*
6. ~~Agitate for 2-3 minutes to clean tripped of loose resin.~~ *Check to see if other kettles are dumping, then shut off agitator and insert Sellers.*
7. ~~At the same time, if not dump the kettle.~~
8. ~~Start Sellers and immediately open kettle drain valve.~~
9. ~~Flush kettle with 600 gallons of water through the Sellers.~~

NOTE: Since 84 Kettle is difficult to drain because of raised lip on drain nozzle, charge RVCM to all batches in 84 kettle.

NOTE: After a kettle has been manually cleaned, Sellers flush to remove all loose polymer.

7. For a detailed description of the full kettle flush system, see SDR 639.1

Reference: W.L. Thomasine, 4/15/71.

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BY A.O. Enckberg

RSV0028653