

CHEMICAL RESOURCES DIVISION
CHEMOLITE PLANT

Operating Standard

Date of Issue: August 14, 1973
Superseding : April 2, 1973

CODE NO: FM-3256 Perfluorooctanoic Acid Distillation

MFG FOR: FM-3175, FM-3395

REASON FOR CHANGE: 1. Update the previous standard.
2. Specify used acrylic acid drums in Steps 16 & 18.

REFERENCE: Operating Standard Dated April 2, 1973.

EQUIPMENT: BC #33, K-600

CHARGE:

<u>Code</u>	<u>Description</u>	<u>BC #33</u>
A. FM-3754	Distilled FM-3206)	
A. FM-3206	Crude Perfluorooctanoic Acid)	
A. FM-3388	Fluorocarbon flush)	8500 lbs.
A. FM-3257	Intercut to C ₈ Acid)	
A. FM-3628	Buffer cut to C ₈ Acid)	
B. RM-3048	Sulfuric Acid	400.0
C. RM-3179	Filter Cel	(0.2% of total FM-3206)
D. FM-3160	Kettle Flush	600.0

ESTIMATED YIELD:

Main Product: FM-3256, approximately 14% of FM-3206 charge or 50% of FM-3754 charge.

By-Product: FM-3160, FM-3210, FM-3257, FM-3388, FM-3628

MATERIAL SPECIFICATIONS:

1. FM-3206 should be as free of water as possible. Decant before charging, if necessary.
2. FM-3754, FM-3257, or FM-3628 may contain white or grey solid material at room temperature. Be sure to charge all solid material to kettle.

Exhibit
2493

State of Minnesota v. 3M Co.,
Court File No. 27-CV-10-28862

SAFETY PRECAUTIONS:

- FM-3754,
FM-3388,
FM-3210, PERFLUOROCTANOIC ACID. A very corrosive acid. White solid in
FM-3257, high concentration, or liquid when diluted with inerts. Avoid
FM-3628, skin contact. Wear full length rubber gloves and a face shield
FM-3206 when handling.
- RM-3048 SULFURIC ACID. An oily liquid which is very corrosive acid.
Avoid skin contact. Wear full-length rubber gloves and a face
shield when handling. Know the location of the nearest safety
shower. Avoid contaminating this material with water or any
combustible substance.
- RM-3179 FILTER CEL. It is a powder which reacts with any HF to form
 SiF_4 gas. Avoid inhalation or contact with these vapors since
they will form HF (acid) in the presence of moisture.

Refer also to Chemical and Film Division Safety Information sheets applying
to any of the above materials.

OPERATING PROCEDURE:

I. PRE-RUN PREPARATION

1. Kettle, receiver and blowcase must be clean and dry. Blank off both
sight glasses or line with Kel-F sheet. The FM-3206 contains HF which
disintegrates glass.
2. Cut in the packed column and blank off the open column.
3. Pressure test the system and obtain a leak rate less than 2 psi per hour
at 40 psig.
4. Check out vacuum control system. It must be able to control at 20 mm
or less pressure before proceeding.
5. Valve to by-pass the decanter.

II. FRACTIONATION

6. (a) Turn on cooling water to kettle on receiver jacket.
(b) Turn on condenser water and set water exit control at 70°F.
7. Lower the kettle pressure to 200 mm through the receiver and vacuum Charge "A"
up to 8,500 lbs. with the FM-3754 being charged last. Do not reduce the
pressure below 200 mm while charging as an excessive loss of low boiling
fluorocarbons may result.

OPERATING PROCEDURE: (Continued)

8. Turn on agitator at speed #3. Vacuum Charge B, Sulfuric Acid. Turn off cooling water to kettle jacket.
9. Turn on 75 psi steam to kettle jacket. Set DP at 25 mm and set reflux timer setting at 2/4 (product take-off setting over reflux setting in seconds). Adjust DP setting as necessary to get specified take-off/reflux ratios.
10. Distill off FM-3160 (inerts) at the rate of about 1000 lbs./hr. to a kettle temperature of 275°F. Do not exceed 3000 lbs. net on the receiver.
11. Drain distillate to blue and yellow OT drums. Watch for a top phase of water while draining and discard any water found. Record weight of water discarded. Stencil drums "FM-3160, lot, net and drum number". To each drum of FM-3160 add about 1 ounce of RM-3177 or 2 ounces of RM-244. Do not sample FM-3160.
12. If more Charge A remains to be distilled, vacuum charge same to the blowcase, and charge to kettle in 1000 lb. increments as the FM-3160 is distilled off. Do not exceed 200 mm vacuum while charging the blowcase. Do not exceed 3500 lb. net in the blowcase.
13. When pot temperature reaches 275°F. and no more Charge A remains to be charged, cool pot to 150°F.
14. Open kettle manhole and add Charge C, filter cel. Close and tighten down manhole.
15.
 - (a) Set reflux timer at 2 sec product/8 sec reflux.
 - (b) Turn on 75 psig steam and set DP at 25 mm.
 - (c) Pull vacuum to 10 mm.
16. Distill precut (FM-3210) to receiver until the head temperature reaches 160°F., at 10 mm. Adjust DP setting as necessary to get specified take-off/reflux ratios. Drain distillate to clean used acrylic acid O/T drums. Stencil "FM-3210, lot, net, drum number". Take a 2 ounce sample to the QC Lab.
17.
 - (a) Turn on heat to trace heaters.
 - (b) Turn on 130°F. water to condenser and receiver jacket.
18. Distill intercut (FM-3257) to the receiver (2/8 timer setting) at full vacuum. Adjust DP setting as necessary to get specified take-off/reflux ratios. Drain each 600 pound accumulation to clean used acrylic acid O/T drums. Stencil "FM-3257, lot, net, drum number". Take a two-ounce sample from each drum to QC Lab.

OPERATING PROCEDURE: (Continued)

19. The FM-3257 cut is ended when head temperature reaches 192°F. at 10 mm. If pressure is not exactly 10 mm, end the FM-3257 cut when the head temperature levels off. If instrumentation is not dependable, the buffer cut (FM-3628) can be started when the ~~FM-3257~~ samples freeze at room temperature. The FM-3257 cut should not exceed 600-750 lbs.

NOTES: (1) When draining receiver, do so rapidly to avoid freeze-ups.
 (2) Feel temperature of reflux return line once per hour and note. This line must remain warm while in FM-3257 and FM-3256 cuts. Consult Foreman if it cools off.

20. (a) Increase condenser temperature to 140°F.
 (b) Distill a 300 pound buffer cut at the same 2/8 timer setting. Stencil "FM-3628, lot, net". Take a 2 ounce sample to QC Lab.

21. Distill main cut with the following reflux timer settings;

	<u>Setting</u>	<u>Approximate Take-Off Rate</u>
First 1800 lbs.	2/8	600 lbs./hr.
Balance of main cut	2/12	400 lbs./hr.

Adjust only DP to obtain approximate take-off rate.

This higher reflux ratio will concentrate high boiler in last drums provided the DP and vacuum systems are functioning normally. Do not accumulate more than 600 pounds in the receiver at one time. Before draining put the kettle on total reflux. Switch vacuum over to kettle only, and break the vacuum on the receiver with inert gas. Drain each 600 lbs. collected to 55-gallon Orange and Yellow stainless steel drum. Stencil "FM-3256, Drum No., Lot No., Net". Take a 2-oz. sample from each drum to QC Lab.

After sampling, very slowly pull vacuum on receiver until the system is equalized. Continue total reflux until head temperature and DP have reached equilibrium, then resume reflux as specified above.

22. When the take-off rate falls below 50 pounds per hour and the pot temperature rises to 300°F. at 10 to 20 mm vacuum, isolate the receiver and drain the last of the FM-3256 as outlined in Step #21.

OPERATING PROCEDURE: (Continued)

23.
 - (a) Return kettle system to full vacuum.
 - (b) Set timer to 2/2.
 - (c) Cool kettle.
 - (d) Vacuum charge "D", FM-3160 (use drum from this fractionation) directly into bottom of receiver. Transfer the FM-3160 to the kettle.

24. Distill about 1/2 of the FM-3160 charge to the receiver with the timer set at 2/2. Then distill the balance at total take-off. The distillation is finished when the take-off rate drops and the kettle temperature reaches 300°F. at 20 mm vacuum or better. Turn on cooling water to kettle and drain receiver to 55-gallon orange and yellow drums. Stencil "FM-3388, lot, net".

25. The bottoms may be drained to the sewer if the final conditions in Step 21 were not 290°F. pot temperature at 20 mm or better. Notify the Foreman if they were not.

SAMPLES, LABORATORY TESTS, RELEASE SPECIFICATIONS:

	<u>Step No.</u>	<u>Spl. Size</u>	<u>Sample Description</u>	<u>Send To</u>	<u>Test</u>	<u>OCM#</u>	<u>Spec.</u>
In-Proc.:	None						
Final:	16	2-oz.	FM-3210	QC Lab	GLC	77.1.25	%Acid Distribution %Fluorocarbon
	18	2-oz.	FM-3257	QC Lab	GLC	77.1.25	%Acid Distribution %Fluorocarbon
					%H ₂ O	4.1	Record
					%F	53.8	Record
	20	2-oz.	FM-3628	QC Lab	GLC	77.1.25	%Acid Distribution %Fluorocarbon
					%H ₂ O	4.1	5.0% maximum
					%F	53.8	0.03% maximum
	21	2-oz.	FM-3256	QC Lab	GLC	77.1.25	%Acid Distribution %Fluorocarbon
					%H ₂ O	4.1	5.0% maximum

Total QC Time: 13.6 Hours

Final Sample Retain Time: 1 Week

To Be Released By:

CONTAINERS AND STORAGE:

<u>Container Code</u>	<u>Container Description</u>	<u>Wt. Per Unit</u>	<u>Special Labels or Stencilling</u>	<u>Storage</u>
34-7000-4449-7	Blue & Yellow O/T	600	FM-3160, Lot#, Net	Field
100	Clean used) Acrylic acid) drums. O/T)	600 600	FM-3210, Lot#, Net) FM-3257, Lot#, Net)	Warehouse
34-7000-4450-5	Orange & Yellow SS	300 600 600	FM-3628) FM-3256) FM-3388)	Warehouse

POST-RUN EQUIPMENT CLEANUP:

1. Between Runs: None.
2. After Last Run: Boil out kettle and receiver with 500-gal. water and 512 pounds of RM-244 (caustic). Follow this with a water boil-out.

WASTE DISPOSAL:

<u>Step No.</u>	<u>Description</u>	<u>Container</u>	<u>Special Labels or Stencilling</u>	<u>Disposition</u>	<u>Approx. Amount</u>
25	Bottoms	--	--	Sewer	--

AUTHOR:

D.C. Termont Date Aug 14, 1973
D. C. Termont

APPROVALS:

G. L. Damerell Date 8-24-73
G. L. Damerell

D. R. Hallberg Date 8 Oct 73
D. R. Hallberg

D. J. Wardrop Date 10-10-73
D. J. Wardrop

A. R. Diesslin Date 11-11-73
A. R. Diesslin

C. G. Klaus Date 10-26-73
C. G. Klaus

C. W. Bentz Date 10-31-73
C. W. Bentz

R. C. Johnson Date 10 31-73
R. C. Johnson

DCT/mc

FACTORY COPY

CHEMICAL RESOURCES DIVISION
CHEMOLITE PLANT

Return to T. Shannon 41-1

Operating Standard

AT CHEMOLITE

Effective: March 26, 1974
Superseding: February 15, 1973

CODE NO: 41-2600-5643-1
SUBJECT: Dry Ammonium Perfluorooctanoate
MFG FOR: 41-2700-3632-4 (FM-3632) or 41-2700-3633-2 (FM-3633)
COMMODITY CLASS: 9419
REASON FOR CHANGE: 1. Include 11-digit codes
2. Specify F-4880 code to designate dirty "Ammonium Salts".
REFERENCE: 41-2700-3165-5 (FM-3165) Operating Standard dated February 15, 1973.
EQUIPMENT: Steam oven, BC #35, Dept. 3060 (Bldg. 15).

CHARGE:

<u>Code</u>	<u>Description</u>	<u>BC #</u>
41-2600-5654-8	A. Wet Ammonium Perfluorooctanoate	250-300

ESTIMATED YIELD: About 70% of F5654 charged.

ESTIMATED MACHINE TIME: 10 Hours.

RAW MATERIAL SPECIFICATIONS:

41-2700-3212-5 (FM-3212) Wet Ammonium Perfluorooctanoate. Check for proper appearance -- a wet, white salt.

SAFETY PRECAUTIONS:

41-2600-5654-8 WET AMMONIUM PERFLUOROOCCTANOATE. This salt is toxic. Wear rubber gloves when handling this material and avoid skin contact.

41-2600-5643-1 DRY AMMONIUM PERFLUOROOCCTANOATE. This salt is toxic. Inhalation of dust is irritating. Wear a respirator and rubber gloves when handling this material.

POST-RUN EQUIPMENT CLEANUP:

1. Between Runs: No cleaning necessary. Be sure oven area and powder room is kept clean.
2. After Last Run: Rinse oven trays thoroughly with water.

BY-PRODUCT DISPOSAL:

<u>Step No.</u>	<u>Description</u>	<u>Container</u>	<u>Special Labels or Stencilling</u>	<u>Disposition</u>
'Note' p.1; 4;6	Ammonium Perfluoro-octanoate which has become contaminated with foreign material, spillage, or floor sweepings	5-gal. pail lined with poly-bag	41-2600-4880-0, (F-4880), Net Wt.	Hold in Bldg 15 for rework in FM-3175 or F-1256

OPERATING PROCEDURE:

NOTE: Read this standard through carefully before starting. The fluorochemicals are very valuable. Avoid losses and contamination. If a spill occurs, sweep up the product and place it in a separate pail lined with poly bag tagged "F-4880". Report the spill to the Foreman because the product can be recovered. See "By-Product Disposal" section, above, for instructions on how to handle spilled material.

I. SPECIAL PRE-RUN CLEANUP FOR OVEN

No cleanup following FM 3165. RR

1. The oven must be absolutely clean for any series. Also, the dual oven air inlet filters must be replaced by clean filters. Check to be sure the room window air filter is clean. Oven cleanup must include the following:
 - a. Wash and scrub the special oven trays and oven rack. Use only the stainless steel trays marked "Use Only For FM-3165 or FM-3353".
 - b. Wash and scrub the inside of the oven, including floor, walls, and ceiling. When oven is clean, close the door and allow the oven and the oven trays to dry before charging.
 - c. Make sure all grinding and blending of other products in the powder room has stopped before any FS654 is charged to the oven. Great care must be taken to prevent the FS643 from being contaminated.

No cleaning will be necessary if any FM-3165 has preceded this run.

II. CHARGING AND DRYING

2. NOTE: Before charging FS654 to the trays, wet down the floor in the oven area to minimize dusting. Also, keep all doors and windows to the outside closed in powder room during charging and throughout drying process.

Spread the FS654 evenly on the trays, breaking up all lumps larger than one inch. Using a clean Fiberglass shovel, charge about 20 pounds of FS654 per tray. Keep lots of FS643 separate in the oven. Be sure to note on the charge card which set of trays (front or back) contain which batch of FS643.

March 26, 1974

OPERATING PROCEDURE: (Continued)

3. Place the trays in the oven and turn on heat and fans. Set temperature controller to 140°F.

WARNING: TEMPERATURES ABOVE 150°F. WILL DECOMPOSE THE PRODUCT.

4. NOTE: Using a water hose, wet down the process area before opening oven to stir contents of trays. Be sure all doors and windows are closed and oven fan is shut off before opening oven door.

Stir the contents of the trays every 2-3 hours with a clean plastic scoop or clean Teflon stirrer to speed up drying and minimize baking of large lumps. Record time and temperature on data sheet after trays have been stirred. After stirring trays, be sure to clean up any product spilled on the floor. Follow the procedure outlined in the "Note" on top of page 2 of this standard, and instructions specified in "By-Product Disposal" section.

5. DO NOT ALLOW PRODUCT TO REMAIN UNATTENDED IN THE OVEN OVER THE WEEKEND! Allow material to remain in oven until dry. This drying step should take only 8-16 hours if the trays are being stirred properly. The salt will appear powdery or dusty when it is dry.

III. DUMPING

6. NOTE: Using a water hose, wet down the process area before opening oven to dump trays. Be sure all doors and windows in powder room are closed and oven fan is shut off before opening oven door.

When material is dry, dump trays into a clean green and yellow 55-gallon openhead drum. Stencil with "41-2600-5643-1", Lot No., and Net Wt.". The net weight of dry salt in each drum is used in blending various lots and care should be taken in order to get the correct weight.

After dumping the trays, be sure to clean up any product spilled on the floor. Follow the procedure outlined in the "Note" on top of page 2 of this standard, and instructions specified in "By-Product Disposal" section.

7. Take a 4-ounce final sample from each lot. Send to Bldg. 41 QC Lab. Label "F5643 Lot No.".
8. Record all dumps and charges on the oven data sheet.

IV. CLEANING AFTER RUN

9. If more FM-3212 is to be charged on the trays, then no cleaning of the trays is necessary. If a material other than ammonium perfluorooctanoate is to be charged onto the trays, then wash the trays thoroughly with water. Do not use the special S.S. trays for anything except FM-3165, F5643 or FM-3353.

March 26, 1974

SAMPLES, LABORATORY TESTS, RELEASE SPECIFICATIONS:

	<u>Step No.</u>	<u>Spl. Size</u>	<u>Sample Description</u>	<u>Send To</u>	<u>Test</u>	<u>QCM#</u>	<u>Spec.</u>
In-Proc.:	None						
Final:	7	4-oz.	41-2600-5643-1	QC Lab	% Nitrogen	120.3	Record (3.22-3.50 desired)
					Free Fluoride	53.4	250 ppm max.
					Iron	90.4	20 ppm max.
					Ash	90.4	300 ppm max.
					GLC	100.0	Record
					pH (2% aqueous)	30.2	> 4

NOTE: All tests are run on the first batch of a lot. If this batch is passing, only "% nitrogen", "pH", and "GLC" are run on the following batches of the lot. If the first batch fails, all tests are run on succeeding batches.

Total QC Time: 4.0 Hours.

Final Sample Retain Time: 1 Year.

To Be Released By: Chemical Division Quality Control.

CONTAINERS AND STORAGE:

<u>Container Code</u>	<u>Container Description</u>	<u>Wt. Per Unit</u>	<u>Special Labels or Stencilling</u>	<u>Storage</u>
34-7000-4455-4	55-gallon green & yellow openhead drum	one lot of FS643 (about 150-220 lbs.)	41-2600-5643-1 (2600), Lot #, Net Wt.	Inside

March 26, 1974

AUTHOR:

E. Shannon
D.C. Termon
D.C. Termon

Date *3-26-74*

APPROVALS:

G.L. Damerell
G.L. Damerell

Date *3-26-74*

D.R. Hallberg
D.R. Hallberg

Date *18 April 74*

D.J. Wardrop
D.J. Wardrop

Date *25 April 1974*

A.R. Diesslin
A.R. Diesslin

Date *4-29-74*

C.G. Klaus
C.G. Klaus

Date *5-1-74*

C.W. Bentz
C.W. Bentz

Date *5-2-74*

Date _____
R.C. Johnson

D.E. Morin
D.E. Morin

Date *6/29/74*

DCT/mc

CHEMICAL RESOURCES DIVISION
CHEMOLITE PLANT

Factory Operating Standard

Effective Date: June 8, 1978
Superceding Date: March 5, 1978

CODE NUMBER: 41-2700-3633-2 (FM-3633)
SUBJECT: Pulverized, blended Ammonium Perfluorooctanoate for FC-143
COMMODITY CLASS: 9419
REASON FOR CHANGE: 1. Eliminate oven drying.
2. Eliminate use of blend drum by using ribbon blender in Dept. 3036.
3. Eliminate hand packaging by packaging directly from ribbon blender discharge.
REFERENCE: 1. FM-3633 Deviation, dated 5/10/78.
2. FM-3165, Lot 216, Deviation.
EQUIPMENT: BC-61, Dept. 3036, with Strong-Scott Pulverizer.

CHARGE:

<u>CODE</u>	<u>Safety Std.</u>	<u>Description</u>	<u>BC 61</u>
A. 41-2600-5695-1	--	Wet Ammonium Perfluorooctanoate	2000 lbs.
		Average Yield:	1400 lbs.
		Estimated Machine Time:	8 Hours

RAW MATERIAL SPECIFICATIONS:

41-2600-5695-1 (F-5695) Wet Ammonium Perfluorooctanoate. Must be released by QC Lab. Use only the amount and lot numbers specified by Process Engineer.

ADDITIONAL SAFETY PRECAUTIONS:

41-2600-5695-1 WET AMMONIUM PERFLUOROCTANOATE. These materials are toxic.
41-2700-3633-2 Inhalation of dust is irritating and must be avoided. Wear a dust mask or respirator and rubber gloves when handling this material. Wash suspected areas of contact with soap and water to avoid prolonged contact. Clean up any spills (see By-Product Disposal Section, this procedure) as soon as possible to avoid contamination of the area.

OPERATING PROCEDURE:

NOTE: Read this procedure carefully before starting. This products is a snow white dry powder which is very valuable. Avoid losses and contamination.

I. PRE-RUN PREPARATION

1. No other material that creates dust should be processed in Bldg. 3 grinding room when this FM-3633 is run; especially no other grinding operations being performed.
2. The ribbon blender must be spotlessly clean before starting this run. Scrape out any material remaining from the previous run, flush with water, scrub with scotchbrite (if needed) and dry. Clean or replace the ventilation hoses and change the dust filter before charging.
3. Inspect and repair cover gaskets.

NOTE: The doors to the area must be kept closed for the rest of this operation in order for the ventilation to work properly.

II. CHARGING AND DRYING

4. Wear a dust mask, rubber gloves, and disposable coveralls when charging F-5695. Charge 60-70% of Charge "A" (F-5695), close the cover (be sure the ventilation trunks are in place), set the mixing tee to 145°F. and start up the ribbon blender.
5. After 3 hours, shut off the ribbon blender. Allow the dust be settle for 20 minutes, then charge the remainder of Charge A. Wear protective equipment and keep dusting to a minimum. Close the cover and resume drying. Clean up all spills immediately. (See By-Product Disposal).
6. Continue to dry for 8 hours. Check the unit hourly to be sure the ribbon blender is running at 145°F.

OPERATING PROCEDURE: (continued)

7. Flush the drain valve by removing 5-10 gallons from the bottom. (Wear dust mask, rubber gloves, disposable coveralls and clean up any spillage. Return this material to the blender and continue to dry for 8 hours at 145°F. The pulverizer may be set up at this time.

III. PULVERIZING

8. Position a fiber drum with poly bag liner and dial scale below the pulverizer discharge chute. Zero scale with the carton in place, then check the scale for accuracy with a 50 lb. weight. Adjust the scale if needed.
 9. Attach the poly bag to the discharge chute.
- NOTE: Operator must wear dust mask, rubber gloves and disposable coveralls while pulverizing and packaging. See "Safety Precautions".
10. Inspect exhaust bag on pulverizer. Empty and clean bag if necessary to allow air to pulverizer. Save product dust from bag as F-4880 (by-product).
 11. Start up pulverizer as in the following order:
 - a. Turn on power switch.
 - b. Turn on the grinder wheel.
 - c. Turn on the screw feed.
 - d. Adjust the vari-drive to speed 4-5. Do Not adjust the vari-drive control unless the screw feed is running.
 - e. Turn on ribbon blender.
 - f. Open valve and ribbon blender.
 12. Collect 50 lbs. of product in the fiber carton, close valve on blender and shut down pulverizer by reversing steps of Step 11. Check the packaging frequently to avoid plugging the pulverizer.
 13. Allow dust above filled container to clear out. Adjust final net wt. to 50 lbs. by hand. Use the silk scale for final adjustments and keep this operation as near the enclosure under the ribbon blender as possible to take advantage of the ventilation. Clean up all spills. See By-Product Disposal.
 14. Tie poly bag shut and cover fiber drum and stack on a pallet (14 drums to a pallet). Tape and stencil the load "41-2700-3633-2, Lot No., Net Wt." Hold in the area for QC release and final labeling.
 15. Take four 8-ounce final samples after the first 700 lbs. is packaged.
 16. Repeat Steps 8-15 until BC 61 is empty.

OPERATING PROCEDURE: (continued)

IV. CLEANUP

17. No clean up is necessary between FM-3633 or FM-3632 lots.

NOTE: Operator will wear dust mask, rubber gloves and boots and disposable coveralls during clean-up.

18. After the last lot in a series, remove pulverizer from ribbon blender and cover with a poly bag.

19. Remove cover to ribbon blender and use a stick and brush to dislodge any solids sticking to the cover, sides or auger. Allow these solids to collect in the bottom of the blender.

20. Drain solids to a green and yellow openhead drum. Cover drum, stencil "41-2700-3633-2, Lot 264, Net Wt." record as output on the production report and send drum to warehouse.

21. Position a green and yellow openhead drum under the drain to BC 61. Use the diaphragm pump to pump one drum of 41-2600-6002-9, Lot 47 (in Bldg. 6 staging area) into BC 61. Allow this material to drain into the green and yellow drum. Recycle from the drum to the unit as needed to flush out solids.

22. Cover drum and stencil "41-2600-6002-9, Lot 264, Net Wt." and send to warehouse. Record F-6002 input and output on the production report.

23. Flush BC 61 floor and area with water and drain to sewer.

WASTE DISPOSAL: None

BY-PRODUCT DISPOSAL:

Step

<u>No.</u>	<u>DESCRIPTION</u>	<u>CONTAINER</u>	<u>STENCILLING</u>	<u>DISPOSITION</u>
3, 23	Dry ammonium perfluoro-octanoate which has become contaminated with foreign material, spillage, and floor sweepings.	5-gal. pail lined with poly bag	41-2700-4880-0, (F-4880), Net Wt.	Hold in Bldg 3
22	Equipment flushing solution	G&Y 55-gal. drum (34-7000-4455-4)	41-2600-6002-9 F-6002, Net Wt. Lot No.	Warehouse

SPECIFICATIONS FOR RELEASE:

1. In-Process None
2. Control Lab: Final
Appearance, white, free flowing powder.
% nitrogen (QCM 120.3); 3.22 - 3.29
Ash (QCM 90.4); 300 ppm max.
pH (2% Aqueous) (QCM 30.2); 4.0 min.
Free Fluoride (QCM 53.4) 250 ppm max.
Iron (QCM 86.0); 20 ppm max.
CMC (QCM 23.1) \geq .6-.8
APHA Color (0.8% Aqueous) (QCM 96.1.2) 25
max.

GLC (QCM 100.0) \geq 95% min. C⁸ 3% max.
C₁₀ + C_x
Surface Tension (QCM 38.14) 26-38 dynes/cm.

Final Sample Retain Time: 24 Months

To be released by: Chemoite Chemical Resources, QC Lab.

CONTAINERS: FC-143, as directed. G & Y 55-gallon openhead drums
(34-7000-4455-4) with thin poly-ethylene bag (34-7000-5390-2).
Less than 100 lbs. weight per unit.

STENCILLING: 41-2700-3633-2, (FM-3633, Remnants, Lot #, Net Wt.)

STORAGE: Inside.

June 8, 1978

AUTHOR:

G. A. Hoffer Date 6/8/78
G. A. Hoffer

APPROVALS:

D. D. Dworak Date 6/8/78
D. D. Dworak

R. B. Krome Date 6/14/78
R. B. Krome

D. J. Wardrop Date 7-17-78
D. J. Wardrop

P. R. Riehle Date 7-19-78
P. R. Riehle

R. R. Krall (SRS) Date 7/27/78
R. R. Krall

C. W. Bentz Date 7/26/78
C. W. Bentz

R. C. Johnson (pa) Date 7-31-78
R. C. Johnson

D. E. Morin Date 8-1-78
D. E. Morin

CHEMICAL RESOURCES DIVISION
CHEMOLITE PLANT

Factory Operating Standard

Effective Date: June 12, 1978
Superceding Date: March 5, 1978

CODE NUMBER: 41-2700-3632-4 (FM-3632)
SUBJECT: Pulverized, blended Ammonium Perfluorooctanoate for FC-126
COMMODITY CLASS: 9419
REASON FOR CHANGE: 1. Eliminate oven drying.
2. Eliminate use of blend drum by using ribbon blender in Dept. 3036.
3. Eliminate hand packaging by packaging directly from ribbon blender discharge.
REFERENCE: 1. FM-3633 Deviation, dated 5/10/78.
2. FM-3165, Lot 216, Deviation.
EQUIPMENT: BC-61, Dept. 3036, with Strong-Scott Pulverizer.

CHARGE:

<u>CODE</u>	<u>Safety Std.</u>	<u>Description</u>	<u>BC 61</u>
A. 41-2600-5695-1	--	Wet Ammonium Perfluorooctanoate	2000 lbs.
		Average Yield:	1400 lbs.
		Estimated Machine Time:	8 Hours

RAW MATERIAL SPECIFICATIONS:

41-2600-5695-1 (F-5695) Wet Ammonium Perfluorooctanoate. Must be released by QC Lab. Use only the amount and lot numbers specified by Process Engineer.

ADDITIONAL SAFETY PRECAUTIONS:

41-2600-5695-1 WET AMMONIUM PERFLUOROOCTANOATE. These materials are toxic.
41-2700-3632-4 Inhalation of dust is irritating and must be avoided. Wear a dust mask or respirator and rubber gloves when handling this material. Wash suspected areas of contact with soap and water to avoid prolonged contact. Clean up any spills (see By-Product Disposal Section, this procedure) as soon as possible to avoid contamination of the area.

OPERATING PROCEDURE:

NOTE: Read this procedure carefully before starting. This products is a snow white dry powder which is very valuable. Avoid losses and contamination.

I. PRE-RUN PREPARATION

1. No other material that creates dust should be processed in Bldg. 3 grinding room when this FM-3632 is run; especially no other grinding operations being performed.
2. The ribbon blender must be spotlessly clean before starting this run. Scrape out any material remaining from the previous run, flush with water, scrub with scotchbrite (if needed) and dry. Clean or replace the ventilation hoses and change the dust filter before charging.
3. Inspect and repair cover gaskets.

NOTE: The doors to the area must be kept closed for the rest of this operation in order for the ventilation to work properly.

II. CHARGING AND DRYING

4. Wear a dust mask, rubber gloves, and disposable coveralls when charging F-5695. Charge 60-70% of Charge "A" (F-5695), close the cover (be sure the ventilation trunks are in place), set the mixing tee to 145°F. and start up the ribbon blender.
5. After 3 hours, shut off the ribbon blender. Allow the dust be settle for 20 minutes, then charge the remainder of Charge A. Wear protective equipment and keep dusting to a minimum. Close the cover and resume drying. Clean up all spills immediately. (See By-Product Disposal).
6. Continue to dry for 8 hours. Check the unit hourly to be sure the ribbon blender is running at 145°F.

OPERATING PROCEDURE: (continued)

7. Flush the drain valve by removing 5-10 gallons from the bottom. (Wear dust mask, rubber gloves, disposable coveralls and clean up any spillage). Return this material to the blender and continue to dry for 8 hours at 145°F. The pulverizer may be set up at this time.

III. PULVERIZING

8. Position a container with poly bag liner and dial scale below the pulverizer discharge chute. Zero scale with the container in place, then check the scale for accuracy with a 50 lb. weight. Adjust the scale if needed.

9. Attach the poly bag to the discharge chute.

NOTE: Operator must wear dust mask, rubber gloves and disposable coveralls while pulverizing and packaging. See "Safety Precautions".

10. Inspect exhaust bag on pulverizer. Empty and clean bag if necessary to allow air to pulverizer. Save product dust from bag as F-4880 (by-product).
11. Start up pulverizer as in the following order:
 - a. Turn on power switch.
 - b. Turn on the grinder wheel.
 - c. Turn on the screw feed.
 - d. Adjust the vari-drive to speed 4-5. Do Not adjust the vari-drive control unless the screw feed is running.
 - e. Turn on ribbon blender.
 - f. Open valve and ribbon blender.
12. Collect 50 lbs. of product in the fiber carton, close valve on blender and shut down pulverizer by reversing steps of Step 11. Check the packaging frequently to avoid plugging the pulverizer.
13. Allow dust above filled container to clear out. Adjust final net wt. to 50 lbs. by hand. Use the silk scale for final adjustments and keep this operation as near the enclosure under the ribbon blender as possible to take advantage of the ventilation. Clean up all spills. See By-Product Disposal.
14. Tie poly bag shut and cover fiber drum and stack on a pallet (14 drums to a pallet). Tape and stencil the load "41-2700-3632-4, Lot No., Net Wt." Hold in the area for QC release and final labeling.
15. Take four 8-ounce final samples after the first 700 lbs. is packaged.
16. Repeat Steps 8-15 until BC 61 is empty.

OPERATING PROCEDURE: (continued)

IV. CLEANUP

17. No clean up is necessary between FM-3633 or FM-3632 lots.

NOTE: Operator will wear dust mask, rubber gloves and boots and disposable coveralls during clean-up.

18. After the last lot in a series, remove pulverizer from ribbon blender and cover with a poly bag.

19. Remove cover to ribbon blender and use a stick and brush to dislodge any solids sticking to the cover, sides or auger. Allow these solids to collect in the bottom of the blender.

20. Drain solids to a green and yellow openhead drum. Cover drum, stencil "41-2700-3632-4, Lot , Net Wt." record as output on the production report and send drum to warehouse.

21. Position a green and yellow openhead drum under the drain to BC 61. Use the diaphragm pump to pump one drum of 41-2600-6002-9, Lot 47 (in Bldg. 6 staging area) into BC 61. Allow this material to drain into the green and yellow drum. Recycle from the drum to the unit as needed to flush out solids.

22. Cover drum and stencil "41-2600-6002-9, Lot , Net Wt." and send to warehouse. Record F-6002 input and output on the production report.

23. Flush BC 61 floor and area with water and drain to sewer.

WASTE DISPOSAL: None

BY-PRODUCT DISPOSAL:

Step

No.

DESCRIPTION

CONTAINER

STENCILLING

DISPOSITION

3, 23	Dry ammonium perfluoro-octanoate which has become contaminated with foreign material, spillage, and floor sweepings.	5-gal. pail lined with poly bag	41-2700-4880-0, (F-4880), Net Wt.	Hold in Bldg 3
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22	Equipment flushing solution	G&Y 55-gal. drum (34-7000-4455-4)	41-2600-6002-9 F-6002, Net Wt. Lot No.	Warehouse
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SPECIFICATIONS FOR RELEASE:

1. In-Process None
2. Control Lab: Final

Appearance, white, free flowing powder.
% nitrogen (QCM 120.3); 3.30 - 3.50
Ash (QCM 90.4); 300 ppm max.
pH (2% Aqueous) (QCM 30.2); 4.0 min.
Free Fluoride (QCM 53.4) 250 ppm max.
Iron (QCM 86.0); 20 ppm max.
CMC (QCM 23.1) _ .6-.8

Surface Tension (QCM 38.14) 26-38 dynes/cm.

Final Sample Retain Time: 24 Months
To be released by: Chemoite Chemical Resources, QC Lab.

CONTAINERS: FC-126, as directed. G & Y 55-gallon openhead drums
(34-7000-4455-4) with thin poly-ethylene bag (34-7000-5390-2).
Less than 100 lbs. weight per unit.

STENCILLING: 41-2700-3632-4, (FM-3632, Remnants, Lot #, Net Wt.)

STORAGE: Inside.

June 12, 1978

AUTHOR:

G. A. Hoffer Date 6/12/78
G. A. Hoffer

APPROVALS:

D. D. Dworak Date 6/12/78
D. D. Dworak

R. B. Kjome Date 6/14/78
R. B. Kjome

D. J. Wardrop Date 7-17-78
D. J. Wardrop

P. R. Riehle Date 7-19-78
P. R. Riehle

J. R. Krall (SRS) Date 7/27/78
J. R. Krall

C. W. Bentz Date 7/20/78
C. W. Bentz

R. C. Johnson Date 7-31-78
R. C. Johnson

D. E. Morin Date 8-1-78
D. E. Morin *DMK*

CHEMICAL RESOURCES DIVISION
CHEMOLITE PLANT

Factory Operating Standard

Effective Date: December 22, 1981
Superceding: February 8, 1978

CODE NUMBER: 41-2700-3256-7
SUBJECT: Perfluorooctanoic Acid Fractionation
COMMODITY CLASS: 9428

CHARGE:

<u>Code</u>	<u>Safety Std.</u>	<u>Description</u>	<u>BC 33</u>
11-0000-0244-1	X	50% NaOH Cleanup	As required
A. 41-2700-3206-7	X	Crude C ₈ Acid/Inerts } Fluorocarbon flush }	Up to 30,000 Lbs. 8000.0 lbs. maximum first charge
A. 41-2700-3388-3	X	Precut	
B. 41-2700-3210-9	X	Intercut (recycle)	
B. 41-2700-3257-0	X	C ₈ Acid with C _x	
C. 41-2600-4169-8	X		
D. 11-0000-3048-3	X	Sulfuric Acid	500.0 lbs.
E. 11-0000-3179-6	X	Filter Cel	(0.2% of total FM-3206)
F. 41-2700-3160-6	X	Fluorocarbon Flush	1200.0 lbs.

YIELD:

41-2700-3256-2	X	Perfluorooctanoic Acid	14% of FM-3206
		Estimated Machine Time:	100 Hours

EQUIPMENT: Dept. 3060, BC-33
(600-gallon Monel kettle system with 300-gallon receiver,
packed column, and jacketed blow case).

ITEMS NEEDING SPECIAL ATTENTION:

1. FM-3206 should be as free of water as possible. Decant before charging, if necessary.
2. FM-3257 or F-4169 may contain white or grey solid material at room temperature. be sure to charge all solid material to kettle.
3. Scale weigh each drum of FM-3206 input. Do not use stencil weight.

ATTACHMENTS: Safety Information Sheets for all inputs, products,
and By-Products.

ADDITIONAL SAFETY PRECAUTIONS:

41-2700-3206-7 PERFLUOROOCTANOIC ACID. A very corrosive acid. White solid
41-2700-3388-3 in high concentration, or liquid when diluted with inerts. Avoid
41-2700-3210-9 skin contact. Wear full length rubber gloves and a face shield
41-2700-3257-0 when handling.
41-2600-4169-8

11-0000-3179-6 FILTER CEL. A gray powder which reacts with any HF to form S_iF_4
gas. Avoid inhalation or contact with these vapors since they
will form HF (acid) in the presence of moisture.

REASON FOR ISSUE:

1. Specify sampling of FM-3206 input.
2. Characterize By-Product codes for low % Cg Acid cuts.
3. Define procedure to recycle blue polyover packs to Cordova.

REFERENCE: Operating Standard dated 2/8/78.

MFG. FOR: 1. Customer Division: Commercial Chemicals Division
2. Person: J. W. Eberlin, Bldg. 53-4N

END PRODUCT USE: Input to 41-2600-6049-0, and 41-2600-6510-1.

OPERATING PROCEDURE:

I. PRE-RUN PREPARATION

1. Cut in the packed column and blank off the open column.
2. Kettle, receiver and blowcase must be clean and dry. Blank off both sight glasses or line with Kel-F sheet. The FM-3206 contains HF which disintegrates glass.
3. Pressure test the system and obtain a leak rate less than 2 psi per hour at 40 psig.
4. Check out vacuum control system. It must be able to control at 20 mm or less pressure before proceeding.
5. Valve to by-pass the decanter.
6. Open by-pass valve in liquid seal loop in reflux return line.

II. FRACTIONATION

A. INERTS REMOVAL

7.
 - a. Turn on cooling water to kettle and receiver jacket.
 - b. Turn on condenser water and set water exit control at 70°F.
8. Lower the kettle pressure to 200 mm through the receiver and vacuum Charge "A" up to 8,000 lbs. Do not reduce the pressure below 200 mm while charging as an excessive loss of low boiling fluorocarbons may result.

Scale weigh each drum of input and record this weight. Do NOT use stencil weight.

The blue poly overpacks are to be recycled to Cordova. Blot out labelling and stencil "When empty return to Cordova, 34-7010-1156-0".

9. Turn on agitator at speed #3. Before adding sulfuric acid, take a 2-oz. sample from the reactor to the QC Lab. Label "41-2700-3256-2, Lot No., Crude Cg Acid, QCM 100.0, Step 9". Do not wait for results. Vacuum Charge D, Sulfuric Acid. Turn off cooling water to kettle jacket. Vent kettle on cold side of condenser.
10. Turn 100 psi steam to kettle jacket. Set DP at 30 mm and set reflux timer setting at 2/4 (2 secs. product takeoff over 4 secs. reflux). Adjust DP setting and steam pressure as necessary to get 600-800 lbs./hr. takeoff rate.

OPERATING PROCEDURE: (continued)

11. Distill off FM-3160 (inerts) at the rate of 600-800 lbs./hr. to a kettle temperature of 275°F. Do not exceed 3000 lbs. net on the receiver.
12. Drain distillate to recycle Black poly overpacks 34-7010-1156-0 as available. Or use blue and yellow, oil type drums 34-7000-4449-7. .
Watch for a top phase of water while draining and discard any water found. Record weight of water discarded. Stencil drums "41-2700-3160-6, Lot ____, Net, and Drum Number". To each drum of FM-3160 add about one (1) ounce of RM-3177 or 2 ounces of RM-244 (preferred). Do not sample FM-3160. (No need for (1) ounce caustic addition if poly lined drums are used).

NOTE: Use Teflon tape on metal bung threads for drums of FM-3160. It will make charging easier when material is used.

13. If more Charge A remains to be distilled, vacuum charge same to the blowcase, and charge to kettle in 1000-lb. increments as the FM-3160 is distilled off. Do not exceed 200 mm vacuum while charging the blowcase. Do not exceed 3500 lbs. net in the blowcase. If more than one lot of FM-3206 is charged, take one 2-ounce sample to the QC Lab (from the drum) of each lot of FM-3206. Label the sample 41-2700-3256-2, Lot No., Crude C₈ Acid QCM 100.C, Step 13.

When the last FM-3206 is charged to the kettle, empty receiver then distill 1200 lbs. of FC's to the receiver at total takeoff (adjust DP to 1200 lbs. in 30 minutes or 200 lbs. in 5 minutes). Transfer FC's back to kettle and discard top water phase. Record pounds of water found. Resume distillation per standard.

14. When pot temperature reaches 275°F. and no more Charge A remains to be charged, cool pot to 150°F.
15. Vacuum Charges B and C (recycle C₈ acid) to the kettle. This material can solidify so charge immediately after removing from hot room.
16. Open kettle manhole and add Charge E (filter cel). Close and tighten down manhole.

B. PRECUT

17.
 - a) Set reflux timer at 2 sec. product/8 sec. reflux.
 - b) Turn on 100 psig steam and set DP at 50 mm.
 - c) Pull vacuum to 10 mm absolute pressure.
18. Distill total precut to receiver until head temperature reaches 160°F. at 10 mm absolute pressure. Precut is usually about 1800 lbs. Slowly increase DP setting and steam pressure to obtain 300 lbs/hour take-off rate. Drain distillate to clean used acrylic acid O/T drums.

Stencil precut "41-2700-3256-2, Lot No., Net Wt., Drum No." Take a 2-ounce in-process sample to QC Lab.

OPERATING PROCEDURE: (continued)

C. INTERCUT

19. a) Increase steam pressure on kettle jacket to 100 psig.
- b) Turn on heat to trace heaters.
- c) Turn on 130°F. water to condenser and receiver jacket.

Adjust condenser exit water temperature as follows:

Reduce water exit controller set point to lowest possible. Manually adjust steam and water through mixing tee until the specified temperature is obtained.

20. Distill intercut to the receiver (2/8 timer setting) at full vacuum. Adjust DP setting as necessary to obtain 500 lbs./hour take-off rate. Drain each 600 pound accumulation to clean used acrylic acid O/T drums. Before draining put the kettle on total reflux. Switch vacuum over to kettle only, and break the vacuum on the receiver with inert gas. After draining, very slowly pull vacuum on receiver until the system is equalized. Continue total reflux until head temperature and DP have reached equilibrium, then resume reflux as specified above.

Stencil intercut "41-2700-3256-2, Lot No., Net Wt., Drum No." Take a 2-ounce in-process sample from each drum to the QC Lab.

Hold the precut and intercut in the building for restencilling as:

41-2700-3210-9	Less than 5% C ₈ Acid
41-2700-3257-0	5 to 60% C ₈ Acid

21. The intercut is ended when head temperature reaches 192°F. at 10 mm. If pressure is not exactly 10 mm, end the intercut when the head temperature levels off.

NOTE: 1. When draining receiver, do so rapidly to avoid freeze-ups.
2. Feel temperature of reflux return line once per hour and note. This line must remain warm while in intercut and main cuts. Consult Supervisor if it cools off.

D. MAIN CUT

22. a) Increase condenser water exit temperature to 140°F.
- b) Increase steam pressure to 125 psig.
23. Distill main cut at a 2/8 reflux timer setting and DP of 40-80. Adjust DP to obtain 600 lbs./hour take-off rate. Drain each 600 lbs. collected to Orange and Yellow stainless steel drum, if Black polyoverpacks (34-7010-1156-0) are not available.

Stencil main cut "41-2700-3256-2, Lot., Net. Wt., Drum No." Take a 2-ounce in-process sample from each drum to QC Lab.

OPERATING PROCEDURE: (continued)

24. When a lot has a Charge C, F-4169, distill the first 1800 lbs. of the maincut at 2/8 and 600 lbs./hour. Then change reflux ratio to 2/12 (400 lbs./hour) for balance of the main cut. This will concentrate the undesirable C_x component in the last drum.
25. When the take-off rate falls below 100 lbs. per hour with the steam pressure greater than 150 psi, switch to total take-off to get as much C_g acid out of the bottoms as possible.

When the take-off rate falls below 50 lbs. per hour and the pot temperature rises to 300°F. at 10 to 20 mm vacuum, isolate the receiver and drain the last of the main cut.

III. FLUOROCARBON FLUSH

26.
 - a) Reduce steam pressure to 50 psig.
 - b) Isolate system at full vacuum. Charge F, FM-3160 from this fractionation, to the receiver and transfer to isolated kettle.
 - c) Break vacuum on system with inert gas. Vent through receiver.
 - d) Leave condenser water temperature at 140°F.
 - e) Put cooling water on receiver jacket.
 - f) Set overhead for total take-off to receiver.
27. Distill the flush to the receiver. End distillation when take-off rate becomes nil.

Drain flush in receiver to used acrylic acid drums or empty FM-3388 drums. Stencil 41-2700-3388-3, Lot No., Net Wt." Take one 2-ounce, in-process sample of the flush to the QC Lab. Label sample FM-3388 and Lot No. of this lot.

28. Turn on cooling water to the kettle. Drain bottoms to OH scrap drums.
CAUTION! HOT SULFURIC ACID!

Take a 2-ounce sample of the bottoms to the Lab for GLC. Label "41-2700-3256-3, Lot No., Acid Bottoms, QCM 100.0, Step 28". Stencil drums "41-3900-6383-6". Hold in Bldg. 15 for Process Engineer. If high C_g Acid content, we will restencil to "41-2600-6037-5, Lot No., Net Wt".

IV. POST CLEANUP

29.
 - a) After draining the hot acidic bottoms to OH scrap drums, scale weigh and record weight.
 - b) Fill kettle half full of water and heat to 150°F. Drain water hot to the sewer.
 - c) First charge the water portion of the 10% caustic solution to the kettle.
 - d) Vacuum charge RM-244 to the kettle with agitation. Boil system with caustic solution and drain to the sewer.
 - e) Water flush kettle and receiver.

DRAINING INFORMATION:

Containers: Use recycled black poly overpacks (34-7010-1156-0) for all distillate. If not available use the following schedule:
 FM-3160: Blue & Yellow, O/T drums (34-7000-4449-7)
 Precut, Intercut: Used acrylic acid drums (34-7002-2506-2) (Code 100)
 Maincut: Orange and Yellow, SS drums (34-7000-4450-5).

Stencilling: Inerts: 41-2700-3160-6, Lot No., Net Wt.
 Precut: 41-2700-3256-2
 Intercut: { Lot No., Net Wt.
 Maincut: { Drum No.

Storage: FM-3160, FM-3210: Field
 FM-3257: Warehouse
 FM-3256: Hold in Bldg. 15 for restencilling. Put Drum #5 and following ones in Hot Room.

Filter/Alternate: None

Red Label: Not Combustible

Flashpoint: None

Weight Per Container: 600 Lbs.

Draining Temperature: 50 to 140°F.

Draining Pressure: 0 to 2 psi

Sample Requirements:

Steps 9 and 13: 1 2-ounce sample from each lot of 41-2700-3206-7.

Step 28: 1 2-ounce sample of bottoms.

Periodic Samples During Draining:

41-2700-3160-6 None
 Precut: 41-2700-3256-2 1 2-ounce sample from receiver
 Intercut: 41-2700-3256-2 1 2-ounce sample from each drum.
 Maincut: 41-2700-3256-2 1 2-ounce sample from each drum.

SPECIFICATIONS FOR RELEASE:In-Process:

<u>Step</u>	<u>QCM #</u>	<u>Property</u>	<u>Specification</u>	<u>Test By</u>
9,13	100.607	GLC Analysis	Record % C ₅ , C ₆ , C ₇ , C ₈ , C ₉ , C ₁₀ , C _x , FC	QC
18	100.607	GLC Analysis	Record % C ₅ , C ₆ , C ₇ , C ₈ , C ₁₀ , C _x , FC	QC
20	4.1	Water	Record %	QC
23	53.8	Free Fluoride	Record %	QC
27	100.607	GLC Analysis	Record % C ₅ , C ₆ , C ₇ , C ₈ , C ₁₀ , C _x , FC	QC
28	100.607	GLC Analysis	Record % C ₈	

Restencil Codes for FM-3256:

<u>Initial Code</u>	<u>Restencil</u>	<u>Characterization</u>
41-2700-3256-2	41-2700-3210-9	C ₅ , C ₆ , C ₇ Acids with <5% C ₈
41-2700-3256-2	41-2700-3257-0	C ₅ , C ₆ , C ₇ Acids with 5 to 60% C ₈
41-2700-3256-2	41-2600-4169-8	C ₈ Acid with >5% C _x
41-3900-6383-6	41-2600-6037-5	Bottoms with >10% C ₈

To Be Released By: CRD QC Lab

SPECIAL RETAIN REQUIREMENTS: 1 Year

WASTE DISPOSAL:

Step No.: 28

Description: Sulfuric Acid Bottoms

Stencilling/Labeling: 41-3900-6383-6 on 3M Scrap Label 34-8002-2809-0
EPA Label #34-7014-3129-7 with EPA Waste Code D002

Container/Disposition: Hold in Bldg. for Process Engineer. Will incinerate
or restencil based on GC Analysis.

Amount: 500-1000 lbs.

BY-PRODUCT DISPOSAL: None

AUTHOR:

MJ Gravadahl Date 12-22-81
M. J. Gravadahl

APPROVALS:

D.D. Dworak Date 12-28-81
D. D. Dworak

R. B. Kjoine Date 12/30/81
R. B. Kjoine

W. B. Leder Date 1/3/82
W. B. Leder

P. F. Riehl Date 1-5-82
P. F. Riehl

D. R. Kraus Date 1/28/82
D. R. Kraus

C. W. Bentz Date 2/1/82
C. W. Bentz

D. E. Morin Date 1-1-82
D. E. Morin
MJG/bw

SPECIALTY CHEMICALS DIVISION
CHEMOLITE PLANT

FACTORY OPERATING STANDARD

Effective Date: December 21, 1988
Superseding: October 6, 1988

This Procedure Needs Updating:

Date: _____
Initials: _____
Safety Sheets: _____
Return To: _____

2182 - D. F. Lund - 777-0294

CODE NUMBER: 41-2700-3206-7

COMMODITY CLASS: 9428

SUBJECT: STABILIZED PERFLUOROOCTANOIC ACID IN INERTS

CHARGE:

<u>Code</u>	<u>Description</u>	Dept 3060 <u>BC-45/Lbs</u>	Dept 3060 <u>BC-34/LBS</u>	<u>#/100#</u>
A. 11-0000-0244-1	50% Sodium Hydroxide	2560.0	3072.0	13.29
B. 41-2700-3108-5	Perfluorooctanoyl Fluoride and Inerts	10000.0	12000.0	51.92
C. 41-2600-6002-9	Water w/Ammonium Salts	2500.0	3400.0	12.98
	or			
C. 11-0000-0995-8	Water			
D. 11-0000-3048-3	93% Sulfuric Acid	3000.0	4000.0	15.58
E. 41-2600-2965-1	Recycle Interphase	As Sched.	As Sched.	
F. 41-2700-3160-6	Fluorocarbon Inerts	1200.0	1200.0	5.89

YIELD:

41-2700-3206-7 Stabilized Perfluoro-
octanoic Acid 4800.0 5500.0

ESTIMATED MACHINE TIME: 30 Hours

EQUIPMENT:

1. BC-34, Dept. 3060, 1250 gallon Hastelloy reactor system.
2. BC-45, Dept. 3060, 1000 gallon Hastelloy reactor system.

ITEMS NEEDING SPECIAL ATTENTION:

1. Blank off the packed column and cut in the open column.
2. A sight glass on the bottom of the reactor will be required to locate phase splits.
3. A Kel-F liner or equivalent will be required on the inside of all sight glasses to protect them from fluoride attack.

REASON FOR CHANGE:

1. To reduce water to 70% of previous charge so entire batch can be reacidified at one time (Ref.2).
2. To specify testing product and flush only when requested by PE (Ref.3).

REFERENCE:

1. 41-2700-3206-7 Factory Operating Standard dated 10/6/88.
2. Personal Communication w/ T. K. Wilkinson, 12/16/88.
3. Personal Communication w/ M. L. Bell, 12/16/88.

MANUFACTURED FOR:

1. Customer Division: ICPD
2. Person: T. K. Wilkinson, Bldg. 53-4N

END PRODUCT USE:

Feed for FM-3256, Perfluorooctanoic Acid Fraction, and eventual conversion to FC-26, FC-126, or FC-143.

OPERATING PROCEDURE:I. PRE-SERIES PREPARATION

1. If the kettle is equipped with a packed column it will need to be blanked off and the open column cut in.
2. The reactor, overhead, receiver and all transfer lines must be clean. A 10% hot caustic/water flush of the system followed by a water flush should be sufficient. Charge the caustic and water to the kettle. Heat with direct steam and then start adding water to fill kettle and overhead. No cleaning is required between lots of FM-3206.
3. Pressure the reactor to 15 psig with nitrogen. If the kettle fails to hold pressure, locate leaks and repair before proceeding.

II. CHARGING

4. Turn on full cooling water to the overhead condenser. If the overhead condenser is equipped with a water temperature controller the set point should be lowered to 55°F.
5. Add Charge A (11-0000-0244-1), sodium hydroxide, to the reactor by isolated vacuum. Close all valves in the charge line when complete. Turn reactor agitator on to 60 RPM.
6. Put the reactor jacket in circulating water and adjust batch set point to 50°F. This will provide maximum cooling and minimize the amount of well water being consumed.
7. Repull maximum vacuum on the reactor and then isolate. Failure to isolate will later result in an excessive loss of valuable low boiling inerts. Set reactor overhead for total reflux back to the kettle.
8. Pull maximum vacuum on the empty receiver and then isolate. Failure to isolate will later result in an excessive loss of valuable low boiling inert.
9. Set up to vacuum Charge B (41-2700-3108-5) perfluorooctanoyl fluoride and inerts into the receiver. Typically Charge B will be charged directly from a bulk storage tank. Contact the Cell Operator or Building Supervisor for the tank currently being used to store FM-3108. Secure the necessary transfer line for pressure transferring FM-3108 into the isolated receiver. Charge B must be weighed and checked for HF electrolyte. Due to receiver size limitations multiple transfers may be necessary. Proceed to next step before starting transfer.

Sometimes Charge B (41-2700-3108-5) will be in drums. Drum stock should be vacuum charged into the isolated receiver. The Operator should wear safety glasses, face shield, rubber coat, rubber pants, and rubber gloves when handling FM-3108 to protect themselves from the possible presence of HF. Proceed to next step before starting charging.

10. With the source of FM-3108 determined and the necessary set up complete, begin charging Charge B (41-2700-3108-5), perfluorooctanoyl acid fluoride and inerts, into the isolated receiver. Continue adding Charge B to the receiver until it is full or the total amount of Charge B specified on the run card is charged.
11. Break vacuum and pressurize receiver to approximately 15 psig with nitrogen.
12. Slowly pressure transfer Charge B from the receiver into the reactor. An exotherm will occur in the reactor during this transfer. Control the rate of addition so that the reactor temperature does not exceed 190°F. Keep a careful eye on the sightglass in the transfer line between the receiver and the reactor. Transfer should be promptly terminated if any top HF phase is encountered in the receiver. The HF phase is typically brown or black as opposed to the clear or sometimes hazy FM-3108 product. If any HF phase is encountered it should be returned to the bulk FM-3108 tank, to the FM-3108 cell system if available, or Discotherm for recovery. Consult with Building Supervisor for best option.
13. If there is additional Charge B to be charged, return to Step 7 thru 12 for charging instructions. Do not repull vacuum on the reactor if it contains any FM-3108 cell product. Proceed to the next step if all of Charge B has been added to the reactor.
14. Update the Bulk Storage Tank log book in the cell office if any bulk FM-3108 was used in this lot. Adjust figures as necessary to account for any HF phase that was transferred.

III. STABILIZATION REACTION

15. With reactor jacket in circulating water, increase the batch set point to 215°F. Leave reactor sealed, agitator at 60 RPM, cold water on overhead condenser, and overhead set for total condensate return to the kettle.
16. React the batch at 215°F for 8 hours. If reactor pressure exceeds 20 psig during this period, carefully and slowly vent reactor from the cold side of the condenser to the receiver with receiver vent closed. Promptly pressure transfer any product collected in the receiver back into the reactor. If pressure is less than 15 psig make up difference with nitrogen.
17. Begin cooling batch to 165°F after 8 hours at 215°F.

IV. pH CHECK

18. When the batch temperature is 165°F less, take an 8-ounce sample of the batch through the drain line. Wear a lowered face shield, rubber coat and gloves when sampling. Check the pH of the sample using pH paper.
19. If the pH is ≥ 10 , proceed to Step 21.
20. If the pH is less than 10, add one drum of Charge A (11-0000-0244-1) to the receiver. Slowly pressure transfer contents to the reactor. Reheat the batch to 215°F and hold for four hours. After the four hour hold, cool and resample for pH as in Step 18. Repeat steps 18-20 until pH is 10 or greater.

V. INERTS STRIP

21. Slowly vent any residual pressure on the reactor from the cold side of the condenser thru the receiver.
22. Set up overhead for total takeoff of distillate to receiver. Open vent on receiver.
23. Increase batch set point to 200°F and begin stripping inerts to receiver under atmospheric conditions. Adjust batch set point and or ΔT setting as necessary to attain a takeoff rate between 1200 and 1800 lbs/hr. Put cooling water on receiver jacket if available.
24. Strip inerts to the receiver until the amount collected is equal to half the amount of Charge B charged to this lot. Put reactor back in total condensate return to the kettle if it becomes necessary to drain the receiver before completing strip.
25. At conclusion of strip, put reactor back in total reflux and begin cooling batch to 150°F.
26. Drain the receiver to drums while the reactor is cooling. Label the inerts "41-2700-3160-6, Lot No., Net Wt.". Refer to the By-Product Information section of this standard for further information. Hold 1200 pounds of FM-3160 by-product in building. It will be used as Charge F later in this run.

VI. REACIDIFICATION

27. Lower the batch setpoint to 130°F. Valve the overhead for total condensate return to the kettle. Make sure full cooling is on the overhead condenser.
28. Add Charge **C (41-2600-6002-9 or 11-0000-0995-8)** water with ammonium salts or water, to the receiver by isolated vacuum. Break vacuum with nitrogen and pressure transfer Charge C from the receiver to the reactor.
29. Add Charge **D (11-0000-3048-3)** to the receiver by isolated vacuum. Break vacuum and pressurize receiver to 15-20 psig with nitrogen. Slowly pressure transfer Charge D to the reactor. Control the rate of addition to keep the batch below 160°F.
30. If specified, add Charge **E (41-2600-2965-1)**, recycle interphase, to the receiver by isolated vacuum. Pressure transfer Charge E from the receiver into the reactor.
31. Carefully and slowly vent any pressure on the reactor from the cold side of the condenser thru the receiver. Set up overhead for total condensate return to the kettle under atmospheric conditions.
32. Increase batch set point to 200°F and begin heating up. Increase or decrease batch set point as necessary to establish a gentle reflux and hold there for 30 minutes.
33. After the 30 minute reflux period, cool batch to 130°F. When batch temperature is 130°F, turn agitator off and let batch phase split for 90 minutes.
34. The bottom product phase in the reactor consists of perfluorooctanoic acid and inerts. The top phase is sulfuric acid and water. Typically there will be a small amount of interphase dirt between the two phases. After the 90 minute phase split, drain the bottom product phase to drums. Expect about 4800 to 5600 pounds of product phase in BC-33 or BC-45 and 5800 to 6800 pounds of product phase from BC-34. Label product "41-2700-3206-6, Lot No., Net Wt.". Take one 8-ounce final sample halfway through draining. Stop draining when the top phase or interphase appears in the sightglass. Do not drain top phase to sewer.

VII. INERT FLUSH

35. Pull vacuum on the receiver and isolate. Vacuum Charge **F (41-2700-3160-6)** fluorocarbon inerts that originated from the inert strip section of this run. Pressure transfer Charge F from the receiver into the reactor.
36. Mix reactor for 15 minutes at 60 RPM and 130°F.
37. Turn agitator off and allow batch to phase split for 45 minutes.

38. The bottom product phase in the reactor consists of dilute perfluorooctanoic acid in inerts. The top phase is sulfuric acid and water. Typically there will be a small amount of interphase dirt between the two phases. Drain the bottom product phase to drums. Expect about 1000 to 1500 pounds. Label the bottom product phase "41-2700-3388-3, Lot No., Net Wt.". Take one 8-ounce final sample halfway through draining. Refer to By-Product Information page of this standard for further information. Stop draining when the top phase or interphase appears in the sightglass.
39. If the phase splits have been good and the expected amount of product has been collected proceed to the next step. If there has been a poor phase split, there may be an excessive amount of interphase that could contain product. If this is the case the interphase should be dained to drums and labeled "41-2600-2965-1, Lot No., Net Wt.". Refer to By-Product Information section of this standard for further information.
40. Drain the top sulfuric acid and water phase in the reactor to the sewer. Set up a water hose to help flush the sewer.

VIII. POST-RUN

41. No between run cleanup is required between lots of FM-3206 or if FM-3256 is scheduled to follow this lot. A caustic and water boil of reactor, overhead, and receiver followed by a water flush should effectively clean the reactor system.
42. Turn off vacuum jets, water to overhead condenser, water to receiver and reactor jackets, and turn off reactor and receiver agitator.

DRAINING INFORMATION:

Product Code: 41-2700-3206-7

Description: Stabilized Perfluorooctanoic Acid in Inerts

Step No.: 34

Label: "41-2700-3206-7, Lot No., Net Wt."

Container / Alternate: Recycle Poly Overpak 55-gallon drum (34-7010-1156-0) or
New Poly Overpak 55-gallon drum (34-7000-4433-1)

Weight per Container: 600 LBS. NET

Red Label: None

Filter / Alternate: None

Draining Temperature: Less than 140°F

Draining Pressure: 0 to 3 psig

Storage Instructions: Inside

Special Draining Instructions: None

Sample Requirements:

QC Lab: One, 8-ounce final sample.

Customer: None

Special Shipping Instructions: None

SPECIFICATIONS FOR RELEASE:IN-PROCESS:

<u>Step</u>	<u>OCM#</u>	<u>Property</u>	<u>Specification</u>	<u>Test By</u>
18	---	pH	≥ 10	Production

FINAL:

<u>OCM#</u>	<u>Property</u>	<u>Specification</u>	<u>Test By</u>
100.607	GLC*	R/R % Inert, C ₅ , C ₆ , C ₇ , C ₈ , C ₁₀ , C _x	QC

* Run only when requested by PE.

To Be Released By: No Release Required

Retain Requirements: 1 year

BY-PRODUCT:

41-2700-3388-3

100.607	GLC*	R/R % Inert, C ₅ , C ₆ , C ₇ , C ₈ , C ₁₀ , C _x	QC
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* Run only when requested by PE.

To Be Released By: No Release Required

Retain Requirements: 1 year

WASTE DISPOSAL:

Step No.: 40
Description: Sulfuric Acid and Water
Container / Disposition: Drain to sewer, flush sewer with water

BY-PRODUCT INFORMATION:

By-Product Code: 41-2700-3160-6
Description: Distilled Inerts
Step No.: 26
Stenciling / Labeling: "41-2700-3160-6, Lot No., Net Wt."
Container / Disposition: Recycle Poly Overpak 55-gallon drum (34-7010-1156-0) or
New Poly Overpak 55-gallon drum (34-7000-4433-1)
Weight Per Container: 600 lbs
Red Label: None
Filter: None
Draining Temperature: Less than 100°F
Draining Pressure: 0 to 3 psig
Storage Instructions: None
Special Draining Instructions: None
Sample Requirements: None
To Be Released By: No Release Required

BY-PRODUCT INFORMATION:

By-Product Code: 41-2700-3388-3

Description: Dilute Perfluorooctanoic Acid and Inerts

Step No.: 38

Label: "41-2700-3388-3, Lot No., Net Wt."

Container: Recycle Poly Overpak 55-gallon drum (34-7010-1156-0) or
New Poly Overpak 55-gallon drum (34-7000-4433-1)

Weight Per Container: 600 lbs

Red Label: None

Filter: None

Draining Temperature: Less than 140°F

Draining Pressure: 0 to 3 psig

Storage Instructions: Inside

Special Draining Instructions: None

Sample Requirements: One, 8-ounce final sample from mid-way through draining.

BY-PRODUCT INFORMATION:

By-Product Code: 41-2600-2965-1

Description: Recycle Interphase from FM-3206

Step No.: 39

Label: "41-2600-2965-1, Lot No., Net Wt."

Container: Recycle Poly Overpak 55-gallon drum (34-7010-1156-0) or
New Poly Overpak 55-gallon drum (34-7000-4433-1)

Weight Per Container: 600 lbs

Red Label: None

Filter: None

Draining Temperature: 175 to 130°F

Draining Pressure: 0 to 3 psig

Storage Instructions: Inside

Special Draining Instructions: None

Sample Requirements: None

To Be Released By: No Release Required

AUTHORS: Process Engineer *D. F. Lund* Date 12/21/88
D. F. Lund

Product Chemist *M. L. Bell* Date 12/28/88
M. L. Bell

APPROVED BY:

MD Schopp
M. D. SCHOPP,
12/29/88

RB Krome 12/28/88
R. B. KROME,

J. E. FINCHER,

D. D. DWORAK,

L. L. ENNETT,

C. W. BENTZ,

J. E. STAIGER
RW/C:\WS2000\F\FM\FM3206_1.288

**SPECIALTY ADHESIVES & CHEMICALS DIVISION
CHEMOLITE PLANT**

FACTORY OPERATING STANDARD

Effective Date: May 22, 1991
Superseding: December 21, 1988

This Procedure Needs Updating:

Date: _____
Initials: _____
Safety Sheets: _____
Return To: _____

CODE NUMBER: 41-2700-3206-7

COMMODITY CLASS: 9428

SUBJECT: STABILIZED PERFLUOROOCCTANOIC ACID IN INERTS

YIELD:

41-2700-3206-7 Stabilized Perfluorooctanoic Acid 4800.0 (BC-45)
5500.0 (BC-34)

ESTIMATED MACHINE TIME: 30.0 hours

EQUIPMENT:

1. BC-34, Dept. 3060, 1250 gallon Hastelloy reactor system.
2. BC-45, Dept. 3060, 1000 gallon Hastelloy reactor system.

ITEMS NEEDING SPECIAL ATTENTION:

1. A sight glass on the bottom of the reactor will be required to locate phase splits.
2. A Kel-F liner or equivalent will be required on the inside of all sight glasses to protect them from fluoride attack.

REASON FOR CHANGE: (Revise Product Structure? YES NO)

1. To update personal protective equipment requirements for handling FM-3108.
2. To reflux cool the batch to clean the overhead and to control reaction rates during FM-3108 charging.

REFERENCE:

1. 41-2700-3206-7 Factory Operating Standard dated 12/21/88.
2. Meetings with supervisors and operators.

MANUFACTURED FOR:

1. Customer Division: ICPD
2. Person: T. K. Wilkinson, Bldg. 236-3A-04.

END PRODUCT USE:

Feed for FM-3256, Perfluorooctanoic Acid Fractionation, and eventual conversion to FC-26, FC-118, FC-126, or FC-143.

OPERATING PROCEDURE:I. PRE-SERIES PREPARATION

1. A packed column or open column can be used.
2. The reactor, overhead, receiver and all transfer lines must be clean. A hot caustic water flush of the system followed by a water rinse should be sufficient. Wear a face shield, rubber jacket and rubber gloves to handle RM-244. Charge the caustic and water to the kettle. Heat with direct steam and then start adding water to fill kettle and overhead while maintaining 200-210°. After flush, drain solution to sewer.

No cleaning is required between lots of FM-3206.

3. Pressure the reactor to 15 psig with nitrogen. If the kettle fails to hold pressure, locate leaks and repair before proceeding.

II. CHARGING

4. Turn on full cooling water to the overhead condenser. If the overhead condenser is equipped with a water temperature controller the set point should be lowered to 55°F.
5. Wear a face shield, rubber jacket and rubber gloves to handle RM-244. Add Charge **A (11-0000-0244-1)**, sodium hydroxide, to the reactor by isolated vacuum. Close all valves in the charge line when complete. Turn reactor agitator on to 60 RPM.
6. Put the reactor jacket in circulating water and adjust batch set point to 50°F. This will provide maximum cooling and minimize the amount of well water being consumed.
7. Repull maximum vacuum on the reactor and then isolate. Failure to isolate will later result in an excessive loss of valuable low boiling inerts. Set reactor overhead for total reflux back to the kettle.
8. Pull maximum vacuum on the empty receiver and then isolate. Failure to isolate will later result in an excessive loss of valuable low boiling inert.

9. Set up to vacuum Charge B (41-2700-3108-5) perfluorooctanoyl fluoride and inerts into the receiver. Typically Charge B will be charged directly from a bulk storage tank. Contact the Cell Operator or Building Supervisor for the tank currently being used to store FM-3108. Secure the necessary transfer line for pressure transferring FM-3108 into the isolated receiver. Charge B must be weighed and checked for HF electrolyte. Due to receiver size limitations multiple transfers may be necessary. Proceed to next step before starting transfer.

Sometimes Charge B (41-2700-3108-5) will be in drums. Drum stock should be vacuum charged into the isolated receiver. Operators should wear full enclosure hood (white cap), rubber coat, rubber pants, rubber boots and rubber gloves when handling FM-3108 to protect themselves from the possible presence of HF. Proceed to next step before starting charging.

10. With the source of FM-3108 determined and the necessary set up complete, begin charging Charge B (41-2700-3108-5), perfluorooctanoyl acid fluoride and inerts, into the isolated receiver. Continue adding Charge B to the receiver until it is full or the total amount of Charge B specified on the run card is charged.
11. Break vacuum and pressurize receiver to approximately 15 psig with nitrogen.
12. Slowly pressure transfer Charge B from the receiver into the reactor. An exotherm will occur in the reactor during this transfer. Control the rate of addition or use reflux cooling so that the reactor temperature does not exceed 190°F.

Keep a careful eye on the sightglass in the transfer line between the receiver and the reactor. Transfer should be promptly terminated if any top HF phase is encountered in the receiver. The HF phase is typically dark brown or black as opposed to the light brown to brown FM-3108 product. If any HF phase is encountered it should be returned to the bulk FM-3108 tank, to the FM-3108 cell system if available, or Discotherm for recovery. Consult with Building Supervisor for best option.

NOTE: Once the batch exceeds 190°F, reflux cooling will be necessary to lower the batch temperature.

NOTE: Prolonged batch temperature above 225°F will breakdown C8 acids to less valuable inerts.

13. If there is additional Charge B to be charged, return to Step 8 through 12 for charging instructions. Do not repull vacuum on the reactor if it contains any FM-3108 cell product. Proceed to the next step if all of Charge B has been added to the reactor.
14. Update the Bulk Storage Tank log book in the cell office if any bulk FM-3108 was used in this lot. Adjust figures as necessary to account for any HF phase that was transferred.

III. STABILIZATION REACTION

15. With reactor jacket in circulating water, increase the batch set point to 215°F. Leave reactor sealed, agitator at 60 RPM, cold water on overhead condenser, and overhead set for total condensate return to the kettle.
16. React the batch at 215°F for 8 hours. If reactor pressure exceeds 20 psig during this period, carefully and slowly vent reactor from the cold side of the condenser to the receiver with receiver vent closed. Promptly pressure transfer any product collected in the receiver back into the reactor. If pressure is less than 15 psig make up difference with nitrogen.
17. After 8 hours at 215°F, reflux cool the batch to 165°F by slowly venting the batch to maintain a moderate reflux rate back to the kettle.

NOTE: Refluxing is necessary to clean the overhead and return unstabilized material to the kettle.

IV. pH CHECK

18. When the batch temperature is 165°F less, take an 8-ounce sample of the batch through the drain line. Wear a lowered face shield, rubber coat and gloves when sampling. Check the pH of the sample using pH paper.
19. If the pH is greater than or equal to 10, proceed to Step 21.
20. If the pH is less than 10, wear personal protective equipment specified in Step 18 and add one drum of Charge C (11-0000-0244-1) to the receiver. Slowly pressure transfer contents to the reactor. Reheat the batch to 215°F and hold for four hours. After the four hour hold, reflux cool the batch to 165°F and resample for pH as in Step 18. Repeat steps 18-20 until pH is 10 or greater.

V. INERTS STRIP

21. Slowly vent any residual pressure on the reactor from the cold side of the condenser through the receiver.
22. Open the vent on the receiver and verify the drain valve is closed. Set the overhead for total take-off of distillate to the receiver.

Adjust batch setpoint and/or ΔT setting as necessary to obtain a take-off rate between 1200 and 1800 lbs./hr. Use cooling water on the receiver jacket if available.

23. Strip inerts to the receiver until the amount collected is equal to half the amount of Charge B charged to this lot. Put reactor back in total condensate return to the kettle if it becomes necessary to drain the receiver before completing strip.
24. At conclusion of strip, put reactor back in total reflux and begin cooling batch to 150°F.
25. Drain the receiver to drums while the reactor is cooling. Label the inerts "41-2700-3160-6, Lot No., Net Wt.". Refer to the By-Product Information section of this standard for further information. Hold the first two drums, 1200 pounds, of FM-3160 by-product in building. It will be used as Charge F later in this run.

VI. REACIDIFICATION

26. Lower the batch setpoint to 130°F. Valve the overhead for total condensate return to the kettle. Make sure full cooling is on the overhead condenser.
27. Add Charge **D (Water, Water with Ammonium Salts or Water with Fluorochemical Acids)** to the receiver by isolated vacuum. Break vacuum with nitrogen and pressure transfer Charge D from the receiver to the reactor.
28. Add Charge **E (11-0000-3048-3)** to the receiver by isolated vacuum. Break vacuum and pressurize receiver to 15-20 psig with nitrogen. Slowly pressure transfer Charge E to the reactor. Control the rate of addition to keep the batch below 200°F.
29. Carefully and slowly vent any pressure on the reactor from the cold side of the condenser through the receiver. Set up overhead for total condensate return to the kettle under atmospheric conditions.
30. Increase batch set point to 200°F and begin heating up. Increase or decrease batch set point as necessary to establish a gentle reflux and hold there for 30 minutes.
31. After the 30 minute reflux period, cool batch to 130°F. When batch temperature is 130°F, turn agitator off and let batch phase split for 90 minutes.

32. The bottom product phase in the reactor consists of perfluorooctanoic acid and inerts. The top phase is sulfuric acid and water. Typically there will be a small amount of interphase dirt between the two phases. After the 90 minute phase split, drain the bottom product phase to drums. Expect about 1/2 of the amount of Charge B. Label product "41-2700-3206-6, Lot No., Net Wt.". Take one 8-ounce final sample halfway through draining. Stop draining when the top phase or interphase appears in the sightglass. Do not drain top phase to sewer.

VII. INERT EXTRACTION

33. Pull vacuum on the receiver and isolate. Vacuum Charge F (41-2700-3160-6) fluorocarbon inerts that originated from the inert strip section of this run to the receiver. Pressure transfer Charge F from the receiver into the reactor.
34. Mix reactor for 15 minutes at 60 RPM and 130°F.
35. Turn agitator off and allow batch to phase split for 45 minutes.
36. The bottom product phase in the reactor consists of dilute perfluorooctanoic acid in inerts. The top phase is sulfuric acid and water. Typically there will be a small amount of interphase dirt between the two phases. Drain the bottom product phase to drums. Expect about 1000 to 1500 pounds. Label the bottom product phase "41-2700-3388-3, Lot No., Net Wt.". Take one 8-ounce final sample halfway through draining. Refer to By-Product Information page of this standard for further information. Stop draining when the top phase appears in the sightglass.
37. Drain the top sulfuric acid and water phase in the reactor to the sewer. Set up a water hose to help flush the sewer.

VIII. POST-RUN

38. No between-run cleanup is required when FM-3206 or FM-3256 is scheduled to follow. If FM-3206 or FM-3256 is not scheduled to follow, a hot caustic and water flush of the system will be necessary. Wear a lowered face shield, rubber jacket and rubber gloves to handle RM-244. Charge caustic and water to the kettle. Heat with direct steam and then start adding water to fill the kettle and overhead while maintaining 200-210°F. Overflow to the receiver. Drain to the chemical sewer. Rinse the system with water.
39. Turn off vacuum jets, water to overhead condenser, water to receiver and reactor jackets, and turn off reactor and receiver agitators.

DRAINING INFORMATION:

Product Code: 41-2700-3206-7

Description: Stabilized Perfluorooctanoic Acid in Inerts

Container / Alternate: Recycle Poly Overpak 55-gallon drum
(34-7010-1156-0)
or
Remanufactured Poly Overpak 55-gallon drum
(34-7029-4109-6)
or
New Poly Overpak 55-gallon drum (34-7002-2745-6)

Weight per Container: 600 LBS. NET

Filter / Alternate: None

Draining Temperature: Less than 140°F.

Draining Pressure: 0 to 3 psig.

Storage Instructions: Inside.

Special Draining Instructions: None.

Sample Requirements:

QC Lab: One, 8-ounce final sample.
Customer: None

Special Shipping Instructions: None

SPECIFICATIONS FOR RELEASE:IN-PROCESS:

<u>Step</u>	<u>OCM#</u>	<u>Property</u>	<u>Specification</u>	<u>Test By</u>
18	---	pH	10 minimum	Production

FINAL:

100.607	GLC*	R/R % Inert, C ₅ , C ₆ , C ₇ , C ₈ , HB	QC
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* Run only when requested by PE.

To Be Released By: No Release Required

Retain Requirements: 1 year

BY-PRODUCT:

41-2700-3388-3

100.607	GLC*	R/R % Inert, C ₅ , C ₆ , C ₇ , C ₈ , HB	QC
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* Run only when requested by PE.

To Be Released By: No Release Required

Retain Requirements: 1 year

WASTE DISPOSAL:

Step No.:	37	2, 38
Description:	Sulfuric Acid and Water	10% Caustic Solution
Container / Disposition:	Drain to sewer, flush sewer with water	Chemical sewer, flush with water
Amount:	Up to 7500 lbs.	Up to 8000 lbs.

BY-PRODUCT INFORMATION:

By-Product Code:	41-2700-3160-6
Description:	Distilled Inerts
Step No.:	25
Container / Disposition:	Recycle Poly Overpak 55-gallon drum (34-7010-1156-0) or Remanufactured Poly Overpak 55-gallon drum (34-7029-4109-6) or New Poly Overpak 55-gallon drum (34-7002-2745-6)
Weight Per Container:	600 lbs
Filter:	None
Draining Temperature:	Less than 100°F
Draining Pressure:	0 to 3 psig
Storage Instructions:	None
Sample Requirements:	None

BY-PRODUCT INFORMATION:

By-Product Code: 41-2700-3388-3

Description: Dilute Perfluorooctanoic Acid and Inerts

Step No.: 36

Container: Recycle Poly Overpak 55-gallon drum (34-7010-1156-0)

Weight Per Container: 600 lbs

Filter: None

Draining Temperature: Less than 140°F

Draining Pressure: 0 to 3 psig

Storage Instructions: Inside

Special Draining Instructions: None

Sample Requirements: One, 8-ounce final sample from mid-way through draining.

AUTHORS: Process Engineer G. E. Bentz Date 5/23/91
 G. E. Bentz
 Product Chemist E. A. Hafiz Date 5/24/91
 E. A. Hafiz

APPROVED BY:

Process Engineering Supervisor: A. C. Hoffmann Date 5/30/91

Health & Safety Eng.: yes no Date _____

Bldg. Senior Supv.: yes no Date _____

Quality Engineering Supervisor: Robert A. Hall Date 10-2-91

ICPD - Chemical Specialties: Thomas K. Wilkinson Date 10/10/91

RW/S:\FM\FM32\FM3206_5.91

**3M COTTAGE GROVE PLANT
CHEMICAL PLANT**

FACTORY OPERATING PROCEDURE

Effective Date: **September 25, 1996**
Superseding: **July 28, 1992**

CODE NUMBER: 41-2700-3206-7 REVISION NO.: R00

SUBJECT: STABILIZATION OF PERFLUOROOCCTANOIC ACID IN INERTS

YIELD:

41-2700-3206-7	Stabilized Perfluorooctanoic Acid	5,800 lbs (BC-34) 4,830 lbs (BC-45) (48.3% of FM-3108 input)
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ESTIMATED MACHINE TIME: 26.0 Hours

EQUIPMENT:

1. BC-34; Dept 3060, 1,250 gallon Hastelloy reactor, 1,000 gallon Monel receiver with packed or open column. 250 gallon JBC charge/weigh vessel. (Normal and preferred reactor system).
2. BC-45; Dept 3060, 1,000 gallon Hastelloy reactor, 1,000 gallon Hastelloy receiver with overhead and open column system. (Backup unit only).

ATTACHMENTS:

1. None

ITEMS NEEDING SPECIAL INSTRUCTIONS:

1. None

SHUTDOWN INSTRUCTIONS:

1. This batch can be shut down at any time without causing a quality or a safety problem. If the reaction is interrupted, additional reaction time must be allowed. Consult the Engineer for details. The product may set become solid in the reactor if the temperature is allowed to drop below 130 °F.

REASON FOR ISSUE:

(Revise Product Structure? Yes ___ No)(New or Changed Emissions? ___ Yes No)

1. To update procedure to the current format.
2. To specify transfer of FM-3160 inerts to bulk tank instead of draining to drums.

REFERENCE:

1. 41-2700-3206-7 Factory Operating Procedure dated July 28, 1992.

MANUFACTURED FOR:

1. Customer Division: SCD
2. Customer Plant Contact: NA
3. Customer Lab Contact: Marylee Maendler - 236-2A-01
4. SMD/SA&C Lab Contact: Tom K. Wilkinson - 53-6S-02

END PRODUCT USE:

Emulsifier in teflon production

FORMULATION NOTES: If a batch size adjustment of more than 5% is required, all the charges should be scaled in proportion to the normal charges.

CHARGE CALCULATIONS: None

PROCESS TOLERANCE:

1. Unless otherwise specified, record data as indicated in the operating procedure for process variables a minimum of once per hour.
2. Unless otherwise specified, time intervals specified are a minimum time. To obtain consistent process conditions the Operator should continue processing at the specified time interval.
3. Unless otherwise specified, maintain process variations within the tolerances listed below. If unable to operate within the acceptable tolerances, contact the Supervisor or Manufacturing Engineer for instructions. Note all additional verbal instructions on the data card.

Attach all written instructions to the Production Report.

4. Unless otherwise specified in the procedure, use stencil weights for charging.

<u>PROCESS VARIABLES</u>	<u>ACCEPTABLE PROCESS TOLERANCES</u>		<u>RECORD HOURLY</u>
	(+)	(-)	<u>Y/N</u>
Agitator speed (RPM)	5	5	Y
Temperature (°F)			
batch	2	2	Y
jacket	5	5	Y
head	2	2	N
condenser water	5	5	Y
Pressure (psig)	10	10	Y
Vacuum (mm Hg)	0.5	0.5	N
(in Hg)	0.5	0.5	N
Charge Weight	2%	2%	Y
Draining Weight	1%	1%	Y
Rates	Target and tolerances are specified in the procedure.		

OPERATING PROCEDURE:

I. PRE-SERIES PREPARATION

1. The packed fractionation column or the open column can be used. Normally the packed column is left in service as the FM-3256 which follows a series requires the packed column.
2. The sight glasses on the reactor need to be protected from fluoride attack with a thin liner of Kel-F™ plastic on the inside.
3. A sight glass on the bottom of the reactor will be required to locate the phase splits.
4. The reactor, overhead, receiver, and all transfer lines must be free from contamination of other products.

Caution: *When charging caustic to the reactor the minimum personal protection required is a face shield, goggles, rubber jacket and neoprene gloves.*

A hot caustic water flush of the system followed by a water rinse should be sufficient. Vacuum charge one or two drums of 11-0000-0244-1, 50% Sodium Hydroxide, to the reactor. Start the agitator at medium speed. Fill the reactor with water and heat with direct steam. When the reactor temperature reaches 200 - 210 °F, start adding water to the reactor while maintaining 200 - 210 °F on the reactor. Overflow the reactor through the column, overhead condenser and allow to flow to the receiver. After the flush is complete cool to 130 °F and drain overhead lines, reactor, and receiver to the sewer. Flush with water to the sewer.

5. Pressure the reactor system including overhead and receiver to 40 psig with nitrogen. The pressure loss should be less than 1 lb for 30 minutes (0.5 lb for 15 minutes). Find and repair leaks as required. Use water to hydrostat system if necessary to find leaks.

II. CHARGING

6. Turn on condenser water and set to 55 °F.
7. Pull full vacuum on the reactor, isolate, and prepare to add the caustic charge:

Caution: *When charging caustic to the reactor the minimum personal protection required is a face shield, goggles, rubber jacket and neoprene gloves.*

Add Charge A (11-0000-0244-1), 50% sodium hydroxide to the reactor by isolated vacuum. Close all valves in the charge line when complete. Start the reactor agitator and adjust to medium high (~90 rpm).

8. Set the reactor jacket control mode to circulating water and adjust the batch temperature set point to 50 °F for maximum cooling.

9. Pull maximum vacuum on the reactor and isolate. Isolation is necessary to prevent possible loss of low boiling inerts. Set the overhead valving for total reflux back to the reactor.
10. Pull maximum vacuum on the empty receiver, then isolate. Isolation is necessary to prevent loss of low boiling inerts.
11. Charge B (41-2700-3108-5), Perfluorooctanoyl acid fluoride/inert cell crude to the isolated receiver from a FM-3108 storage tank with nitrogen pressure (typically ~ 25 psi). Consult with the cell operator on which tank to use. Double check all valving on the transfer line to prevent misdirected flow into another tank or vessel. Charge B must be weighed in the receiver. The 1,000 gallon receiver will hold the entire 12,000 lb charge (BC-34) and also the entire 10,000 lb charge (for BC-45). If the JBC is used multiple weighings are necessary. The maximum charge for the JBC is 3,500 lbs. Charge B must be checked for an dark electrolyte upper phase especially if the storage tank inventory is low. Transfer any electrolyte phase to the storage tank designated by the cell operator.

Log the transfer in the Storage Tank Log on the computer in the Cell Office (or have the cell operator do it). Record the ST used and the FM-3206 lot charged.

12. Break the vacuum with nitrogen and pressurize the receiver to ~ 15 psig with nitrogen.
13. Slowly pressure transfer Charge B from the receiver into the reactor. An exotherm will occur in the reactor during this transfer. Control the rate of addition or use reflux cooling so that the reactor temperature does not exceed 190 °F.

Watch the sight glass in the transfer line between the receiver and the reactor for a dark brown or black electrolyte (HF) phase. Product phase will be clear to light brown. If any HF phase is encountered terminate the transfer and transfer the electrolyte to the DISCO or storage tank designated by the cell operator.

Note: Above 190 °F, reflux cooling will be necessary to lower the batch temperature. Prolonged batch temperatures above 225 °F. will break down C8 acids to less valuable inerts.

14. If there is additional Charge B to be charged, return to Step 9-13 for charging instructions. Do not pull vacuum again on the reactor if it contains any FM-3108 cell product. Proceed to the reaction step if all of Charge B has been added to the reactor. Be sure the storage tank log on the cell office computer was updated.

III. STABILIZATION REACTION

15. Set the reactor jacket in circulating water, increase the batch temperature set point to 215 °F. The reactor should remain sealed, set the agitator at 60 RPM, set the condenser water control at 55 °F and set the condensate valving for total return to the reactor.
16. Hold the batch at reaction temperature of 215 °F for 8 hours.

17. After completion of the reaction, set the batch temperature set point to 165 °F, then reflux cool the batch to 165 °F by slowly venting the batch to maintain a moderate reflux rate. Venting too quickly can cause a loss of low boiling inerts through the condenser if it is overloaded.

Note: Refluxing is necessary to clean the overhead and return unstabilized material to the reactor.

IV. PH CHECK

18. When the batch temperature is 165 °F or less, take an 8-ounce sample of the batch through the drain line. Wear a lowered face shield, rubber coat and gloves when sampling. Check the pH of the sample using pH paper. The pH should be greater than or equal to 10. If it is proceed to the Inert Strip.
19. If the pH is less than 10 (*not expected*), add one drum of Charge C (11-0000-0244-1), 50% Sodium Hydroxide to the receiver. Observe the safety precautions below:
Caution: *When charging caustic to the reactor the minimum personal protection required is a face shield, goggles, rubber jacket and neoprene gloves.*
Slowly pressure transfer contents to the reactor. Reheat the batch to 215 °F and hold for 4 hours using the original reaction conditions. After the 4 hour hold return to Step 17.

V. INERT STRIP

20. Slowly vent any residual pressure on the reactor from the cold side of the condenser through the receiver.
21. Open the vent on the receiver and verify the drain valve is closed. Set the overhead for total take-off of distillate to the receiver.
22. Set the batch temperature setpoint to 200 °F, and set the ΔT setting to 30 °F, or as necessary to obtain a takeoff rate of 1,200 to 1,800 lbs/hr. Set the receiver jacket for cooling water.
23. Distill the inerts to the receiver until the amount collected is equal to one-half the amount of Charge B (Crude Cell Product) charged. Normal amount stripped is 6,000 lbs (BC-34) or 5,000 (BC-45). If it is necessary to drain the receiver before completing the strip (not normal), put the reactor on total condensate return to the reactor.
24. When the inert strip is complete, set the reactor temperature set point to 100 °F. Isolate reactor from receiver.
25. Drain two drums, 1,200 lbs of the inerts from the receiver to drums to be used in the inert extraction as Charge F later in this batch. Pressure the receiver to ~ 20 psig (or as necessary) the balance of the inert in the receiver to the bulk FM-3160 inert tank. Verify that the inert line is valved off to the receiver of the other two reactor systems. Watch carefully for a water phase near the end. Sewer any water phase encountered. Verify that the tank is less than 98% full on the level gauge on 1st floor under the JBC receiver. Also note the tank pressure. The tank pressure is in psia so subtract 14.7 lbs to get normal gauge pressure.
Record both the bulk inert and the drummed inert on the Byproduct yield section of the yield card. Refer the Byproduct section for more details.

VI. REACIDIFICATION

26. Verify the batch temperature is less than 130 °F. (Do not wait for temperature to reach 100 °F). The overhead valving should be total condensate return to the reactor. The condenser water control should remain on cooling (< 55 °F).
27. Add Charge D1 (41-2600-6002-9) Water/Ammonium salts, and/or D2 (11-0000-0995-8), Water to the receiver by isolated vacuum. Break vacuum with nitrogen and pressure transfer Charge D from the receiver to the reactor. [Note: Total of Charge D1 and D2 is 3,400 lbs for a normal batch size in BC-34; (BC-45 = 2,800)].
28. Add Charge E (11-0000-3048-3), Sulfuric Acid (93%), to the receiver (or to the JBC weigh tank) by isolated vacuum. Break the vacuum with nitrogen, pressure to 15 - 20 psig with nitrogen and slowly pressure transfer the sulfuric acid. Control the addition rate to keep the batch temperature below 200 °F. The acid will convert the sodium salt in the reactor to acid.
29. Carefully and slowly vent any pressure on the reactor from the cold side of the condenser through the receiver. Set up overhead for total condensate return to the reactor under atmospheric conditions.
30. Set the batch temperature set point to 200 °F and begin heating. Adjust the batch temperature as necessary to establish a gentle reflux for 30 minutes.
31. When the reflux hold is complete, set the batch temperature set point at 130 °F. When the batch temperature reaches 130 °F, turn off the agitator and let the batch phase split for 90 minutes.
32. The bottom product phase in the reactor consists of perfluorooctanoic acid and inerts. The top phase is sulfuric acid and water. Typically there will be a small amount of interphase dirt between the two phases.

After the 90 minute phase split, drain the bottom product phase to drums. Expect about one-half of the amount of Charge B. Label and sample the product per Draining page. Stop draining when the top phase or interphase appears in the sight glass. Do *not* drain the top phase to the sewer at this time.

VII. INERT EXTRACTION

33. Pull vacuum on the receiver and isolate. Vacuum Charge F (41-2700-3160-6), Crude C8 inert mixture that was saved in drums earlier in the run to the receiver. Pressure transfer Charge F from the receiver into the reactor using nitrogen.
34. Mix the reactor for 15 minutes at 60 rpm and 130 °F.
35. After the 15 minute mix turn the agitator off and allow the batch to phase split for 45 minutes.

36. When the phase split is complete, drain the lower phase to used polyoverpak drums as *Byproduct 41-2700-3388-3*. Refer to the Byproduct section for details. The bottom product phase in the reactor consists of dilute perfluorooctanoic acid in inerts. The top phase is sulfuric acid and water. Typically there will be a small amount of interphase dirt between the two phases. Stop draining when you see the top phase in the sight glass. Sampling of the product phase is not necessary.
37. Drain the top sulfuric acid phase in the reactor to the sewer after determining that the product yield is normal. Set up a water hose in the sewer to help flush the sewer. Be sure the exhaust ventilation on the sewer is working.

VIII. POST-RUN

38. No cleaning is required between lots of FM-3206 or prior to FM-3256 Fractionation.
39. If cleaning is necessary, clean with hot caustic and water.
Caution: *When charging caustic to the reactor the minimum personal protection required is a face shield, goggles, rubber jacket and neoprene gloves.*
Charge 11-0000-0244-1, Sodium Hydroxide (50%), 2 drums and 11-0000-0995-8, Water, to fill the reactor. Heat to 210 °F with direct steam and then start adding water to fill the reactor and overhead while maintaining 200 - 210 °F. Overflow to the receiver. When the overhead and receiver has been flushed with hot caustic, cool to 130 - 140 °F, and drain to the chemical sewer. Rinse the system with water to the chemical sewer.
40. If the system will be down, shut off the vacuum jets, water to the overhead condenser, water to reactor jacket, water to receiver jacket, and agitators in reactor and receiver.

DRAINING INFORMATION:

Container: 1st Choice: Recycle polyoverpaks (34-7010-1156-0) from FM-3256/F-8281/F-8282/FM-3206 drums.
2nd Choice: New polyoverpak (34-7039-5732-3)
3rd Choice: Blue new polyoverpak (34-7002-2745-6)

Filter: None

Label(s): 41-2700-3206-7, Lot ____, Net ____, Inside.

Weight Per Container: 700 lbs net

Draining Temperature: 110 - 140 °F

Draining Pressure: 0 - 3 psig

Special Draining Instructions:

Product will solidify at room temperature

Special Handling Instructions:

Remove to Bldg 3 Hot Room, normal, (or Bldg 15 Hot room) for use in FM-3256.

Final Sample Requirements:

QC Lab: One 8-ounce

Customer: None

Storage: Hot room (Bldg 3) normal. Warehouse if FM-3256 not scheduled

BY-PRODUCT DRAINING INFORMATION:

Step: 25

Code: **41-2700-3160-6**

Description: Distilled Crude C8F160 Inert Mixture

Container: 1. None, pump to bulk FM-3160 tank
2. 1st Alternate: Green polyoverpak drum (34-7029-4109-6)
3. 2nd Alternate: Blue polyoverpak drum (34-7002-2745-6)

Label(s): Drums only: 41-2700-3160-6, Lot___, Net___, Inside

Weight Per Container: 600 lbs net (for drums)

Amount: 6,000 lbs (4,800 to bulk tank & 1,200 to drums)

Draining Temperature: 100 °F or less

Draining Pressure: 0 - 20 psig

Special Draining Instructions:

Check the volume and pressure on the bulk tank on the 1st floor readout. The volume should be less than 98%. Pressure the distilled inert mixture from the receiver to the bulk tank (15-98) with nitrogen pressure of 20 psi or as required. Watch carefully for an upper water phase near the end. Tank pressure reads out in psia. Subtract 14.7 to get normal gauge pressure. Both readings readout on gauge on 1st floor under JBC weigh tank.

Final Sample Requirements: None

Storage: Outside bulk FM-3160 tank (15-98)

BY-PRODUCT DRAINING INFORMATION: (continued)

Step: 36

Code: 41-2700-3388-3

Description: Dilute Perfluorooctanoic Acid and Inerts

Container: 1. Recycle polyoverpak drum (34-7010-1156-0)
2. 1st Alternate: New polyoverpak drum (34-7039-5732-3)
3. 2nd Alternate: Blue polyoverpak drum (34-7002-2745-6)

Label(s): 41-2700-3388-3, Lot ____, Net ____, Inside

Weight Per Container: 600 lbs net

Amount: 1,200 lbs

Draining Temperature: 100 °F or less

Draining Pressure: 0 - 20 psig

Final Sample Requirements: None

Storage: Inside

PRE-SERIES CLEAN-UP:

Hot caustic and water flush per Step 4 if required.

BETWEEN RUNS:

None required.

CLEANING AFTER LAST LOT OF SERIES:

Normally no cleaning required as FM-3256 fractionation will follow. If cleaning is required, refer to Step 4 for a hot caustic and water flush.

Product No.: 41-2700-3206-7
Revision Level: 00

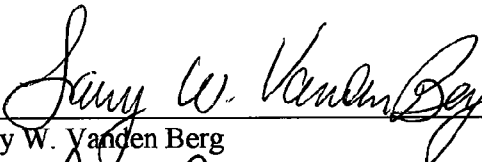
3M Restricted
Page 12 of 12

WASTE DISPOSAL:

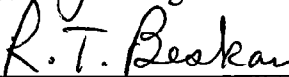
Step No. 4
Description: 5% Sodium Hydroxide in water solution
Waste Stream Code: None
Container: None
Disposition: Drain to Phase I chemical sewer
Amount: 10,000 lbs each 7th lot (if required)

Step No. 37
Description: Sulfuric acid and water solution
Waste Stream Code: None
Container: None
Disposition: Drain to Phase I chemical sewer
Amount: ~ 7,500 lbs

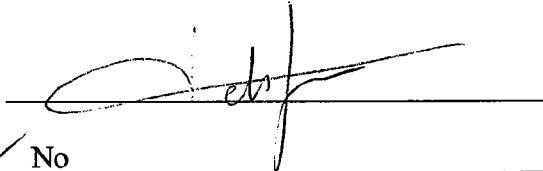
AUTHOR: Manufacturing Engineer:


Larry W. Vanden Berg

APPROVED BY: Product Chemist:


Robert T. Beskar

Product Manager/Team Leader:



Health & Safety Eng.: Yes No

Environmental Coordinator: Yes No

COPIES TO: (Reference SOP 408-007)

SMD/FP&TC, Frank W. Klink, 53-6S-02
lww\fm3206.r00

**3M COTTAGE GROVE PLANT
CHEMICAL PLANT
FACTORY OPERATING PROCEDURE**

Effective Date: July 22, 1993
Superseding: May 8, 1990

CODE NUMBER: 41-2700-3256-2

SUBJECT: FRACTIONATION OF PERFLUOROCTANOIC ACID

COMMODITY CLASS: 9428

YIELD:
41-2700-3256-2 Perfluorooctanoic Acid 7,500.0 lbs.

Estimated Machine Time: 90 Hours

EQUIPMENT:

1. Dept. 3060, BC-34 (1250 gallon Hastelloy kettle system).
2. Dept. 3060, BC-33 (1000-gallon Hastelloy kettle system) only with approval of Process Engineer.

ITEMS NEEDING SPECIAL ATTENTION:

1. FM-3206, FM-3210 and F-4169 are Hot Room charges.
2. FM-3206 should be as free of water as possible. Decant before charging, if necessary.

REASON FOR CHANGE: (Revise Product Structure? YES NO)

1. To add FM-3210 after charging and stripping FM-3206.
2. To specify recycle poly overpak drums in an attachment.
3. To add a charge calculations section.
4. Remove FM3388 as a charge.
5. Change sample size to 2 ounces.

ATTACHMENTS: Recycle drums.

CHARGE CALCULATION: 1. Charge C = 15,000 - (Charge A + Charge B)/3
2. Other charges are fixed or have no constraints.

REFERENCE:

1. 41-2700-3256-2 Factory Operating Standard, 5/8/90.
2. Meetings with Building 15 Supervisors and BC-34 Operators.
3. Conversation with T.K. Wilkinson on 7/21/93.

MFG. FOR: T. K. Wilkinson, SMD, 53-6S-02.

END PRODUCT USE: FC-118, FC-126 and FC-143 surfactants.

PROCESS TOLERANCE:

1. Unless otherwise specified, record data for process variables a minimum of once per hour.
2. Unless otherwise specified, time intervals specified are a minimum time. To obtain consistent process conditions the Operator should continue processing at the specified time interval.
3. Unless otherwise specified, maintain process variations within the tolerances listed below. If unable to operate within the acceptable tolerances, contact the Supervisor, or Process Engineer for instructions. Note all additional verbal instructions on the data card. Attach all written instructions to the Production Reports.
4. Unless otherwise specified in the procedure, use stencil weights for charging.

<u>PROCESS VARIABLES</u>	<u>ACCEPTABLE PROCESS TOLERANCES</u>	
	(+)	(-)
Agitator speed (RPM)	5	5
Temperature (°F)		
batch	5	5
jacket	5	5
head	5	5
condenser water	5	5
Pressure (psig)	5	5
Vacuum (mm Hg)	10	10
(in Hg)	0.5	0.5
Charge weight	10	10
Draining weight	10	10
Rates	Target and tolerances are specified in the procedure	

OPERATING PROCEDURE:

I. PRE-RUN PREPARATION

1. Cut in the packed column and blank off the open column.
2. Kettle, receiver and blowcase must be clean and dry. Back flush unit through the receiver, condenser and fractionating column with hot water for 3 hours. Heat the water by putting jacket in circ water with a setpoint of 200°F. Thoroughly flush blowcase and transfer line with hot water. The blowcase is to be used only for FM-3256 for the duration of the FM-3256 series. No cleanup is required if this lot follows FM-3206 or FM-3256.

SPECIAL CLEANING NOTES:

- If this run follows an FC-24 or FC-23 fractionation, boil out the system with a 20% caustic solution for 2 hours and then back flush with hot water for 2 hours.
3. Pressure test the system and obtain a leak rate less than 2 psi per hour at 40 psig.
 4. Test the vacuum control system. It must be able to control at 10 mm or less pressure before proceeding.
 5. Valve the overhead to by-pass the decanter.
 6. Open the by-pass valve in the liquid seal loop in the reflux return line. Close valves in condensate return line to the receiver. Condensate return should be routed to kettle.

II. INERT STRIP

7. Put the reactor jacket in circulating water with batch setpoint of 140°F. Turn on condenser water and set water exit temperature at 55°F.
8. Add Charge **A (41-2700-3206-7)**, Stabilized Cell Product, to the kettle by isolated vacuum. Use local exhaust ventilation to control vapors when charging. Turn on agitator and adjust speed to 70 RPM.
9. Break vacuum with nitrogen. Open kettle manhole and add Charge **B (11-0000-3179-6)** to the kettle. Close and tighten down manhole.

10. - Set the overhead for atmospheric distillation of inerts. Route condensate to return to kettle.
 - Put the jacket in "CIRC-WATER" and batch control mode.
 - Set the ΔT at 30^oF.
 - Set the batch temperature setpoint at 285^oF.
 - Set the jacket high limit at 280^oF.
 - Set the reflux timer for 5 seconds takeoff and 10 seconds reflux.
11. Distill off FM-3160 inerts to the receiver at a rate of 800-1000 lbs/hr. Adjust the ΔT setpoint to control the takeoff rate. Increasing ΔT will increase the jacket temperature and boil up rate. Decreasing ΔT will decrease jacket temperature and boil up rate.
12. Allow at least 3000 lbs. of FM-3160 distillate to collect in the receiver before draining. Drain distillate as FM-3160 to recycle polyoverpaks (34-7010-1156-0) but maintain a 1500 lbs. heel in the receiver to allow for phase separation of any water. Drain any water phase as 41-2600-6002-9 and remove.
13. As each 2400-3600 lbs. of FM-3160 is drained, add 2400-3600 lbs. of Charge **C (41-2700-3206-7)** to the kettle by isolated vacuum from the blowcase. Do not use more than 200 mm Hg vacuum while charging.
14. Continue to distill inerts to the receiver and add additional Charge C to the kettle until the kettle is full.
15. When the kettle temperature reaches 285^oF and no more Charge D remains to be charged (or the kettle is full) cool pot to 150^oF.
16. While cooling to 150^oF, add Charge **D (41-2700-3210-9)**, if scheduled, and Charge **E (11-0000-3048-3)** to the kettle by isolated vacuum. Put kettle on total reflux while pulling vacuum. Wear a lowered face shield, rubber coat and gloves when handling sulfuric acid. Use local exhaust ventilation to control vapors.
17. Drain the receiver. Drain the bottom inert phase as FM-3160. Drain the top water phase as F-6002. See By-Product Draining Information. Save 1200 lbs of FM-3160 for later use as Charge H.

III. C₈ ACID PRE-CUT

- NOTES:**
- Sample each drum of precut, intercut, maincut and post-cut as FM-3256, Dr____, using consecutive drum numbers throughout the fractionation.
 - Label each drum according to C₈ and HB content as specified on the QC page. Drums must be given consecutive drum numbers throughout the fractionation. Discard all unused labels.
 - While draining, set DP to 0 and ΔT to 10°F.
18. After adding Charge E, set the overhead for total reflux and start pulling vacuum on the kettle. Lower the vacuum to 50 mm in 50 mm increments over a 30 minute period. Vacuum must be lowered slowly to prevent boilovers.
 19. When the vacuum is 75 mm Hg or less, start heating the batch as follows:
 - Put the jacket in "CIRC WATER".
 - Set the control mode to "BATCH".
 - Set the ΔT at 30°F.
 - Set the jacket high limit at 280°F.
 - Set the splitter for 5 seconds takeoff and 25 seconds reflux but route takeoff condensate back to kettle.
 20. Raise the batch setpoint to 160°F. When the head and batch temperatures do not change for 10 minutes, start takeoff to the receiver. Leave the splitter set at 5 seconds takeoff and 25 seconds reflux. Gradually increase batch temperature as pre-cut is distilled.
 21. Gradually lower the vacuum setpoint to 10 mm Hg as precut is distilled. Adjust the ΔT setpoint to get a takeoff rate of 400-600 lbs./hr.
 22. Distill precut to the receiver until the head temperature reaches 160°F at 10 mm Hg.
 23. When the precut is complete, put the kettle on total reflux and then break vacuum on the receiver. Drain the precut to polyoverpak drums and take one, 2-ounce (in an 8-ounce bottle) in-process sample as FM-3256 lot____, Dr____ for "GLC" and "% Water". Label the sample as "SAMPLE #1".
- Label the drums based on QC results (as specified on the QC page).
24. After draining the precut, add any scheduled Charge F (41-2700-3257-0) to the receiver by isolated vacuum and then transfer to the reactor.

IV. INTERCUT & MAINCUT

- NOTES:**
- Use DP control to fractionate the inter and main cuts. The ΔT set point can be used to limit the jacket temperature and swings in steam pressure.
 - Feel temperature of reflux return line once per hour and note. This line must remain warm while in intercut and main cuts. Consult Supervisor if it cools off.
 - While draining, set DP to 0 and ΔT to 10°F.
25. Turn on steam to tracing. Turn on 120°F water to condenser. Put the receiver jacket in manual and set jacket to drain. Once the jacket is drained, set jacket to steam and valve opening to 35% (the receiver jacket should be about 130°F).
 26. Switch the reactor jacket to direct steam and the control mode to DP. Raise the jacket limit to 380°F. Maintain vacuum at 10 mm Hg.
 27. Set the splitter for 5 seconds takeoff and 25 seconds reflux. Set the ΔT setpoint at 100°F.
 28. Gradually raise the DP to 40-45 mm Hg to start reflux. Continue on total reflux until the batch and head temperatures do not change for 10 minutes.
 29. When the head and batch temperatures are stable, start takeoff to the receiver. Adjust the DP to maintain a takeoff rate of 500 - 600 lbs.hr. It may be necessary to increase the ΔT setpoint.
 30. Drain and take one, 2-ounce (in an 8-ounce bottle) in-process sample each 600 lbs. accumulation as in step 23 for "GLC" and "% Water". Label the sample as "SAMPLE #1".

When the % C₈ is 95% or greater, continue takeoff to the receiver until the head temperature is 204-206°F at 10 mm Hg or the jacket rises 20°F in one hour, whichever occurs first.
 31. When the head temperature is 204-206°F at 10 mm HG (or jacket rises 20°F), break vacuum on the receiver. Drain the receiver to new poly overpak drums. Take one 2-ounce (in an 8-ounce bottle) in-process sample for "GLC" and "% Water".

Label sample as "SAMPLE 1". Label drums according to C₈ and HB content.
 32. After draining the receiver, pull vacuum on the receiver to equalize the system.

V. FRACTIONATING AFTER THE MAIN CUT

33. IF NO CHARGE G IS ADDED, resume takeoff to the receiver. Sample and drain each 600 lbs accumulation. Take one 2-ounce (in and 8-ounce bottle) in-process sample from each draining for "GLC" and "% Water". Label the sample as "SAMPLE #1".
- Label drums according to C_8 and HB content.
 - Go to total take-off when QC results indicate C_8 content is less than 97.5% and HB content is greater than 2.5%. Go to step 37.
34. IF CHARGE G IS SCHEDULED, add Charge **G (41-2600-4169-8)** to the kettle by isolated vacuum. Switch the splitter to 5 seconds takeoff and 50 seconds reflux.
35. When the head and batch temperatures do not change for 10 minutes, resume takeoff to the receiver. Sample and drain each 600 lbs accumulation. Take one 2-ounce (in and 8-ounce bottle) in-process sample from each draining for "GLC" and "% Water". Label the sample as "SAMPLE #1".
- Label drums according to C_8 and HB content.
36. When the take-off rate falls below 100 lbs. per hour with the jacket temperature greater than 350°F, switch to total take-off to get as much C_8 /HB out of the bottoms as possible.
37. When the take-off rate falls below 30 lbs. per hour and the batch temperature rises to 350°F. at maximum vacuum, isolate the receiver and drain to poly overpak drums.

VI. FLUOROCARBON FLUSH

NOTE: If FM-3206 or FM-3256 follow, omit steps 39-43.

38. After ending the maincut, switch to circulating water and cool the batch to 200°F.
39. Isolate system at full vacuum. Add Charge **H (41-2700-3160-6)** to the receiver and transfer to the isolated kettle.
40. Break vacuum on system with nitrogen. Leave condenser water temperature at 120°F. Put 50°F water on the receiver jacket.
41. Set overhead for total take-off to receiver.
42. Distill the flush to the receiver at a rate of 400-600 lbs/hour. End distillation when take-off rate is 30 lbs./hr or less. Drain the flush to polyoverpak drums as FM-3388.

VII. DUMPING BOTTOMS

43. Turn on cooling water to the kettle and cool batch to 150°F. Open the drain valve and flush the sulfuric acid bottoms to the sewer with water.

VIII. POST RUN CLEANUP

44. - Fill kettle half full of water and heat to 150°F. Drain water hot to the sewer.
- Charge the water portion of the 10% caustic solution to the kettle.
 - Wearing rubber gloves and a face shield, vacuum charge RM-244 to the kettle with agitation. Boil system with caustic solution and drain to the sewer.
 - Water flush kettle, overhead and receiver.

CAUTION: Wear safety equipment when charging and draining RM-244 solutions.

MAINCUT DRAINING INFORMATION:

Containers: Use new blue poly overpacks (34-7002-2745-6)
 Stencilling: 41-2700-3256-2, Lot, Net___, Drum___
 Storage: Hold in Bldg. 15 Hot Room for F-7164 and F-8214.
 Filter/Alternate: None
 Weight Per Container: 600 lbs.
 Draining Temperature: 120-140°F
 Draining Pressure: 0 to 2 psi

Sample Requirements:

Inerts: 41-2700-3160-6 None
 Precut: 41-2700-3210-9 1, 2-ounce in-process sample from each drum or draining.
 Intercut: 41-2700-3257-0 1, 2-ounce in-process sample from each drum or draining.
 Maincut: 41-2700-3256-2 1, 2-ounce in-process sample from each drum or draining.
 41-2600-8281-7
 41-2600-8282-5
 Postcut: 41-2600-4169-8 1, 2-ounce in-process sample from each drum or draining.

Labelling Information For All Cuts:

<u>% C_g</u>	<u>% HB</u>	<u>Label as</u>
5.0 max.	0	41-3900-6257-2
70.0 max.	0	41-2700-3210-9
70.0-89.9	0	41-2700-3257-0
90.0-96.0	0	41-2600-8281-7
96.1-100.0	0.0-0.5	41-2700-3256-2
97.0 min.	0.6-3.0	41-2600-8282-5
Any	3.1 min.	41-2600-4169-8
5.0 max.	90-100	41-3900-6257-2

BY-PRODUCT DRAINING INFORMATION:

Step: 12,17
Description: C₈ Inerts
Labelling: 41-2700-3160-6, Lot ____, Net ____, Dr ____
Container/Disposition: Recycle poly overpak drums (34-7010-1156-0)
/Remove to warehouse
Amount: About 30,000 lbs. (600 lbs/drum)

Step: 12,17
Description: Water phase from inerts
Labelling: 41-2600-6002-9, Lot ____, Net ____, Dr ____
Container/Disposition: Recycle poly overpak drums (34-7010-1156-0)
/Warehouse
Amount: Up to 4000 lbs. (400 lbs/drum)

Step: 23
Description: Precut
Labelling: 41-2700-3210-9, Lot ____, Net ____, Dr ____
Container/Disposition: Recycle poly overpak drums (34-7010-1156-0)
or
Remanufactured poly overpak drums (34-7029-4109-6)
or
New poly overpaks (34-7002-2745-6)
/Remove to warehouse at end of series
Amount: 1800-2400 lbs (600 lbs/drum)

Step: 30
Description: Intercut
Labelling: 41-2700-3257-0, Lot ____, Net ____, Dr ____
Container/Disposition: Remanufactured poly overpak drums (34-7029-4109-6)
or
New poly overpaks (34-7002-2745-6)
/Remove to bldg 15 or bldg 3 hot room
Amount: 1200-1800 lbs. (600 lbs/drum)

BY-PRODUCT DRAINING INFORMATION: (continued)

Step: 30 & 31
Description: Fract. C₈ Acid Maincut
Labelling: 41-2600-8281-7, Lot ____, Net ____, Dr ____
Container/Disposition: Remanufactured poly overpak drums (34-7029-4109-6)
or
New poly overpaks (34-7002-2745-6)
/Remove to bldg 15 hot room.
Amount: Up to 1200 lbs. (600 lbs/drum)

Step: 34
Description: Fract. C₈ Acid Maincut
Labelling: 41-2600-8282-5, Lot ____, Net ____, Dr ____
Container/Disposition: Remanufactured poly overpak drums (34-7029-4109-6)
or
New poly overpaks (34-7002-2745-6)
/Remove to bldg 15 hot room.
Amount: Up to 1200 lbs. (600 lbs/drum)

Step: 35 & 36
Description: C₈ Acid w/C_x
Labelling: 41-2600-4169-8, Lot ____, Net ____, Dr ____
Container/Disposition: Recycle poly overpak drums (34-7010-1156-0)
or
Remanufactured poly overpak drums (34-7029-4109-6)
or
New poly overpak drums (34-7002-2745-6)
/Remove to warehouse if end of series
Amount: Up to 1200 lbs. (600 lbs/drum)

Step: 37 & 38
 Description: Pefluorooctanoic Acid in Inerts
 Labelling: 41-3900-6257-2, Lot ____, Net ____, Dr ____
 Container/Disposition: Recycle poly overpak drums (34-7010-1156-0)
 /Incinerator
 Amount: Up to 1200 lbs. (600 lbs/drum)

Step: 43
 Description: Fluorocarbon flush
 Labelling: 41-2700-3388-3, Lot ____, Net ____, Dr ____
 Container/Disposition: Recycle poly overpaks (34-7010-1156-0)
 or
 Remanufactured poly overpak drums (34-7029-4109-6)
 /Remove to warehouse at end of series
 Amount: 1200 lbs (600 lbs/drum)

WASTE DISPOSAL:

Step No.:	44	45
Description:	Sulfuric Acid Bottoms	Dilute NaOH Solution
Stencilling/Labelling:	None	None
Container/Disposition:	Drain to chemical sewer	Drain to chemical sewer
Amount:	500 lbs.	Up to 8,000 lbs.

BY-PRODUCT DISPOSAL: None.

AUTHOR:

Process Engineer

G. E. Bentz
G. E. Bentz

APPROVED BY:

Product Chemist

G. E. Bentz (for R. B. Kjome)
R. B. Kjome

Product Manager/Technical Supervisor:

J. W. Van der Beek (for Jos Vets)

Health & Safety Eng.: yes no

COPIES TO: (Reference SOP-408-008)

For Fluorochemical Products: (except Electrofluorination)

SMD/FP&TC - R. F. Henderson - 53-4N-02

SCD - Chemical Specialties/Environmental Protection Products: T. K. Wilkinson - 236-3A-04

rw/s:\FM\FM32\FM3256.428

3M COTTAGE GROVE PLANT
CHEMICAL PLANT

FACTORY OPERATING PROCEDURE

Effective Date: October 15, 1993
Superseding: March 3, 1988

CODE NUMBER: 41-2700-3108-5
COMMODITY CLASS: 9528
SUBJECT: FLUORINATION OF OCTANOYL CHLORIDE
EQUIPMENT:

Dept. 3020, BC-04, C1, one cell system.
BC-10, C3/4, two cell system.
BC-14, C5/10, six cell system.
BC-17, C11/20, ten cell system.

ITEMS NEEDING SPECIAL ATTENTION:

1. Follow all safety procedures to prevent exposure to HF, HF containing intermediates and cell electrolyte.
2. Closely monitor organic feed. Never withhold organic from the cells and never slug feed organic to the cells.
3. If recovered HF is used, use only FM-3361.
4. As a time saver, Charges C and D can be premixed in the feed tank at any time.

REASON FOR CHANGE: (Revise Product Structure? YES X NO)

1. To specify purging the system with nitrogen to prevent forming explosive mixtures with hydrogen and oxygen or OF₂.
2. To include all cell systems on this procedure.
3. To specify operating ranges.
4. To give instructions on the use of feed factors and automatic feedrate control.

REFERENCE: 41-2700-3108-5, Factory Operating Standard dated 3/2/88.

END PRODUCT USE: Manf. of Fluorad^R surfactants and Fluorinert^R brand inert

CHARGE CALCULATIONS:

1. Premix 6.4 lbs of DMDS (RM-5091) per 100 lbs of Octanoyl Chloride (RM-3194). This translates to 27 lbs RM-5091 per 420 lbs RM-3194 (1 drum)
2. Only use drums of RM-3194 that have been released by the QC Lab.

Production Rate - 8.3-8.5/10 KAH
HF usage - 8.0 lb/10 KAH
RM-3194 - 4.0 lb/10 KAH
Feedrate - 90.0% of theory

PROCESS TOLERANCE:

1. Unless otherwise specified, record data for process variables a minimum of once per hour.
2. Unless otherwise specified, time intervals specified are a minimum time. To obtain consistent process conditions the operator should continue processing at the specified time interval.
3. Unless otherwise specified, maintain process variations within the tolerances listed below. If unable to operate within the acceptable tolerances, contact the Supervisor, or Process Engineer for instruction. Note all additional verbal instructions on the data card. Attach all written instructions to the Production Reports.
4. Unless otherwise specified in the procedure, use stencil weights for charging.

<u>PROCESS VARIABLES</u>	<u>ACCEPTABLE PROCESS TOLERANCES</u>	
	(+)	(-)
Amps	500	500
Temperature (°F)		
cell	10	10
condenser	10	10
Pressure (psig)	2	2
Vacuum (mm Hg)	10	10
Charge weight	5%	5%
Draining weight	5%	5%
Rates	Target and tolerances are specified in the procedure	

OPERATING PROCEDURE:**I. PRE-RUN PREPARATION**

1. Inspect the clean packs for bent plates and check for shorts. Insert Teflon spacers if necessary.
2. Assemble the cells after they have been cleaned, dried and packs weighed. Check for shorts after re-installing the packs. Remove and repair any packs that are shorted.
3. Pressure test the system at 40 psig to check for leaks and operation of the high pressure alarms.
4. Check that all valves and pumps of the cell system are operating properly.
5. Flush the cell and overhead per the FM-3329 standard. Transfer the FM-3329 to the Discotherm and recover as FM-3361 or push to the electrolyte storage tank until the Discotherm becomes available. Repeat the flush if the initial flush comes out dirty.
6. Replace the fluid in the pump seal lubricant tank with clean, filtered refrigeration oil.
7. Add Charges C (11-0000-3194-4) and D (11-0000-5091-1) to the organic feed tank by isolated vacuum. Relieve vacuum through the bottom of the tank with nitrogen.

II. PRE-RUN PROCEDURE

9. Approximately two hours before start up time:
 - Verify the refrigeration systems for the second and third stages are at operating temperatures. Turn on cooling water to the first stage.
 - Verify the water is on to the scrubber.
10. Check the valves on the overhead before start-up. Valve the overhead so that all the condensate flows into the decanter. Close the product takeoff line to the PWT.
11. Open the gas exit valves through the catalyst tube and control valve. Set the pressure controller at 15 psig.
12. Valve the circulation piping to pump out of the surge tank and the bottoms of the cells, and into the sides of the cells. Check that the recirculating electrolyte will flow through the screen filter

13. Add Charge A (11-0000-3176-2) to the cells. Pump a slipstream for 15 minutes to fill the decanter with HF. Shut off the pump and close the slipstream line. Add additional Charge A (11-0000-3176-2) to bring levels up to "0" inches (top of the pack).

Record the amount Charge A on the Production Report. The amount will be used in the next step to calculate Charge B.

14. Calculate the amount of Charge B to add to the system using the calculation below.

CALCULATION:

Charge B = 0.0104 X Charge A (round up to 5 pound increments)

EXAMPLE:

Charge B = 0.0104 X 17,000 = 176.7 pounds
= 180 pounds (rounded up)

Add the calculated amount of Charge B (11-0000-5091-1) to the cells. Start the circulating pump(s).

15. **SAFETY:** Start nitrogen flow at a moderate rate to purge the system of any oxygen that would form an explosive mixture with hydrogen generated during cell operation. Continue to maintain the nitrogen purge to remove any OF₂ which may be generated during initial cell start up.
16. Check that the third stage refrigerant outlet temperature is colder than -80°F. Have the refrigeration mechanic adjust the system if it is not -80 °F or colder.
17. Set the rectifier for current control and then close the main breaker. Gradually raise the current setpoint to 10,000 amps over a one hour period. Raise the current slowly to control OF₂ generation.

SAFETY: Shut down the system and contact Supervisor/Process Engineer if any popping noise is heard from the overhead or if unusual OF₂ odors are identified

18. Raise the pressure setpoint to 20 psig. The voltage should not be above 5.5 volts/cell at 10,000 amps. Contact the Supervisor/Process Engineer if the voltage exceeds 5.5 volts/cell.
19. Shut off the nitrogen purge when the current reaches 10,000 amps.
20. Run in on DMDS for three hours at 10,000 amps.

21. Start feeding premixed Charges C (11-0000-3194-5) and D (11-0000-5091-1), and E (11-0000-3176-2) to the system after the three hour run in. Put the organic and HF feed loops in the RSP mode. Enter an organic factor of 4.0 lbs/10 KAH and an HF factor of 8.0 lbs/10 KAH. The controller will adjust feedrates according to amperage setpoint and number of cells on line. Check the organic tank hourly as overcharging will increase tar formation. Have Instrumentation adjust flow meter calibration so weight tank and flow meter readings agree.
22. Run at 10,000 amps for 8 hours or as directed by PE. Then gradually raise the amps and pressure to those indicated below until the maximum amperes and pressure are obtained.

<u>SYSTEM</u>	<u>AMPS</u>	<u>PRESSURE</u>
C-1	15,000(maximum)	20
	20,000(maximum with electrolyte cooler)	25
C-3/4	10,000(maximum)	20
C-5/10	15,000(maximum)	25
C-11/20	15,000	25
	20,000(maximum)	30
	20,000+(PE to approve)	30

23. Start Provox trends of pressure and first stage outlet water temperature. These trends should be level and are a good indication of how stable the system is running. Static electrolyte levels will be adjusted by reducing or increasing the HF feedrate to give steady operation before raising the current further.
24. Open the takeoff line to the PWT 24 hours after starting to feed Charges C & D.

III. STEADY STATE OPERATING PROCEDURE

25. Unless specified otherwise, follow the conditions listed below:

CURRENT - MAXIMUM AMPS FOR THE SYSTEM

PRESSURE - Pressure correlating to the maximum amps

VOLTAGE - Expect 5.5 - 6.5 volts per cell. Whenever the voltage exceeds 6.5 volts on any cell, take immediate action. If the voltage on any cell exceeds 6.0 volts, feel the bottom and side header to be sure they are warm. If the piping is not warm, circulate around the cell to dislodge any tars that may have built up. A withdrawal may also be necessary.

ORGANIC FEED RATE - 4.0 lb/10KAH per cell. Keep 40-50 psig nitrogen pressure on the organic tank to insure proper metering of the organic. Refill the organic tank with 6.4 lbs of RM-5091 per 100 lbs of RM-3194 whenever necessary and adjust % DMDS per Step 8.

NOTE: NEVER SLUG FEED ORGANIC!!!! Slug feeding will result in production of large amounts of gaseous HCl which will cause the cells to foam and boil over. It will also produce excess amounts of tar.

PRODUCT (FM-3108) TRANSFER AND SAMPLES - Transfer the PWT to the designated storage tank whenever the PWT contains 4000 lbs or more of product or when shutting down for weekends and holidays. Take one 2-ounce in-process sample in a poly bottle from each transfer and submit to the QC Lab for GLC analysis. Label 41-2700-3108-5, Lot ____, date, and the amount transferred.

SAFETY: Also attach a piece of "Danger Contains HF" warning tape to the bottle.

ELECTROLYTE (FM-3109) SAMPLING - Sample the electrolyte as requested by the cell engineer. Full rubber suit and air fed "white hats" must be worn when sampling. Fill a 4-ounce poly bottle half full of electrolyte and attach a pink in-process label and label as FM-3109, Lot ____, Date. Also attach a piece of "Danger - Contains HF" warning tape to the bottle. Immediately hand carry the sample to QC Lab, place in the refrigerator and put the Electrolyte Sample tag on the front QC bench.

(continued)

Step 25 (continued)

ELECTROLYTE LEVELS - Maintain levels at "0" inches when running below 15,000 amps. Allow levels to drop to 4 inches below the "0" mark at currents above 15,000 amps. Cycles in pressure and first stage outlet water temperature are indications the levels are too high for that amperage. Shut down and check levels daily or as directed by the cell engineer. Make up levels with fresh HF or recovered electrolyte (FM-3361).

ELECTROLYTE WITHDRAWALS - Make at least one electrolyte withdrawal per week (preferably late in the week). Additional withdrawals can be made if headers act plugged or if voltage is running above 6.5 volts on any cells. Shut down the system and pressure 200-300 lbs from each cell to the Discotherm feed tank. Additional withdrawals will be necessary if a header acts plugged or if voltage continues above 6.5 volts. Consult with the Supervisor about the need for additional withdrawals.

Recover the HF as FM-3361.

BUSS TEMPERATURES - Make sure cooling fans are on when running at 15,000 amps or greater. Check buss temperatures daily on each cell when running at 15,000 amps or greater. Use the infrared temperature camera and scan the buss assembly on each cell. Attach the printout to the cell card. Lower the current to 15,000 amps if any buss temperature is above 250 °F. Locate a portable fan and direct flow on hot buss. If this does not correct the problem the supervisor will arrange to tighten the busswork.

IV. SHUTDOWN PROCEDURE

26. When the run is terminated, stop feeding organic. The Cell Engineer will specify if and when individual cells are to be cut out and emptied. This will depend on maintenance requirements.
27. Drain down the overhead to the product weigh tank. Phase off any product present. Transfer any electrolyte present (FM-3109) to a storage tank or the Discotherm as directed by the Process Engineer. Proceed with disassembly and cleanup.
28. Drum off any remaining organic/DMDS mixture to new poly overpak drums as 41-2700-3699-3. See DRAINING INFORMATION (BY-PRODUCT): section for labeling information.

DRAINING INFORMATION(PRODUCT):

Container: Transfer directly to storage tank.

Filter: N/A

Packaging Supplies: N/A

Customer Use Labels: N/A

Label: 41-2700-3108-5

Weight per Container: N/A

Draining Temperature: Room Storage

Draining Pressure: As Required

Special Draining Instructions:

Wear rubber coat, gloves and a lowered face shield when operating valves to transfer to the storage tank and while observing the sightglass.

Update the storage tank log.

Final Sample Requirements: See Quality Report.

QC Lab: One, 1-ounce final sample in a 4-ounce poly bottle, when the product is clear in the sight glass.

Label with "HF Hazard Tape".

Wear rubber suit, rubber gloves and a face shield while taking samples. Use local exhaust ventilation.

Special Handling Instructions: None

Storage: BC-25 storage tanks.

DRAINING INFORMATION (BY-PRODUCT):

Code: 41-2700-3109-3

Description: Electrolyte

Container: None (Push to Discotherm and recover as FM-3361. If the Discotherm is not available, material may be transferred to ST #9 or ST #10.)

Packaging Suppliers: None

Label: None

Weight per Container: N/A

Amount: Varies

Draining Temperature: At cell temperature

Draining Pressure: As required

Special Draining Instructions: This material contains HF, wear appropriate protective equipment when exposure is possible.

Sample Requirements: None

Storage: None (Discotherm the material immediately. If the Discotherm is unavailable, use ST #9 or ST #10 and record on the Storage Tank log.)

DRAINING INFORMATION (BY-PRODUCT):

Code: 41-2700-3699-3

Description: Leftover Organic

Container: 41-7029-4109-6, Remfg'd poly overpack drums

Packaging Suppliers: None

Label: 41-2700-3369-3 (Order when required)

Weight per Container: 400 Lbs. Net

Amount: Varies

Draining Temperature: Room Temperature

Draining Pressure: As required

(continued)

DRAINING INFORMATION(BY-PRODUCT): (continued)

Special Draining Instructions: Transfer directly to another FM-3108
Organic Weigh Tank if possible.

Sample Requirements: None

Storage: Inside

PRE-SERIES CLEANUP: After the cells have been reassembled and pressure checked with nitrogen, conduct an HF flush.

BETWEEN RUNS: Conduct a cell turn around.

CLEANING AFTER LAST LOT OF SERIES: N/A

WASTE DISPOSAL: None

AUTHOR: Process Engineer G. E. Bentz

G. E. Bentz

APPROVED BY: Product Chemist R. T. Beskar

R. T. Beskar

Product Manager/Technical Supervisor: [Signature]

Health & Safety Eng.: yes no

COPIES TO:

FCTC - Electrochemical Fluorination: D. J. Kracht - 236-3C-89

rw/T: \RDW\FM3108.216

**3M COTTAGE GROVE PLANT
CHEMICAL PLANT**

FACTORY OPERATING PROCEDURE

Effective Date: **March 10, 1997**
Superseding: November 25, 1996

CODE NUMBER: 41-2700-3256-2 REVISION NO.: R01

SUBJECT: FRACTIONATION OF PERFLUOROOCCTANOIC ACID

YIELD:

41-2700-3256-2 Perfluorooctanoic Acid 7,500 - 10,500 lbs.

ESTIMATED MACHINE TIME: 90 - 110 Hours

EQUIPMENT:

1. Dept. 3060, BC-34 (1250 gallon Hastelloy reactor system).
2. Dept. 3060, BC-33 (1000-gallon Hastelloy reactor system) only with approval of Mfg. Engineer.

ITEMS NEEDING SPECIAL ATTENTION:

1. FM-3206, FM-3210, FM-3257, and F-4169 are Hot Room charges. Store in hot room 48 hours before changing.
2. FM-3206 should be as free of water as possible. Decant before charging, if necessary.

REASON FOR CHANGE:

(Revise Product Structure? ___ Yes x No)(New or Changed Emissions? ___ Yes x No)

1. To change from adding Charge A and Charge C from drums to charging from Storage Tank 15.
2. To update pre-run and post-run cleaning instructions and to include a caution note to not relieve pressure abruptly through the carbon packing.
3. To change instructions for draining the receiver during vacuum fractionation.
4. To update delta T, DP and reflux timer settings to current practice.

SHUTDOWN INSTRUCTIONS:

1. This run can be shutdown at any time without causing a safety or quality problem except as noted below. If the process is shut down for short periods (less than 8 hrs) the batch should be left on total reflux. This will avoid the time lost in cool down and heatup. If the fractionation is interrupted the column head temperature must be allowed to stabilize on total reflux before resuming takeoff to assure proper separation of components.

ATTACHMENTS: None

CHARGE CALCULATION:

1. Charge A = 13,600 lbs of FM-3206
2. Charge B = 100 lbs of RM-3179
3. Charge C = 25,600 lbs of FM-3206
4. Charge D = Optional Charge of FM-3210 if Specified
5. Charge E = Optional Charge of FM-3257 if Specified
6. Charge F = 500 lbs of RM-3048
7. Charge G = Optional Charge of F-4169 if Specified
8. Charge H = Optional 1200 lbs of FM-3160 for Column Flush if FM-3206 or FM-3256 does not follow.

REFERENCE:

1. 41-2700-3256-2 Factory Operating Standard, 11/25/96, Revision 0.

MFG. FOR:

1. Customer Division: SCD
2. Customer Plant Contact: NA
3. Customer Lab Contact: Marylee Maendler - 236-2A-01
4. SMD/FP&TC Contact: Dale Neuman - 53-6S-02

END PRODUCT USE:

FC-118, FC-126 and FC-143 surfactants used in teflon manufacture; FC-26 which is sold as the acid.

PROCESS TOLERANCE:

1. Unless otherwise specified, record data for the process variables as requested in the FOP a minimum of once per hour.
2. Unless otherwise specified, time intervals specified are a minimum time. To obtain consistent process conditions the Operator should continue processing at the specified time interval.
3. Unless otherwise specified, maintain process variations within the tolerances listed below. If unable to operate within the acceptable tolerances, contact the Supervisor, or Mfg. Engineer for instructions. Note all additional verbal instructions on the data card. Attach all written instructions to the Production Reports.
4. Unless otherwise specified in the procedure, use stencil weights for charging.

<u>PROCESS VARIABLES</u>	<u>ACCEPTABLE PROCESS TOLERANCES</u>		<u>RECORD</u>
	(+)	(-)	<u>Y or N</u>
Agitator speed (RPM)	5	5	Y
Temperature (°F)			
batch	5	5	Y
jacket	5	5	Y
head	2	2	Y
condenser water	5	5	Y
Pressure (psig)	5	5	Y
Column <i>D.P.</i> (mm Hg)	5	5	Y
Vacuum (mm Hg)	2	2	Y
(in Hg)	0.5	0.5	N
Charge weight	5	5	Y
Draining weight	2	2	Y
Rates	Target and tolerances are specified in the procedure.		

OPERATING PROCEDURE:

I. PRE-RUN PREPARATION

1. The packed column must be in service; blank the open column if necessary.
2. The reactor, overhead, receiver, and all transfer lines must be free from contamination of other products. No cleanup is required if this lot follows FM-3206, FM-3256, or FM-4569. If this lot follows any other lot, a hot caustic water flush of the system followed by a water rinse is required.

Caution: *When charging caustic to the reactor the minimum personal protection required is a face shield, goggles, rubber jacket and neoprene gloves.*

Vacuum charge two drums of **11-0000-0244-1**, 50% Sodium Hydroxide, to the reactor. Start the agitator at 60 rpm. Fill the reactor with water.

Caution: *When flushing the reactor and overhead system with caustic and water, do not bump material through the carbon packing. The carbon packing is very brittle and bumping may damage the packing.*

Set batch setpoint to 250 °F. the jacket high limit to 350 °F and the delta T limit to 300 °F. Set the jacket mode to direct steam and heat the water. When the reactor temperature reaches 205 - 210 °F, start adding water to the reactor at a controlled rate, maintaining a batch temperature of 200 - 210 °F. Overflow the reactor through the packed column, overhead condenser and allow to flow to the receiver. After flushing the overhead for 1 hour, cool to 130 °F. Put jacket in neutral and drain overhead lines, reactor, and receiver to the sewer. Refill the reactor with water, and repeat the flush through entire system with water for 1 hour. Drain overhead lines, receiver and reactor to the sewer.

Open the reactor and receiver manholes and inspect. Contact supervisor if additional cleanup is necessary.

3. Pressure the reactor system including overhead and receiver to 40 psig with nitrogen. The pressure loss should be less than 1 psig after 30 minutes. Find and repair leaks as required. Use water to hydrostat system if necessary to find leaks.
4. Test the vacuum control system. It must be able to control at 10 mm or less absolute pressure before proceeding.

5. Check the D.P. lines to make sure they are open and not plugged:
 - a. Put the control mode to D.P. and make sure the reactor is either on vacuum or at 0 psig and vented.
 - b. Set the jacket to Circ Water. (The conditions in 5(a) and 5(b) will open the shutoff valves to the D.P. lines. The valves will be shut under the following conditions, jacket control in neutral, Batch control with either Circ Water or Steam, and when reactor pressure exceeds 7 psig).
 - c. Go to 3rd floor and observe the small rotameters on the purge lines to the D.P. lines. There should be flow of nitrogen visible. Adjust the rotameters to midscale if necessary. Observe the rotameters for several minutes to make sure the readings are stable.
 - d. If there is no flow or if there was flow initially and the flow stopped it means the corresponding *D.P.* line is plugged. High pressure *D.P.* line means the line on the bottom of the column. Low pressure line means the line to the top of the packed column.
 - e. If there is flow through the rotameters one more test is required to determine if the lines are clear and there is not a leak in the tubing going to the *D.P.* lines. Put the jacket control in neutral to close the solenoid valves to the *D.P.* lines. Now watch the rotameters. If flow continues through the rotameters then there is a leak in the tubing. If the flow stops--(it may take a couple of minutes for the longer high pressure side *D.P.* line)--then the line is OK.
 - f. If the *D.P.* lines do not pass this test take steps to blow nitrogen or steam through the lines to clear the lines. If you cannot clear the lines call maintenance and have a fitter clean/repair the line. The most likely place for a plug is in the lower section of the line on the top of the reactor.
 - g. The D.P. control will not work if the lines are not clear.

6. Valve the overhead to by-pass the decanter. Open the by-pass valve in the liquid seal loop in the reflux return line. Close valves in condensate return line to the receiver. Condensate return should be routed to reactor for startup.

II. INERT STRIP

7. Put the reactor jacket in circulating water with batch setpoint of 120°F, a jacket high limit of 350°F and a delta T limit of 100°F. Turn on condenser water to "COOLING" and set water exit temperature setpoint at 55°F.
8. Add Charge A (41-2700-3206-7), Stabilized Cell Product, to the reactor. Use nitrogen pressure to transfer Charge A from Storage Tank 15.
9. Vent reactor to scrubber. Open reactor manhole and add Charge B (11-0000-3179-6), Filter Cel, to the reactor. Close and tighten down manhole. Turn on agitator and adjust speed to 70 RPM.
10. Set the system for distillation as follows:
 - a. Set the overhead for atmospheric distillation of inerts. Route condensate takeoff to return to reactor during heatup.
 - b. Verify the jacket is in "CIRC-WATER" and set the batch control mode to D.P.
 - c. Set the ΔT at 50°F. **Adjust** ΔT as required to maintain a 1,200-1,800 lb/hr take-off rate.
 - d. Set the **D.P.** setpoint to 100 mm. This will record the D.P., but the ΔT will be the limiting set point.
 - e. Set the jacket high limit at 350°F.
 - f. Set the reflux timers for **10** seconds **takeoff** and **5** seconds **reflux**. (Reflux ratio 0.5 to 1.) Set the splitter valve to REFLUX/TAKEOFF.
11. Distill off FM-3160 inerts to the receiver at a rate of 1,200-1,800 lbs/hr. Adjust the ΔT setpoint to maintain the takeoff rate. Increasing ΔT will increase the jacket temperature, boil up rate and take-off rate. Decreasing ΔT will decrease jacket temperature and boil up rate.

12. Allow at least 10,000 - 12,000 lbs. (4,000 lbs for BC-33), of FM-3160 distillate to collect in the receiver before draining. Verify the outside inert tank is not full (> 96% on the gauge on 1st floor), then transfer the distillate as FM-3160 to the outside inert storage tank. **Leave a 1,000 lb heel of inerts in the receiver** so the tank is not pressured up with excess nitrogen pressure. Also, there can be an upper water phase in the receiver. Water should not go to the outside inert storage tank. Watch for the water phase in the receiver near the end of the inert strip. Vent the pressure slowly through the overhead if pressure was used to transfer the inerts.

Record the amount of FM-3160 inerts drained on the Byproduct Yield Card, whether drained to the bulk tank or to drums. If the tank is full or not operational, drain from the receiver to polyoverpaks (see By-Product Draining section for suitable recycle drums) but maintain a 1,000 lbs. heel in the receiver to allow for phase separation of any water. Drain any water phase as By-Product 41-2600-6002-9 and remove.

13. As each 1,200-2,400 lbs of FM-3160 is distilled, add 1,200-2,400 lbs of Charge C (41-2700-3206-7) to the reactor. (Add what you distill). Smaller more frequent additions are preferable to large slugs.
14. Continue to distill inerts to the receiver and add additional Charge C to the reactor until the reactor is full or until storage tank 15 is empty and all scheduled FM-3206 has been added. If there is an amount of Charge D scheduled, charge after all of Charge C has been added. Add Charge D (41-2700-3210-9) by first charging to storage tank 15 followed by pressuring to the reactor.

Increase the ΔT as necessary to maintain a take off rate of **1,200-1800 lb/hr**. At rates below 1200 lb/hr, the column efficiency decreases.

When the column **head** temperature reaches **210 °F**, increase the reflux; set the reflux timers for **5** seconds **takeoff** and **10** seconds **reflux** (Reflux ratio 2:1).

Switch jacket to steam: When the batch reaches **240-250°F**, drain the jacket, and switch the jacket to "STEAM" mode.

15. When the **reactor** temperature reaches **285°F** and no more Charge C or D remains to be charged (or the reactor is full), set the jacket to CIRC-WATER and BATCH-AUTO and cool the batch to 150°F.
16. While cooling to 150°F, maintain splitter valve settings, but route distillate to return to reactor.
17. Drain the balance of the receiver. Drain the bottom inert phase as FM-3160. If the next scheduled run is **not** FM-3206 or FM-3256, save 1,200 lbs of FM-3160 for later use as Charge H. Drain the top water phase as F-6002. See By-Product Draining Information.

5

III. FIRST PRE-CUT (100 mm Hg up to 245 °F head temperature)

NOTES:

- o The first pre-cut will be taken as a total cut in the receiver and then drummed.*
 - o Sample the precut as FM-3256, Dr _____, **using consecutive drum numbers** throughout the fractionation. Do not include drums of F-6002 drained when numbering drums of pre-cut. Discard all unused labels.*
 - o Label each drum according to C8 and HB content as specified on the QC page. If the C8 content is less than 10%, the QC lab will only report the C8 value. Drums with less than 10% C8 are scrap.*
 - o While draining the receiver, maintain vacuum on reactor and column, and continue to operate column with the same reflux timer settings, routing take-off to the reactor. When draining is complete, use single stage vacuum to pull vacuum on receiver, before switching back to BC-34's vacuum on the receiver. After re-establishing the normal vacuum level and a steady head temperature, switch take-off back to receiver.*
18. While cooling to 150°F, maintain splitter valve settings, with distillate routed to reactor. Pull vacuum on reactor system for first precut. Set the vacuum loop to AUTO and lower the vacuum to **100 mm Hg** in **50 mm** increments over a 30 minute period. Vacuum must be lowered slowly to prevent boilovers.
19. When the vacuum is **100 mm Hg** or less, start heating the batch as follows:
- a. Put the jacket in "CIRC WATER".
 - b. Set the control mode to "COL DP".
 - c. Set the ΔT at 50-100°F. Increase as necessary to maintain ~~200-300~~³⁰⁰⁻⁴⁰⁰ lb/hr takeoff rate.
 - d. Set the **D.P.** setpoint to 100 mm. This will record the **D.P.**, but ΔT will be the limiting set point.
 - e. Set the jacket high limit at 350°F.
 - f. Set the splitter for **5** seconds takeoff and **20** seconds reflux (Reflux ratio 4:1) but route takeoff back to reactor to keep the line clear.

20. When the head temperature is stable for 10 minutes, start takeoff to the receiver. Leave the splitter set at 5 seconds takeoff and 20 seconds reflux. Monitor take-off rate and adjust the delta T setpoint to maintain a take-off rate of **300-400 lbs/hr**.

When the batch reaches 240-250°F, drain the jacket, and switch the jacket to "STEAM" mode.

21. Distill precut to the receiver until the head temperature reaches **245°F at 100 mm Hg**. Expect 1,500 to 3,500 lbs of distillate in the receiver for normal batches.
22. When the precut is complete, switch vacuum directly to reactor, through condenser and packed column. Continue to operate column with the same reflux timer settings, routing take-off to the reactor. Isolate receiver from reactor and break vacuum on the receiver with nitrogen. Pressure receiver to 20 psig with nitrogen.

Drain the precut to polyoverpak drums, watching for an upper water phase during draining. Take one, 2-ounce (in a 4-ounce bottle) in-process sample. Label as FM-3256 Lot___, Dr___, "**SAMPLE #1**".

If there is a water phase, drain it as byproduct 41-2600-6002-9 as in Step 12. Do not include the F-6002 byproduct as part of the Drum numbers in the fractionation.

When draining is complete, use single stage vacuum to pull maximum vacuum on receiver.

Label the drums based on QC results (as specified at the end of the Maincut Draining Information).

23. After draining the precut, add any scheduled Charge **E (41-2700-3257-0)**, 2nd Precut, to the receiver by vacuum. The maximum charge that will fit is 3,000 - 4,000 lbs.

Safety Note: *Wear a lowered face shield, goggles, rubber coat, and neoprene gloves when handling sulfuric acid. Use local exhaust ventilation to control vapors.*

Add Charge **F (11-0000-3048-3)**, Sulfuric acid to the receiver by vacuum. Pressure receiver to 20 psig and transfer to the reactor. Again use single stage vacuum system to pull maximum vacuum on the receiver. Isolate receiver from single stage vacuum system then switch back to BC-34's vacuum system on the receiver. Use delta T setpoint of 15°F while vacuum pulls down and column D.P. levels. Reset delta T setpoint back to 100°F after column D.P. levels out.

IV. SECOND PRECUT (10 mm Hg up to Product cut purity))

NOTES:

- o The second precut will be taken off in 2 drum drainings until 96% C8. When the C8 is greater than 96%, the product will be taken as one cut until near the end. The maincut should be drained before getting near the end. This will eliminate getting too many high boilers in the maincut. The key variables to watch for ending the main cut are a dropping D.P. and an increase in steam pressure.*
 - o Feel temperature of reflux return line once per hour and note. This line must remain warm while in intercut and main cuts. If a line plugs with C8 blow it out with nitrogen or melt it.*
 - o While draining the receiver, maintain vacuum on reactor and column, and continue to operate column with the same reflux timer settings, routing take-off to the reactor. When draining is complete, use single stage vacuum to pull vacuum on receiver, before switching back to BC-34's vacuum on the receiver. After re-establishing the normal vacuum level and a steady head temperature, switch take-off back to receiver.*
24. Lower the vacuum set point to **10 mm Hg**. Set the reflux splitter to **5** seconds takeoff and **30** seconds reflux (Reflux ratio 6: 1).
 25. Turn on steam to the tracing. Set condenser water setpoint to **80°F**. Put the receiver in JKT-MAN and set to WARM-WATER. Set jacket valve position to 50-70% to heat the receiver jacket to about 130°F. Monitor receiver jacket temperature hourly to verify that the temperature is holding close to 130°F.
 26. Verify reactor jacket is set to DIRECT STEAM and the control mode to D.P.. Raise the jacket high limit to 380°F. Set the ΔT setpoint at 100°F.
 27. Adjust the D.P. to 50-80 mm Hg to establish a steady and strong take-off. Maintain take-off to the reactor until the head temperature is stable for 10 minutes.
 28. When the head temperature is stable, start take-off to the receiver. Raise the condenser temperature slowly throughout the intercut until the condenser temperature reaches 120 °F.

Condenser water note:

Raising the condenser water temperature too fast may make it impossible to attain the desired 10 mm Hg vacuum. A good guide to raising the condenser water temperature is: Condenser water temperature should be no higher than (Column head temperature - 90 °F). For example, if the head temperature is 190 °F, the condenser water temperature should not be higher than 100 °F.

29. Continue the second precut distillation to the receiver. Monitor take-off rate and adjust the column D.P. setpoint to maintain a take-off rate of **400-500 lbs/hr**.

Drain the receiver every two drums per instructions below until you reach 96% purity.

Switch vacuum directly to reactor, through condenser and packed column. Continue to operate column with the same reflux timer settings, routing take-off to the reactor. Isolate receiver from reactor and break vacuum on the receiver with nitrogen. Pressure receiver to 20 psig with nitrogen.

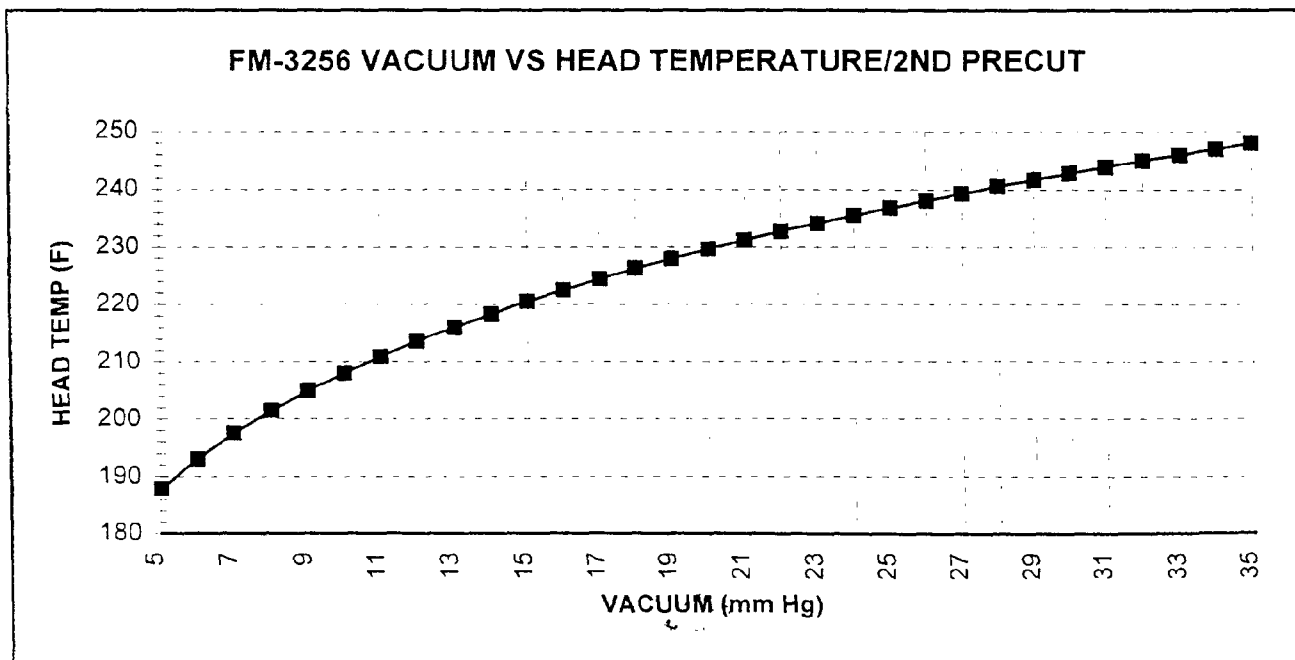
Drain the precut to **new** polyoverpak drums. Take one, 2-ounce (in a 4-ounce bottle) in-process sample. Label as FM-3256 Lot ___, Dr ___, "**SAMPLE #1**".

When draining is complete, use single stage vacuum to pull maximum vacuum on receiver. Isolate receiver from single stage vacuum system then switch back to BC-34's vacuum system on the receiver. Use delta T setpoint of 15°F while vacuum pulls down and column D.P. levels. Reset delta T setpoint back to 100°F after column D.P. levels out.

Label the drums based on QC results (as specified at the end of the Maincut Draining Information).

When the % C8 of the last draining is 96% or greater, proceed to Product Cut.

The following graph shows the head temperature of high purity C8 versus the vacuum. If the insulation on the column vapor line is good this line will approximate the head temperature at the end of the 2nd precut and beginning of the product cut.



V. PRODUCT CUT

NOTES:

- o The product cut will be taken as a total cut to the receiver and then drummed. **However, do not wait so long in draining that you get HB into the maincut. It is imperative that the operator monitor the jacket steam pressure and the D.P. to determine the first sign of the end of the maincut. End the maincut as soon as any one of these signals the end of the cut.***
 - o Use D.P. control to fractionate the inter and main cuts. The ΔT set point can be used to limit the jacket temperature and swings in steam pressure.*
 - o Feel temperature of reflux return line once per hour and note if it is cooled. If a line plugs with C8 blow it out with nitrogen or melt it.*
 - o While draining the receiver, maintain vacuum on reactor and column, and continue to operate column with the same reflux timer settings, routing take-off to the reactor. When draining is complete, use single stage vacuum to pull vacuum on receiver, before switching back to BC-34's vacuum on the receiver. After re-establishing the normal vacuum level and a steady head temperature, switch take-off back to receiver.*
30. Set the splitter for 5 seconds takeoff and 30 seconds reflux. (Reflux ratio 6:1). Set the ΔT setpoint at 100°F.
31. Adjust the D.P. to 50-80 mm Hg to establish a steady and strong take-off. Maintain take-off to the reactor until the head temperature is stable for 10 minutes.
32. When the head temperature is stable, start take-off to the receiver. The condenser temperature should remain at 120 °F.

33. To maintain the proper take-off rate (and good fractionation) it will be necessary to make adjustments in the *D.P.* setpoint. Maintain a product takeoff rate of **400-500 lb/hr**. Rate will drop below this near the end of the cut.

Continue the product cut distillation to the receiver until:

- a. The jacket steam pressure rises sharply and remains above 90 psig, or
- b. The jacket temperature rises to 300°F, or
- c. The *D.P.* drops off from its steady state value.

Any one of these three items can signal the end of the large product cut where you should drain the receiver.

When the receiver requires draining, switch vacuum directly to reactor, through condenser and packed column. Continue to operate column with the same reflux timer settings, routing take-off to the reactor. Isolate receiver from reactor and break vacuum on the receiver with nitrogen. Pressure receiver to 20 psig with nitrogen.

Drain the product cut to **new** polyoverpak drums. Take one, 2-ounce (in a 4-ounce bottle) in-process sample. Label as FM-3256 Lot____, Dr____, "**SAMPLE #1**".

When draining is complete, use single stage vacuum to pull maximum vacuum on receiver. Isolate receiver from single stage vacuum system then switch back to BC-34's vacuum system on the receiver. Use delta T setpoint of 15°F while vacuum pulls down and column *D.P.* levels. Reset delta T setpoint back to 100°F after column *D.P.* levels out.

34. Continue on product cut fractionation, draining in two drum increments until the high boilers in the product exceeds 3.0%. Drain receiver per step 33.

When the high boilers in the product exceeds 3%, proceed as follows. If an amount of Charge G is specified on the run card, proceed to section **VI. FIRST POST CUT**. Otherwise, proceed to section **VII. SECOND POST CUT (TOTAL TAKEOFF)**.

VI. FIRST POST CUT

NOTES:

- o This cut is only done if F-4169 is added to the reactor. Normally you will continue on maincut until end of the maincut is signalled in Step 34 and then go to total takeoff per step 40.*
 - o Feel temperature of reflux return line once per hour and note if not hot. Clear the line with nitrogen or by melting if necessary.*
 - o The first post cut will be taken to the receiver and drummed as each **600 lb** is collected.*
 - o While draining the receiver, maintain vacuum on reactor and column, and continue to operate column with the same reflux timer settings, routing take-off to the reactor. When draining is complete, use single stage vacuum to pull vacuum on receiver, before switching back to BC-34's vacuum on the receiver. After re-establishing the normal vacuum level and a steady head temperature, switch take-off back to receiver.*
35. Isolate the receiver from the reactor and switch vacuum to reactor overhead. Maintain splitter valve settings with distillate routed to the reactor. Add any scheduled Charge G (41-2700-4169-0), Postcut, to the receiver by vacuum. Pressure receiver to 20 psig and transfer to the reactor. Use single stage vacuum system to pull maximum vacuum on the receiver. Isolate receiver from single stage vacuum system then switch back to BC-34's vacuum system on the receiver. Use delta T setpoint of 15°F while vacuum pulls down and column D.P. levels. Reset delta T setpoint back to 100°F after column D.P. levels out.
36. Set the splitter for **5** seconds takeoff and **30** seconds reflux. (Reflux ratio 6:1). Set the ΔT setpoint at 100°F.
37. Adjust the *D.P.* to 50-80 mm Hg to establish a steady and strong take-off. Maintain take-off to the reactor until the head temperature is stable for 10 minutes.
38. When the head temperature is stable, start take-off to the receiver. Maintain the condenser temperature at 120 °F.

39. To maintain the proper take-off rate (and good fractionation) it will be necessary to make adjustments in the *D.P.* setpoint. Maintain a product takeoff rate of **275-350 lb/hr**. Rate will drop below this near the end of the cut.

When 600 lbs of distillate has been collected in the receiver, switch vacuum directly to reactor, through condenser and packed column. Continue to operate column with the same reflux timer settings, routing take-off to the reactor. Isolate receiver from reactor and break vacuum on the receiver with nitrogen. Pressure receiver to 20 psig with nitrogen.

Drain the product to **new** polyoverpak drums. Take one, 2-ounce (in a 4-ounce bottle) in-process sample. Label as FM-3256 Lot___, Dr___, "**SAMPLE #1**".

When draining is complete, use single stage vacuum to pull maximum vacuum on receiver. Isolate receiver from single stage vacuum system then switch back to BC-34's vacuum system on the receiver. Use delta T setpoint of 15°F while vacuum pulls down and column *D.P.* levels. Reset delta T setpoint back to 100°F after column *D.P.* levels out.

40. Continue fractionation, draining in one drum increments until the high boilers in the product exceeds 3.0%. Drain receiver per step 39.

When the high boilers in the product exceeds 3%, proceed to section **VII. SECOND POST CUT (TOTAL TAKEOFF)**.



VII. SECOND POST CUT (TOTAL TAKEOFF)

NOTES

- o The second post cut cut will be taken as a total cut to the receiver and then drummed.*
 - o Feel temperature of reflux return line once per hour and note if not hot. Clear the line with nitrogen or by melting if necessary.*
 - o While draining the receiver, maintain vacuum on reactor and column, and continue to operate column with the same reflux timer settings, routing take-off to the reactor. When draining is complete, use single stage vacuum to pull vacuum on receiver, before switching back to BC-34's vacuum on the receiver. After re-establishing the normal vacuum level and a steady head temperature, switch take-off back to receiver.*
41. Set the vacuum to 1 mm to get maximum vacuum. Set the splitter for total takeoff. Set the ΔT setpoint at **100°F**.
 42. Adjust the *D.P.* to 50-80 mm Hg to establish a steady take-off. Maintain take-off to the reactor until the head temperature is stable for 10 minutes.
 43. When the head temperature is stable, start take-off to the receiver. Maintain the condenser temperature at 120 °F.
 44. Distill to get as much C8 Acid/HB out of the bottoms as possible. When the take-off rate falls below 30 lbs per hour and the batch temperature rises to 350°F with maximum vacuum, stop the distillation. Isolate receiver from reactor and break vacuum on the receiver with nitrogen. Pressure receiver to 20 psig with nitrogen.

Drain the product to polyoverpak drums. Take one, 2-ounce (in a 4-ounce bottle) in-process sample. Label as FM-3256 Lot ____, Dr ____, "**SAMPLE #1**".

VIII. FLUOROCARBON FLUSH

NOTE: Omit steps 45-49 if FM-3206 or FM-3256 follows this lot.

45. After ending the postcut, switch jacket to circulating water, batch control mode to batch auto, and start cooling the batch to 200°F.
46. Isolate system at full vacuum. Add Charge H (41-2700-3160-6) to the receiver and pressure transfer to the isolated reactor.
47. Break vacuum on system with nitrogen. Leave condenser water temperature at 120°F. Put 50°F water on the receiver jacket. Open receiver vent to scrubber.
48. Set overhead valving for total take-off to receiver. Set jacket high limit to 350°F, and delta T limit to 50°F.
49. Distill the flush to the receiver at a rate of 400-600 lbs/hour. End distillation when take-off rate is 30 lbs/hr or less. Drain the flush to polyoverpak drums as FM-3388.

IX. DRAINING BOTTOMS

50. Turn on cooling water to the reactor and cool batch to 150°F. Break vacuum with nitrogen and pressure to 10 psig. When the temperature has dropped to 200 °F, slowly add water to the reactor. At first, it will exotherm, then will cool. Fill reactor half full with water. Open the drain valve and flush the sulfuric acid bottoms to the sewer with water.

XI. POST RUN CLEANUP

NOTE: No cleanup is necessary if a FM-3206 or FM-3256 is to follow.

51. **Caution:** *When charging caustic to the reactor the minimum personal protection required is a face shield, goggles, rubber jacket and neoprene gloves.*

Vacuum charge two drums of 11-0000-0244-1, 50% Sodium Hydroxide, to the reactor. Start the agitator at 60 rpm. Fill the reactor with water.

Caution: *When flushing the reactor and overhead system with caustic and water, do not bump material through the carbon packing. The carbon packing is very brittle and bumping may damage the packing.*

Set batch setpoint to 250 °F, the jacket high limit to 350 °F and the delta T limit to 300 °F. Set the jacket mode to direct steam and heat the water. When the reactor temperature reaches 205 - 210 °F, start adding water to the reactor at a controlled rate, maintaining a batch temperature of 200 - 210 °F. Overflow the reactor through the packed column, overhead condenser and allow to flow to the receiver. After flushing the overhead for 1 hour, cool to 130 °F. Put jacket in neutral and drain overhead lines, reactor, and receiver to the sewer. Refill the reactor with water, and repeat the flush through entire system with water for 1 hour. Drain overhead lines, receiver and reactor to the sewer.

Open the reactor and receiver manholes and inspect. Contact supervisor if additional cleanup is necessary.

MAINCUT DRAINING INFORMATION:

Containers: New black poly overpacks (34-7039-5732-3)

Labeling: 41-2700-3256-2, Lot, ___Net___, Drum___, Inside

Storage: Hold in Bldg. 15 Hot Room for F-7164.

Filter/Alternate: None

Weight Per Container: 600 lbs.

Draining Temperature: 120-140°F

Draining Pressure: 0 to 2 psig

Special Draining Instructions:

To avoid C8 set up in the drain line, blow the drain line clear from the bottom drain valve to the draining drum. Then suck through the drain line to the receiver briefly before shutting drain valve.

Sample Requirements:

Inerts: 41-2700-3160-6 None

Precut: 41-2700-3210-9 1. 2-ounce in-process sample from each drum or draining.

Intercut: 41-2700-3257-0 1. 2-ounce in-process sample from each drum or draining.

Maincut: 41-2700-3256-2 1. 2-ounce in-process sample from each 41-2600-8281-7 or 41-2600-8282-5 drum or draining.

Postcut: 41-2600-4169-8 1. 2-ounce in-process sample from each drum or draining.

Labeling Information For All Cuts:

<u>% C8</u>	<u>% HB</u>	<u>Label as:</u>	<u>Description:</u>
0.0 - 9.9	0	13-0017-0281-3	Scrap
10.0 - 69.9	0	41-2700-3210-9	First Precut
70.0 - 89.9	0	41-2700-3257-0	Second Precut
<u>Product Cuts</u>			
90.0 - 96.0	0	41-2600-8281-7	Product Cut
96.1 - 100.0	0.0 - 0.5	41-2700-3256-2	Product Cut (Heart cut)
97.0 - 99.4	0.6 - 3.0	41-2600-8282-5	Product Cut
<u>Postcut</u>			
10.0 - 96.9	3.1 - 89.9	41-2600-4169-8	Postcut
0.0 - 10.0	90-100	13-0017-0281-3	Scrap

BY-PRODUCT DRAINING INFORMATION:

Step: 12, 17

Description: C₈ Cyclic Inert Mixture

Labeling: Outside inert tank: None (Record byproduct yield on production card).
Drums: 41-2700-3160-6, Lot ____, Net ____, Dr ____, Inside.

Wt per Container: 600 lbs net for drums

Container/Disposition: Pump/pressure to outside inert tank. If drummed use new or remanufactured poly overpak drums (Refer to list of suitable recycle drums at end of ByProduct Section). Remove drums to warehouse.

Amount: 20,000 - 30,000 lbs.

Step: 12, 17

Description: Water phase from inerts

Labeling: 41-2600-6002-9, Lot ____, Net ____, Dr ____, Inside.

Wt per Container: 400 lbs net.

Container/Disposition: 1. Recycle poly overpak drums (34-7010-1156-0) (Refer to list of suitable recycle drums at end of ByProduct Section).
2. Remanufactured poly overpak drums (34-7029-4109-6)
3. New poly overpaks (34-7002-2745-6)
/Remove to warehouse at end of series

Amount: Up to 4,000 lbs.

BY-PRODUCT DRAINING INFORMATION: (continued)

Step: 22

Description: First Precut

Labeling: 41-2700-3210-9, Lot ____, Net ____, Dr ____

Wt per Container: 600 lbs net

Container/Disposition: 1. Recycle poly overpak drums (34-7010-1156-0) (Refer to list of suitable recycle drums at end of ByProduct Section).
2. New black poly overpak drums (34-7039-5732-3)
/Remove to warehouse at end of series

Amount: 1,800-2,400 lbs

Step: 29

Description: Second Precut

Labeling: 41-2700-3257-0, Lot ____, Net ____, Dr ____, Inside

Wt per Container: 600 lbs net

Container/Disposition: 1. New Black Polyoverpaks (34-7039-5732-3)
2. Used FM-3256 drums
3. New poly overpaks (34-7002-2745-6)
/Remove to bldg 15 hot room.

Amount: 1,200-1,800 lbs.

BY-PRODUCT DRAINING INFORMATION: (continued)

Step: 33, 39
Description: First post cut
Labeling: 41-2600-8282-5, Lot ____, Net ____, Dr ____, Inside
Wt per Container: 600 lbs net
Container/Disposition: 1. New Black Polyoverpak drums (34-7039-5732-3)
2. Used FM-3256 poly overpak drums
2. New poly overpaks (34-7002-2745-6)
/Remove to bldg 15 hot room.
Amount: Up to 1,200 lbs.

Step: 44
Description: Second Postcut
Labeling: 41-2600-4169-8, Lot ____, Net ____, Dr ____, Inside.
Wt per Container: 600 lbs net
Container/Disposition: 1. Recycle poly overpak drums (34-7010-1156-0) (Refer to list of suitable
recycle drums at end of ByProduct Section).
2. New poly overpaks (34-7039-5732-3)
/Remove to warehouse at end of series.
Amount: Up to 1,200 lbs.

BY-PRODUCT DRAINING INFORMATION: (continued)

Step: 22 or 44

Description: Perfluorooctanoic Acid (< 10% C8) in Inerts for Scrap Disposal

Labeling: 13-0017-0281-3, (Refer to Waste Stream Profile)

Wt per Container: 600 lbs net

Container/Disposition: 1. Recycle poly overpak drums (34-7010-1156-0) (Refer to list of suitable recycle drums at end of ByProduct Section).
2. Remanufactured poly overpak drums (34-7029-4109-6)
3. New poly overpaks (34-7002-2745-6)/Incinerator

Amount: Up to 1,200 lbs.

Step: 49

Description: Fluorocarbon flush

Labeling: 41-2700-3388-3, Lot ____, Net ____, Dr ____, Inside.

Wt per Container: 600 lbs net

Container/Disposition: 1. Recycle poly overpak drums (34-7010-1156-0) (Refer to list of suitable recycle drums at end of ByProduct Section).
2. Remanufactured poly overpak drums (34-7029-4109-6)
3. New poly overpaks (34-7002-2745-6)
/Incinerator

Amount: 1,200 lbs

RECYCLE DRUMS FOR BY-PRODUCTS

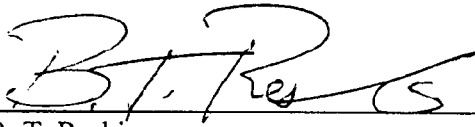
BY-PRODUCT FM-3160 (if drummed)	FOR FM-3206 FM-3210, F-4169 or Scrap
USE DRUMS FROM LIST BELOW	USE DRUMS FROM LIST BELOW
1st Choice - New Drums	
2nd Choice - Remanufactured Drums	FM-3206
3rd Choice from list below	FM-3210
FM-3129	FM-3256
FM-3719	FM-3257
FM-3160	F-4169
FM-3144	F-8281
F-6566	F-8282
F-6567	F-8420--> F-8424

WASTE DISPOSAL:

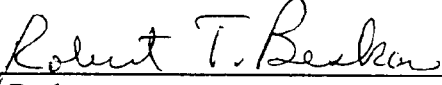
Step No.: **50**
 Description: Sulfuric Acid Bottoms
 Stenciling/Labeling: None
 Container/Disposition: Drain to chemical sewer
 Amount: 500 lbs.

Step No.: **51**
 Description: Dilute Sodium Hydroxide Solution
 Stenciling/Labeling: None
 Container/Disposition: Drain to chemical sewer
 Amount: Up to 8,000 lbs.

AUTHOR:

Manufacturing Engineer: 
B. T. Reski

APPROVED BY:

Quality Representative: 
R. T. Beskar

Product Manager\Technical Supervisor: 

Health & Safety Eng.: Yes ___ No x

Environmental Coordinator: ___ Yes x No

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Copies:
G. A. Groeneveld - 208-1C-01

**3M COTTAGE GROVE PLANT
CHEMICAL PLANT**

FACTORY OPERATING PROCEDURE

Effective Date: **April 29, 1998**
Superseding: **November 17, 1997**

CODE NUMBER: 41-2600-7164-6 **REVISION NO.:** R02

SUBJECT: ONE-PLATED PERFLUORO-OCTANOIC ACID

YIELD: 2850 Lbs

ESTIMATED MACHINE TIME: 30 Hours

EQUIPMENT:

1. BC-32, Dept. 3060, 300-gallon Hastelloy kettle with hastelloy overhead and 200 gallon glass-lined receiver.

ATTACHMENTS:

1. BC 32 Reactor Flowsheet
2. Automatic Distillation Operation for BC -32
3. PPE requirements for the F-7164 process.
4. Safe operating limits for the F-7164 process in BC -32

ITEMS NEEDING SPECIAL INSTRUCTIONS:

1. Distillation rates over 600 lbs/hr should be considered bump-overs and redistillation must be conducted.
2. Bottoms must be drained per Waste Disposal section. Do not drain to chemical sewer if bottoms contain dichromate from last one-plate distillation.
3. Do Not boil DI water through the glass-lined receiver. This may cause premature glass failure. Follow the cleanup instructions included in the cleanup section.
4. In order to help prevent vacuum system plugs, do not vacuum charge Dichromate (charge D) or Filter cell (charge C). These charges should be slowly added through the BC 32 sight glass and mixed into the batch before restarting the vacuum system.

REASON & DOCUMENTATION OF CHANGE:

(Revise Product Structure? ___ Yes X No); (New or Changed Emissions? ___ Yes X No)

MOC Required: X Yes ___ No: (Reference SOP 400-012)

Review Team required (Job Function):

- Operations: Rick Pechacek
- Engineering
- Maintenance
- Process Engineering - Dean Graham
- Safety/Hygiene - Gerri Mirkin
- Other

1. Description of Change:
Clarify the charging section with respect to adding C8 powder and sulfuric acid and to specify adding sulfuric acid immediately following the liquid C8 acid charge. The sulfuric acid will flush the line out and will prevent the charge line from plugging with frozen C8 acid. The charge letters have been changed to reflect the charge order. No negative consequences are foreseen as a result of this change.

2. Description of Change:
To specify to close the receiver drain valve and to use a vacuum level of 100 mm Hg during vacuum charging C8 acid and sulfuric acid. Vacuum charging these materials with lower vacuum levels with the receiver drain valve open can contribute to plugging the vacuum ejectors. Continuous vacuum is used to prevent the possibility of losing vacuum to during charging.

REFERENCE:

1. 41-2600-7164-6, Factory Operating Procedures dated 3/1/96 and 11/17/97.
2. Meetings with Building 15 Supervisors and BC-32 Operators.

END PRODUCT USE: F-7164 is an intermediate in the production of ammonium salt surfactants/emulsifiers. These surfactants/emulsifiers are sold to external companies, and are used as additives in the production of fluorochemical polymers such as Teflon.

CHARGE CALCULATIONS:

1. **Charge A1-A7:** Maximum Charge A is 3000 Lbs.

The composite of Charge A must meet the following requirements:

C8 Acid:	96.5% Minimum	Target 98%
C7 Acid:	1.4% Maximum	
C6 Acid:	0.4% Maximum	
High Boilers:	0.5% Maximum	

2. **Charge A8-A9:** Spray dried C8 powder charges FC143

3. **Charge B1:** Standard Charge B is 440 Lbs of RM-2706.

If %H₂O in Charge A exceeds 1%, increase amount of Charge B as follows:

$$\text{Additional Charge B} = (0.00334) \times (\% \text{H}_2\text{O in Charge A}) \times (\text{Charge A})$$

4. **Charge B2:** Additional acid for C8 powder charges, A8 and A9 FC143

If powder charges A8 or A9 are added, Charge B2 is calculated as follows:

$$\text{Charge B2} = (0.440) \times (\text{Charge A8} + \text{Charge A9 powder charges})$$

5. **Charge C:** Standard Charge is 20 lbs of RM-3179.

6. **Charge D:** Standard Charge is 60 Lbs of RM-8510.

7. Manufacturing Engineer will provide spreadsheet printout with charge calculations and quantities.

PROCESS TOLERANCE:

1. Unless otherwise specified, record data for the process variables listed below a minimum of once per hour.
2. Unless otherwise specified, time intervals specified are a minimum time. To obtain consistent process conditions the Operator should continue processing at the specified time interval.
3. Unless otherwise specified, maintain process variations within the tolerances listed below. If unable to operate within the acceptable tolerances, contact the Supervisor or Manufacturing Engineer for instructions. Note all additional verbal instructions on the data card.

Attach all written instructions to the Production Report.

4. Unless otherwise specified in the procedure, use stencil weights for charging.

	<u>ACCEPTABLE PROCESS TOLERANCES</u>		<u>RECORD DATA</u>	
	(+)	(-)	(Y)	(N)
Agitator Speed (RPM)	5	5	Y	
Temperature (°F)				
batch	5	5	Y	
jacket	5	5	Y	
head	5	5	Y	
condenser water outlet	5	5	Y	
Pressure (psig)	5	5	Y	
Vacuum				
(mm Hg)	10	10	Y	
Charge Weight	1%	1%	Y	
Draining Weight	1%	0%	Y	

Rates

Target and tolerances are specified in the procedure.

EMERGENCY SHUTDOWN INSTRUCTIONS:

1. The process (reactor, receiver, and agitators) can be shutdown any time during the operation of the equipment by turning off the agitator; shut off vacuum system, put the receiver and reactor jackets in neutral, and completely isolate the system.
2. Once confirming that the acid in the receiver or reactor is melted, the system can be restarted at any time by following the remainder of the FOP. If shut down for more than 4hrs and the temperature in the receiver has fallen below 120 degrees F, contact manufacturing engineer prior to restarting the operation.

*if the condenser water outlet temp is less than 120°F then the receiver temp will be less than 120°F also
6/2/98 DV*

Note: Once the jackets on the receiver and reactor are put into neutral any acid contained in these vessels will begin to solidify and harden. Remelting the acid may take up to a couple of days. Confirming that the acid is melted prior to starting up the agitator is essential to prevent agitator damage.

OPERATING PROCEDURE:

I. PRE-SERIES PREPARATION

1. The operator will need to be familiar with the Provox CRT and how it is used to operate BC-32. If questions exist contact the Supervisor.
2. A general flowsheet of the BC-32 process piping is attached. The operator should review it and use it as reference during the remainder of this standard.
3. The open column will be used for this run. If not already done, valve in the open column and blank off both top and bottom of packed column.
4. Reactor, overhead condenser, receiver, and transfer lines normally do not require cleaning since this unit is dedicated to the distillation of C8 acid. However, a clean up is needed following an F-7117 run. If additional cleaning is required it will be requested by the Manufacturing Engineer. See instructions in PRE-SERIES CLEANUP section in the back of this standard.

II. CHARGING

5. Verify the steam tracing to overhead piping and to BC-32 drain lines is hot . The steam supply valve for overhead tracing is located directly behind the overhead condenser. The steam supply valve for the BC-32 drain line is located on first floor against the wall underneath the BC-32 reactor. Verify the steam pressure is at 7 to 10 psig.
6. Close manual valve for liquid trap in reflux return line. Open all manual valves in takeoff line leading to the receiver. Open manual valve, in the vacuum line, on top of the receiver located between receiver and automatic vacuum and vent valves. Using the CRT, close the vent valve on large receiver. Using the CRT, open the vacuum valve on large receiver. Using the CRT close the cold side vent and vacuum block valve on the overhead condenser. Verify BC-36 and BC-37 are isolated from the BC-32 vacuum system. Using the CRT, close the receiver drain valve during charging.
7. Verify both water and steam to the shell of the overhead condenser are on and adjust setpoint for condenser water to 130°F.
8. Bring up the provox CRT BC32OH screen and put the receiver jacket in "condwatr" position. Using the same CRT screen verify automatic valve #14 is in the closed position. This allows the heated water from the outlet of the overhead condenser to flow through the receiver jacket.
9. Verify that the splitter valve is in the correct position to fill the receiver. This valve is the three-way valve located at the product exit side of the condenser. **The splitter valve is no longer an automatic valve.**
10. *open provox controlled vacuum valve, screen #3 DSV #13*
Adjust vacuum set point to 100 mm Hg using stage three only (Using the Auto Stage setting will initiate all three stages). If stage three produces insufficient vacuum at any time during the operation, then change the vacuum status to stages 2-3 and then 1-3 or auto stage as needed to provide adequate vacuum levels. Verify vacuum block valve on the receiver is open and manual vent valve on top of BC-32 reactor is closed in preparation for vacuum charging.

Charging liquid C8 (Charges A1-A7) and Sulfuric Acid (Charge B1)

11. Wear butyl rubber gloves, rubber jacket, rubber pants, rubber boots, face shield, safety goggles, and position local exhaust ventilation at the bungs while adding Charges A1-A7 (C8 acid) and Charge B1 (Sulfuric acid) as shown in Attachment 3, PPE requirements. Add Charges A1-A7 (**liquid Perfluorooctanoic Acid**), into the reactor through the bottom drain line by vacuum (see step 10) using the dedicated 1" charge hose. Use continuous vacuum pulled through the open column, the condenser, and the receiver while adding Charge A. Verify that the receiver drain valve is closed during charging. Refer to Attachment 3 for PPE requirements when handling Charges A1-A7 (FM-3256, F-8281, and F8282)

12. Refer to Attachment 3 for PPE requirements when handling Charge B1 (RM-2706). Position local exhaust ventilation at the drum bungs while charging. Using vacuum (see step 10) add Charge B1 (11-0000-2706-7), Sulfuric Acid, to the reactor through the bottom drain line. If charges A8 and A9 are included for this run then add ½ of the total of charges B1 + B2.

Adding Charge B, Sulfuric acid, immediately after adding Charges A1-A7 serves to flush the dedicated 1" charge line. When finished charging, disconnect the 1" charge line and flush with water to the sewer.

Charging powder F-6514 (Charges A8-A9) and Sulfuric Acid (Charge B2)

NOTE: When charging F-6514 powder by vacuum, charge slowly and pull vacuum through the large receiver using stage 3 (see step 10). This will reduce the amount of powder reaching the jets and causing plugging problems. Manhole charging is acceptable. This is usually easier for fiber cartons with poly bags. Be sure to use local exhaust when charging through manhole.

13. If Charges A8 and A9 are not included in this series, skip directly to step 14. Add powder Charges A8-A9 (**Ammonium Perfluorooctanoate F-6514**) after the liquid Charge A is added and after adding half of Charges B1 + B2 per step 12 (This is the sum of B1 + B2 Sulfuric Acid). (The balance of Charge B1 and B2 serves as a charge line flush and helps prevent the charge hose from plugging.) Verify the receiver drain valve is closed during charging. Add the powder Charges A8 and A9 at a slow rate (about 150 lbs/30 minutes) so the salts have an opportunity to mix into the batch. Refer to Attachment 3 for PPE requirements when handling Charge A powder (F-6514). Gloves should be washed thoroughly after powder handling, before removing.

Charging Filter Cell (Charge C) and Potassium Dichromate (Charge D)

14. With the vacuum system off (break vacuum using nitrogen) and with the receiver drain valve closed, slowly add Charge C (11-0000-3179-6), Filter Cell, to reactor through sight glass. Wear a dust mask and safety glasses at a minimum, and position local exhaust ventilation when handling Charge C. Refer to Attachment 3 for PPE requirements for handling Charge C (RM-3179)
15. With the vacuum system off (break vacuum using nitrogen) and with the receiver drain valve closed, add Charge D (11-0000-8510-7), Potassium Dichromate, slowly through the sight glass. Refer to Attachment 3 when handling Charge D (RM-8510)
16. Put reactor in "batch" control and jacket in circulating water position. Set batch set point at 165°F, jacket hi-limit at 300°F, delta-t at 350°F, and agitator at 75 rpm. For Automatic control, refer to Attachment 2, Automatic Distillation Operation of BC-32.

**3M COTTAGE GROVE PLANT
CHEMICAL PLANT**

FACTORY OPERATING PROCEDURE

Effective Date: **June 16, 1997**
Superseding: **March 3, 1997**

CODE NUMBER: 41-2700-3206-7 REVISION NO.: R03

SUBJECT: STABILIZATION OF PERFLUOROOCCTANOIC ACID IN INERTS

YIELD:

41-2700-3206-7	Stabilized Perfluorooctanoic Acid	5,800 lbs (BC-34) 4,830 lbs (BC-45) (48.3% of FM-3108 input)
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ESTIMATED MACHINE TIME: 24.0 Hours

EQUIPMENT:

1. BC-34; Dept 3060, 1,250 gallon Hastelloy reactor with packed or open column, 1,000 gallon Monel receiver, 1250 gallon Hastelloy receiver, and ST-15, 3,000 gal storage tank for FM-3206.

ATTACHMENTS:

1. None

ITEMS NEEDING SPECIAL INSTRUCTIONS:

1. None

SHUTDOWN INSTRUCTIONS:

1. This batch can be shut down at any time without causing a quality or a safety problem. If the reaction is interrupted, additional reaction time must be allowed. Consult the Engineer for details. The product may become solid in the reactor if the temperature is allowed to drop below 130 °F.

REASON FOR ISSUE:

(Revise Product Structure? Yes ___ No)(New or Changed Emissions? ___ Yes No)

1. To update per operator input:
 - Instructions for blowing back Crude FM-3108 transfer lines.
 - Added instructions for reflux cooling the batch when transferring Charge B to reactor.
 - Added instructions for reflux cooling the batch after stabilization reaction.
 - Updated instructions for starting and maintaining inert strip using the delta T setpoint.
 - Changed amount of inerts to strip to 6500 lbs and changed the order for draining inerts.
 - Added instructions for reflux cooling the batch when transferring Charge E to reactor.
2. To add a caution about the carbon packing now in use in the packed column. The packing is very brittle and care should be taken to not suddenly relieve pressure from the reactor through the packed column.
3. To change agitator speed during the stabilization reaction to 80 rpm.

REFERENCE:

1. 41-2700-3206-7 Factory Operating Procedure, R01, dated January 2, 1997.

MANUFACTURED FOR:

1. Customer Division: SCD
2. Customer Plant Contact: NA
3. Customer Lab Contact: Marylee Maendler - 236-2A-01
4. SMD/FP&TC Lab Contact: Dale Neuman - 53-6S-02

END PRODUCT USE:

Emulsifier in PTFE and fluoropolymer production

FORMULATION NOTES: If a batch size adjustment of more than 5% is required, all the charges should be scaled in proportion to the normal charges.

CHARGE CALCULATIONS: None

PROCESS TOLERANCE:

1. Unless otherwise specified, record data as indicated in the operating procedure for process variables a minimum of once per hour.
2. Unless otherwise specified, time intervals specified are a minimum time. To obtain consistent process conditions the Operator should continue processing at the specified time interval.
3. Unless otherwise specified, maintain process variations within the tolerances listed below. If unable to operate within the acceptable tolerances, contact the Supervisor or Manufacturing Engineer for instructions. Note all additional verbal instructions on the data card.

Attach all written instructions to the Production Report.

4. Unless otherwise specified in the procedure, use stencil weights for charging.

<u>PROCESS VARIABLES</u>	<u>ACCEPTABLE PROCESS TOLERANCES</u>		<u>RECORD HOURLY</u>
	(+)	(-)	<u>Y/N</u>
Agitator speed (RPM)	5	5	Y
Temperature (°F)			
batch	2	2	Y
jacket	5	5	Y
head	2	2	N
condenser water	5	5	Y
Pressure (psig)	10	10	Y
Vacuum (mm Hg)	0.5	0.5	N
(in Hg)	0.5	0.5	N
Charge Weight	2%	2%	Y
Draining Weight	1%	1%	Y
Rates	Target and tolerances are specified in the procedure.		

OPERATING PROCEDURE:

I. PRE-SERIES PREPARATION

1. The packed fractionation column or the open column can be used. Normally the packed column is left in service as the FM-3256 which follows a series requires the packed column.
2. The sight glasses on the reactor need to be protected from fluoride attack with a thin liner of Kel-F™ plastic on the inside.
3. A sight glass on the bottom of the reactor and receiver will be required to locate the phase splits.
4. The reactor, overhead, receiver, and all transfer lines must be free from contamination of other products.

Caution: *When charging caustic to the reactor the minimum personal protection required is a face shield, goggles, rubber jacket and neoprene gloves.*

A hot caustic water flush of the system followed by a water rinse is required. Vacuum charge two drums of 11-0000-0244-1, 50% Sodium Hydroxide, to the reactor. Start the agitator at 60 rpm. Fill the reactor with water.

Caution: *When flushing the reactor and overhead system with caustic and water, do not bump material through the carbon packing. The carbon packing is very brittle and bumping may damage the packing.*

Set batch setpoint to 250 °F, the jacket high limit to 350 °F and the delta T limit to 300 °F. Set the jacket mode to direct steam and heat the water. When the reactor temperature reaches 205 - 210 °F, start adding water to the reactor at a controlled rate, maintaining a batch temperature of 200 - 210 °F. Overflow the reactor through the packed column, overhead condenser and allow to flow to the receiver. After flushing the overhead for 1 hour, cool to 130 °F. Put jacket in neutral and drain overhead lines, reactor, and receiver to the sewer. Refill the reactor with water, and repeat the flush through entire system with water for 1 hour. Drain overhead lines, receiver and reactor to the sewer.

Open the reactor and receiver manholes and inspect. Contact supervisor if additional cleanup is necessary.

5. Pressure the reactor system including overhead and receiver to 40 psig with nitrogen. The pressure loss should be less than 1 psig after 30 minutes. Find and repair leaks as required. Use water to hydrostat system if necessary to find leaks.

II. CHARGING

6. Turn on condenser water and set to 55 °F.
7. Pull full vacuum on the reactor, through the receiver. Prepare to add the caustic charge:

Caution: When charging caustic to the reactor the minimum personal protection required is a face shield, goggles, rubber jacket and neoprene gloves.

Add Charge A (11-0000-0244-1), 50% sodium hydroxide to the reactor. Close all valves in the charge line when complete. Start the reactor agitator and adjust to 80 rpm.

Isolate reactor and turn off vacuum system. Isolation is necessary to prevent possible loss of low boiling inerts. Set the overhead valving for total take-off with condensate routed back to the reactor.

8. Set the reactor jacket to circulating water and adjust the batch temperature setpoint to 50 °F.
9. Prepare to add Charge B (41-2700-3108-5), Perfluorooctanoyl acid fluoride/inert cell crude to the isolated reactor from a FM-3108 storage tank (ST-11 or ST-14) using the dedicated transfer pump. Consult with the cell operator on which tank to use.

Double check all valving on the transfer line to prevent misdirected flow into another tank or vessel. Charge B must be metered through the mass flow meter when charging to the reactor.

13. Start to pump Charge B from the storage tank through the mass flow meter into the reactor. An exotherm will occur in the reactor during this transfer. Control the rate of addition and use reflux cooling (with total take-off to the reactor) so that the reactor temperature does not exceed 190 °F.

Note: Above 190 °F, reflux cooling will be necessary to lower the batch temperature. Prolonged batch temperatures above 225 °F. will break down C8 acids to less valuable inerts.

If the reactor pressure increases above 10-15 psig, verify that reflux cooling has started. If necessary, to start reflux cooling, carefully vent reactor to scrubber through equalizing line above the 1000 gallon receiver, leaving the equalizing line block valve on the top of the receiver closed.

During the transfer, a conductivity meter will monitor for an electrolyte (HF) phase. The mass flow meter will also monitor for a low density (HF) phase. If either meter detects an electrolyte phase, the transfer will be stopped automatically. If an HF phase is encountered, blow back the electrolyte to the storage tank and notify the cell operator for disposition of the electrolyte phase encountered..

14. If there is additional Charge B to be charged, return to Step 9-13 for charging instructions. Do not pull vacuum again on the reactor if it contains any FM-3108 cell product. Proceed to the reaction step if all of Charge B has been added to the reactor. Be sure the storage tank log on the cell office computer was updated.

Log the transfer in the Storage Tank Log on the computer in the Cell Office. Record the ST used and the FM-3206 lot charged.

III. STABILIZATION REACTION

15. Set the reactor jacket in circulating water, the batch temperature setpoint to 215 °F, the jacket high limit to 260 °F and the delta T limit to 300 °F. The reactor should remain sealed.

Set the agitator at 80 rpm, the condenser water control at 55 °F and set the condensate valving for total take-off routed to the reactor.

16. When the batch temperature reaches 215 °F, hold the batch for **6 hours**.
17. After completion of the reaction, lower the agitator to 60 rpm, and set the condenser water control at 55 °F and the condensate valving for total take-off routed to the reactor.

Set the batch temperature set point to 165 °F. Reflux cool the batch to 165 °F by carefully venting the reactor to the scrubber through equalizing line above the receiver. Leave the equalizing line block valve on the top of the receiver closed. Establish a reflux rate so that the condenser water temperature increases to 65-70 °F. Venting too quickly can cause a loss of low boiling inerts through the condenser if it is overloaded.

Note: Refluxing is necessary to clean the overhead and return unstabilized material to the reactor.

IV. PH CHECK

18. When the batch temperature is 165 °F or less, take an 8-ounce sample of the batch through the drain line. Wear a lowered face shield, rubber coat and gloves when sampling. Check the pH of the sample using pH paper. The pH should be greater than or equal to 10. If it is, proceed to the Inert Strip.
19. If the pH is less than 10, add one drum of Charge C (11-0000-0244-1), 50% Sodium Hydroxide to the receiver. Observe the safety precautions below:

Caution: When charging caustic to the reactor the minimum personal protection required is a face shield, goggles, rubber jacket and neoprene gloves.

Slowly pressure transfer contents to the reactor. Reheat the batch to 215 °F and hold for 4 hours using the original reaction conditions in steps 15-16. After the 4 hour hold return to Step 17.

V. INERT STRIP

20. Slowly vent any residual pressure on the reactor from the cold side of the condenser through the equalization line above the receiver to the scrubber.
21. Open the vent on the receiver and verify the drain valve is closed. Set the overhead for total take-off routed to the receiver.
22. Set the batch temperature setpoint to 215 °F, the jacket high limit to 260 °F, and set the delta T setting to 100 °F. Set the receiver jacket to emergency cooling.

When inerts start to distill to the receiver, reduce the delta T limit to 50 °F. Adjust the delta T limit as necessary to maintain a takeoff rate of 1,500 to 2,000 lbs/hr.

23. Distill the inerts to the receiver until the amount collected is greater than one-half the amount of Charge B charged. Normal amount stripped is 6,500 lbs for BC-34, or 5,500 for BC-45.
24. When the inert strip is complete, set the reactor temperature set point to 100 °F. Isolate reactor from receiver.
25. The inerts in the receiver will be split into three portions:

The first 4,800 lbs (75% of the inerts in the receiver), will be pressure transferred to the bulk FM-3160 inert storage tank.

The next 1,200 lbs (18% of the inerts in the receiver), will be drained to drums and used as Charge F later in this batch.

The final amount contains water and inerts, and will be left in the receiver. Charge D will be added to the receiver and mixed with this material.

Pressure the receiver to about 20 psig and transfer 4,800 lbs of FM-3160 inerts in the receiver to the bulk FM-3160 inert storage tank. Verify that the inert line is valved off to the receiver of the other two reactor systems. Verify that the tank is less than 98% full on the level gauge on 1st floor readout. Also note the tank pressure. The tank pressure is in psia, so subtract 14.7 lbs to get normal gauge pressure.

Drain two drums, 1,200 lbs, of the FM-3160 inerts from the receiver to drums. Save for later use as Charge F in this batch.

Watch for a water phase. Stop if water phase is encountered. Leave water phase in receiver. Charge D will be added to the receiver and mixed with this water phase.

Record both the FM-3160 bulk inerts and the drummed inerts on the Byproduct yield section of the yield card. Refer to the Byproduct section for more details.

VI. REACIDIFICATION

26. Verify the batch temperature is less than 130 °F. (Do not wait for temperature to reach 100 °F). The overhead valving should be total take-off with condensate routed to the reactor. The condenser water control should remain on cooling at 55 °F. Increase agitation to 80 rpm.
27. Vacuum Charge **D1 (41-2600-6002-9)** Water/Ammonium salts, and/or **D2 (11-0000-0995-8)**, Water to the receiver. Break vacuum with nitrogen and pressure transfer Charge D from the receiver to the reactor.

Note: Total of Charge D1 and D2 is 3,400 lbs for a normal batch size in BC-34 (BC-45 = 2,800).

28. Vacuum Charge **E (11-0000-3048-3)**, Sulfuric Acid (93%), to the receiver. Break the vacuum with nitrogen, pressure to 15 - 20 psig with nitrogen. Slowly pressure transfer the sulfuric acid to the reactor. Control the addition rate to keep the batch temperature below 200 °F.

If the reactor pressure increases above 10-15 psig, or if the batch temperature increases above 200 °F, verify that reflux cooling has started. If necessary, to start reflux cooling, carefully vent reactor to scrubber through equalizing line above the receiver, leaving the equalizing line block valve on the top of the receiver closed.

This transfer can usually be done in 15 to 20 minutes. The acid will convert the sodium salt in the reactor to acid.

29. Carefully and slowly vent any remaining pressure on the reactor from the cold side of the condenser through the receiver equalization line to the scrubber. Verify overhead is set for total take-off with condensate routed to the reactor with scrubber vent open.
30. Set the batch temperature setpoint to 200 °F, the jacket high limit to 260 °F, and set the delta T setting to 100 °F. When inerts start to reflux back to reactor, reduce the delta T limit to 50 °F. Adjust the delta T limit as necessary to maintain a steady atmospheric reflux for 30 minutes.
31. When the reflux hold is complete, set the batch temperature set point at 130 °F. When the batch temperature reaches 130 °F, turn off the agitator and let the batch phase split for 90 minutes.

32. After the 90 minute phase split, transfer the bottom product phase to ST-15, the 3,000 gallon Hastelloy storage tank. The transfer line to ST-15 ties into the BC-34 reactor drain line.
- a. Check the load cell reading on ST-15 to make sure this batch will fit. The maximum capacity of ST-15 is **40,000 lbs**. Do not fill the tank beyond this unless the tank has been visually checked and it has been determined that the batch will fit. Expect a yield of about one-half of the amount of Charge B. Check the temperature of ST-15. The agitator on ST-15 should be on at 40 rpm. ST-15 temperature should be maintained at 150 °F (± 10 deg F).
 - b. Transfer the lower product phase with nitrogen pressure. Use 10 psi more than the pressure on ST-15. The bottom product phase in the reactor consists of perfluorooctanoic acid and inerts. The top phase is sulfuric acid and water. Expect a small amount of interphase dirt between the two phases. Stop the transfer when the top phase or interphase appears in the sight glass. Expect a yield of about one-half of the amount of Charge B. Do *not* drain the top phase to the sewer at this time. Blow the transfer line empty with nitrogen to ST-15. Do not vent the pressure from ST-15 unless the pressure is above 40 psig. Any venting should be done only through the BC-34 condenser to prevent loss of inerts.
 - c. Record the net amount of the transfer (based on the change of the load cell readings of ST-15) on the yield section of the Draining Page. Blow the transfer line empty with nitrogen

VII. INERT EXTRACTION

33. Pull vacuum on the receiver and isolate. Vacuum Charge **F (41-2700-3160-6)**, Crude C8 inert mixture that was saved in drums earlier in the run to the receiver. Pressure transfer Charge F from the receiver into the reactor using nitrogen.
34. Mix the reactor for 15 minutes at 60 rpm and 130 °F.
35. After the 15 minute mix turn the agitator off and allow the batch to phase split for 45 minutes.
36. When the phase split is complete, drain the lower phase to used polyoverpak drums as **Byproduct 41-2700-3388-3**. Refer to the Byproduct section for details. The bottom product phase in the reactor consists of dilute perfluorooctanoic acid in inerts. The top phase is sulfuric acid and water. Typically there will be a small amount of interphase dirt between the two phases. Stop draining when you see the top phase in the sight glass. Sampling of the product phase is not necessary.
37. Drain the top sulfuric acid phase in the reactor to the sewer after determining that the product yield is normal. Set up a water hose in the sewer to help flush the sewer. Be sure the exhaust ventilation on the sewer is working.

VIII. POST-RUN

38. No cleaning is required between lots of FM-3206 or prior to FM-3256 Fractionation.

39. If cleaning is necessary, clean with hot caustic and water.

Caution: *When charging caustic to the reactor the minimum personal protection required is a face shield, goggles, rubber jacket and neoprene gloves.*

A hot caustic water flush of the system followed by a water rinse is required. Vacuum charge two drums of 11-0000-0244-1, 50% Sodium Hydroxide, to the reactor. Start the agitator at 60 rpm. Fill the reactor with water.

Caution: *When flushing the reactor and overhead system with caustic and water, do not bump material through the carbon packing. The carbon packing is very brittle and bumping may damage the packing.*

Set batch setpoint to 250 °F, the jacket high limit to 350 °F and the delta T limit to 300 °F. Set the jacket mode to direct steam and heat the water. When the reactor temperature reaches 205 - 210 °F, start adding water to the reactor at a controlled rate, maintaining a batch temperature of 200 - 210 °F. Overflow the reactor through the packed column, overhead condenser and allow to flow to the receiver. After flushing the overhead for 1 hour, cool to 130 °F. Put jacket in neutral and drain overhead lines, reactor, and receiver to the sewer. Refill the reactor with water, and repeat the flush through entire system with water for 1 hour. Drain overhead lines, receiver and reactor to the sewer.

Open the reactor and receiver manholes and inspect. Contact supervisor if additional cleanup is necessary.

40. If the system will be down, shut off the vacuum jets, water to the overhead condenser, water to reactor jacket, water to receiver jacket, and agitators in reactor and receiver.

DRAINING INFORMATION:

Container: 1st Choice: Drain to ST-15 Bulk FM-3206 Storage Tank
2nd Choice: Recycle polyoverpaks (34-7010-1156-0) from FM-3256/F-8281/F-8282
3rd Choice: New polyoverpak (34-7039-5732-3)

Filter: None

Label(s): Bulk - None required
If drummed: 41-2700-3206-7, Lot____, Net____, Inside.

Weight Per Container: ST-15 maximum is 40,000 lbs. (700 lbs net if drummed)

Draining Temperature: 120 - 150 °F (Maintain tank at 140 to 160 °F)

Draining Pressure: 0 - 40 psig as required. If draining to drums use 0-3 psig.

Special Draining Instructions:

Product will solidify at room temperature

Special Handling Instructions:

Bulk: Maintain the ST-15 above 140 deg F.
Drummed material must be put in a 150 deg F Hot Room prior to use.

Final Sample Requirements:

QC Lab: One 8-ounce

Customer: None

Storage: ST-15

BY-PRODUCT DRAINING INFORMATION:

Step: 25

Code: 41-2700-3160-6

Description: Distilled Crude C8F160 Inert Mixture

Container: 1. None, pump to bulk FM-3160 tank
2. 1st Alternate: Green polyoverpak drum (34-7029-4109-6)
3. 2nd Alternate: Blue polyoverpak drum (34-7002-2745-6)

Label(s): Drums only: 41-2700-3160-6, Lot ___, Net ___, Inside

Weight Per Container: 600 lbs net (for drums)

Amount: 6,000 lbs (4,800 to bulk tank & 1,200 to drums)

Draining Temperature: 100 °F or less

Draining Pressure: 0 - 20 psig

Special Draining Instructions:

Check the volume and pressure on the bulk tank on the 1st floor readout. The volume should be less than 98%. Pressure the distilled inert mixture from the receiver to the bulk tank (15-98) with nitrogen pressure of 20 psig or as required. Watch carefully for an upper water phase near the end. Tank pressure reads out in psia. Subtract 14.7 to get normal gauge pressure. Both readings are on 1st floor next to BC-34 new receiver.

Final Sample Requirements: None

Storage: Outside bulk FM-3160 tank (15-98)

Step: 36

Code: **41-2700-3388-3**

Description: Dilute Perfluorooctanoic Acid and Inerts

Container: 1. Recycle polyoverpak drum (34-7010-1156-0)
2. 1st Alternate: New polyoverpak drum (34-7039-5732-3)
3. 2nd Alternate: Blue polyoverpak drum (34-7002-2745-6)

Label(s): 41-2700-3388-3, Lot ____, Net ____, Inside

Weight Per Container: 600 lbs net
Amount: 1,200 lbs

Draining Temperature: 100 °F or less
Draining Pressure: 0 - 20 psig

Final Sample Requirements: None

Storage: Inside

PRE-SERIES CLEAN-UP:

Hot caustic and water flush per Step 4 if required.

BETWEEN RUNS:

None required.

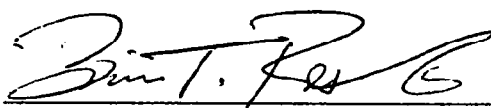
CLEANING AFTER LAST LOT OF SERIES:

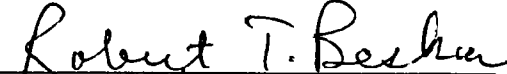
Normally no cleaning required as FM-3256 fractionation will follow. If cleaning is required, refer to Step 4 for a hot caustic and water flush.

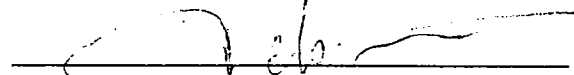
WASTE DISPOSAL:

Step No. 4
Description: 5% Sodium Hydroxide in water solution
Waste Stream Code: None
Container: None
Disposition: Drain to Phase I chemical sewer
Amount: 10,000 lbs each 7th lot (if required)

Step No. 37
Description: Sulfuric acid and water solution
Waste Stream Code: None
Container: None
Disposition: Drain to Phase I chemical sewer
Amount: ~7,500 lbs

AUTHOR: Manufacturing Engineer: 
Brian T. Reski

APPROVED BY: Product Chemist: 
Robert T. Beskar

Product Manager/Team Leader: 

Health & Safety Eng.: Yes No

Environmental Coordinator: Yes No

COPIES TO: (Reference SOP 408-007)

SMD/FP&TC, Frank W. Klink, 53-6S-02
lww\fm3206.r01

50%

3000 lbs NH₄H
 12000 lb all urea
 4000 lb H₂SO₄
 3400 lbs H₂O

3M COTTAGE GROVE PLANT
CHEMICAL PLANT

FACTORY OPERATING PROCEDURE

Effective Date: **September 10, 1997**
 Superseding: July 25, 1997

CODE NUMBER: 41-2700-3206-7 **REVISION NO.:** R04

SUBJECT: STABILIZATION OF PERFLUOROCTANOIC ACID IN
INERTS

YIELD:

41-2700-3206-7 Stabilized Perfluorooctanoic Acid 7,200 lbs

ESTIMATED MACHINE TIME: 24.0 Hours

EQUIPMENT:

1. BC-34; Dept 3060, 1,250 gallon Hastelloy reactor with packed or open column, 1,000 gallon Monel receiver (old reciever), and ST-15, 3,000 gal storage tank for FM-3206. Normally run in a five lot series in order to fill ST-15

ATTACHMENTS:

1. Personal Protective Equipment Requirements Summary Sheet for FM-3206.
2. FM-3206 Safe Operating Limits

ITEMS NEEDING SPECIAL ATTENTION:

1. All references to the receiver in this FOP refer to the 1000 gallon monel receiver (old receiver). This procedure does not require the use the the new 1250 gallon hastelloy receiver.

SHUTDOWN INSTRUCTIONS:

- 1. This batch can be shut down at any time without causing a quality or a safety problem. If the reaction is interrupted, additional reaction time must be allowed. Consult the Engineer for details. The product may become solid in the reactor if the temperature is allowed to drop below 130 oF.

REASON FOR ISSUE:

(Revise Product Structure? ___Yes _x_ No)(New or Changed Emissions?
___Yes _x_ No)

- 1. To update the instructions for transferring FM-3160 to the outside inert storage tank. A maximum of 35 psig of nitrogen pressure can be used for the pressure transfer of inerts to the outside storage tank.
- 2. To add attachment 2, FM-3206 Safe Operating Limits.

REFERENCE:

- 1. 41-2700-3206-7 Factory Operating Procedure, R03, dated July 25, 1997.

MANUFACTURED FOR:

- 1. Customer Division: SCD
- 2. Customer Plant Contact: NA
- 3. Customer Lab Contact: Marylee Maendler - 236-2A-01
- 4. SMD/FP&TC Lab Contact: Dale Neuman - 53-6S-02

END PRODUCT USE: Emulsifier in PTFE and fluoropolymer production

FORMULATION NOTES: If a batch size adjustment of more than 5% is required, all the charges should be scaled in proportion to the normal charges.

CHARGE CALCULATIONS: None

PROCESS TOLERANCE:

1. Unless otherwise specified, record data as indicated in the operating procedure for process variables a minimum of once per hour.
2. Unless otherwise specified, time intervals specified are a minimum time. To obtain consistent process conditions the Operator should continue processing at the specified time interval.
3. Unless otherwise specified, maintain process variations within the tolerances listed below. If unable to operate within the acceptable tolerances, contact the Supervisor or Manufacturing Engineer for instructions. Note all additional verbal instructions on the data card.

Attach all written instructions to the Production Report.

4. Unless otherwise specified in the procedure, use stencil weights for charging.

<u>PROCESS VARIABLES</u>	<u>ACCEPTABLE PROCESS TOLERANCES</u>		<u>RECORD HOURLY</u>
	(+)	(-)	<u>Y/N</u>
Agitator speed (RPM)	5	5	Y
Temperature (oF)			
batch	2	2	Y
jacket	5	5	Y
head	2	2	N
condenser water	5	5	Y
Pressure (psig)	10	10	Y
Vacuum (mm Hg)	0.5	0.5	N
(in Hg)	0.5	0.5	N
Charge Weight	2%	2%	Y
Draining Weight	1%	1%	Y
Rates	Target and tolerances are specified in the procedure.		

OPERATING PROCEDURE:

I. PRE-SERIES PREPARATION

1. The packed fractionation column or the open column can be used. Normally the packed column is left in service as the FM-3256 which follows a series requires the packed column.
2. The sight glasses on the reactor and receivers need to be protected from fluoride attack with a thin liner of Kel-F™ plastic on the inside. Inspect and replace any sightglass that has been etched from fluoride attack.
3. A sight glass on the bottom of the reactor and receiver will be required to locate the phase splits.
4. The reactor, overhead, receiver, and all transfer lines must be free from contamination of other products.

PPE Note: Refer to attachment 1 for the PPE requirements for handling RM-244.

A hot caustic water flush of the system followed by a water rinse is required. Vacuum charge two drums of **11-0000-0244-1**, 50% Sodium Hydroxide, to the reactor. Start the agitator at 60 rpm. Fill the reactor with water.

Caution: When flushing the reactor and overhead system with caustic and water, do not bump material through the carbon packing. The carbon packing is very brittle and bumping may damage the packing.

Set batch setpoint to 250 oF, the jacket high limit to 350 oF and the delta T limit to 300 oF. Set the jacket mode to direct steam and heat the water. When the reactor temperature reaches 205 - 210 oF, start adding water to the reactor at a controlled rate, maintaining a batch temperature of 200 - 210 oF. Overflow the reactor through the packed column, overhead condenser and allow to flow to the receiver. After collecting 8000 lbs of water in the receiver, cool to 130 oF. Put jacket in neutral and drain overhead lines, reactor, and receiver to the sewer. Refill the reactor with water, and repeat the flush through entire system with water until 8000 lbs of water is collected in the receiver. Drain overhead lines, receiver and reactor to the sewer.

Open the reactor and receiver sightglasses and inspect. Contact supervisor if additional cleanup is necessary.

- 5. Pressure the reactor system including overhead and receiver to 40 psig with nitrogen. The pressure loss should be less than 1 psig after 30 minutes. Find and repair leaks as required. Use water to hydrostat system if necessary to find leaks.

Pressure ST-15 to 40 psig with nitrogen. The pressure loss should be less than 1 psig after 30 minutes. Find and repair leaks as required. Vent ST-15 through reactor transfer line with condenser water on.

II. CHARGING

- 6. Turn on condenser water and set to 55 oF.
- 7. Pull full vacuum on the reactor, through the receiver. Prepare to add the RM-244 caustic charge:

PPE Note: Refer to attachment 1 for the PPE requirements for handling RM-244.

Add Charge A (11-0000-0244-1), 50% sodium hydroxide to the reactor. Close the flush bottom valve when Charge A is complete. Close remaining valves in the charge line. Start the reactor agitator and adjust to 80 rpm.

*top
m r d*

Isolate reactor and turn off vacuum system. Isolation is necessary to prevent possible loss of low boiling inerts. Set the overhead valving for total take-off with condensate routed back to the reactor.

- 8. Set the reactor jacket to circulating water and adjust the batch temperature setpoint to 50 oF.
- 9. Prepare to add Charge B (41-2700-3108-5), Perfluorooctanoyl acid fluoride/inert cell crude to the isolated reactor from a FM-3108 storage tank (ST-11 or ST-14). This transfer uses a dedicated transfer pump, transfer line, and mass flow meter. Consult with the cell operator on which tank to use.

Double check all valving on the dedicated transfer line to prevent misdirected flow. Normally, this transfer line is locked out to prevent transfers to or from other tanks and vessels.

Review FOP through step 10 before starting the transfer. The transfer of Charge B is started by performing the following steps:

- Open the valve on the bottom of ST 11 or ST 14 (Cell Operator at Cell/Tank Farm Console).
- Select the BC34 Transfer display on the BC-34 console.
- Select the C8 Charge selector and select the CHARGE setpoint.

The Charge B transfer will be initiated by starting the transfer pump and by opening the valve above BC-34. The FM-3108 will be metered through the mass flow meter and totalized. The transfer program is set to transfer 12000 lbs of FM-3108.

10. Monitor the transfer of Charge B from the storage tank to the reactor. An exotherm will occur in the reactor during this transfer. Control the rate of addition by throttling the hand valve above the reactor. Use a transfer rate of about 100 lbs/min.

Use reflux cooling (with total take-off to the reactor) so that the reactor temperature does not exceed 190 oF.

Note: Above 190 oF, reflux cooling will be necessary to lower the batch temperature. Prolonged batch temperatures above 225 oF. will break down C8 acids to less valuable inerts.

If the reactor pressure increases above 10-15 psig, verify that reflux cooling has started. If necessary, to start reflux cooling, carefully vent reactor to scrubber through equalizing line above the 1000 gallon receiver, leaving the equalizing line block valve on the top of the receiver closed.

During the transfer, a conductivity meter will monitor for an electrolyte (HF) phase. The mass flow meter will also monitor for a low density (HF) phase. If either meter detects an electrolyte phase, the transfer will be stopped automatically. If an HF phase is encountered, blow back the electrolyte to the storage tank and notify the cell operator for disposition of the electrolyte phase encountered..

Note: To blow back the electrolyte, the transfer valves must be forced opened. Change the interlock selector to OVERRIDE. The valves may now be opened and the line blown back. After 10 minutes, the interlock selector automatically changes back to ACTIVE.

11. If there is additional Charge B to be charged, return to Step 9-10 for charging instructions. Do not pull vacuum again on the reactor if it contains any FM-3108 cell product. Proceed to the reaction step if all of Charge B has been added to the reactor.

Log the transfer in the Storage Tank Log on the computer in the Cell Office.
Record the ST used and the FM-3206 lot charged.

When charging to the reactor, the transfer program performs the following:

- Monitors for high conductivity or low density during the transfer. If either is encountered,
the C8 Charge selector is put on HOLD, the pump is stopped and the BC-34 valve is closed.
- Monitors for an operator request to HOLD or STOP.
- Stops the transfer pump and closes the BC-34 valve when complete or when the operator
selects HOLD or STOP.

III. STABILIZATION REACTION

12. Set the reactor jacket in circulating water, the batch temperature setpoint to 215 oF, the jacket high limit to 260 oF and the delta T limit to 300 oF. The reactor should remain sealed.

Set the agitator at 80 rpm, the condenser water control at 55 oF and set the condensate valving for total take-off routed to the reactor.

13. When the batch temperature reaches 215 oF, hold the batch for **6 hours**.
14. After completion of the reaction, lower the agitator to 60 rpm. Verify the condenser water control is set at 55 oF and the condensate valving for total take-off routed to the reactor.

Set the batch temperature set point to 165 oF. Reflux cool the batch to 165 oF by carefully venting the reactor to the scrubber through equalizing line above the receiver. Leave the equalizing line block valve on the top of the receiver closed. Establish a reflux rate so that the condenser water temperature increases to 65-70 oF. Venting too quickly can cause a loss of low boiling inerts through the condenser if it is overloaded.

Note: Refluxing is necessary to clean the overhead and return unstabilized material to the reactor.

IV. pH CHECK

15. When the batch temperature is 165 oF or less, prepare to take an 8-ounce sample of the batch through the drain line.

PPE Note: Refer to attachment 1 for the PPE requirements for handling Sample 1.

Flush the sample line and take the sample. Check the pH of the sample using pH paper. The pH should be greater than or equal to 10. If the pH of the sample is greater than or equal to 10, proceed to the Inert Strip.

16. If the pH is less than 10, prepare to add one drum of Charge C (11-0000-0244-1), 50% Sodium Hydroxide to the receiver.

PPE Note: Refer to attachment 1 for the PPE requirements for handling RM-244.

Isolate the reactor from the receiver. Pull vacuum on the receiver and vacuum Charge C to the receiver. Release vacuum and pressure receiver with nitrogen. Slowly pressure transfer contents to the reactor. Reheat the batch to 215 oF and hold for 4 hours using the original reaction conditions in step 13. After the 4 hour hold return to Step 14.

V. INERT STRIP

17. Slowly vent any residual pressure on the reactor from the cold side of the condenser through the equalization line above the receiver to the scrubber.
18. Open the vent on the receiver and verify the drain valve is closed. Set the overhead for total take-off routed to the receiver.
19. Set the batch temperature setpoint to 215 oF, the jacket high limit to 260 oF, and set the delta T setting to 100 oF. Set the receiver jacket to emergency cooling.

When inerts start to distill to the receiver, reduce the delta T limit to 50 oF.

Adjust the delta T limit as necessary to maintain a takeoff rate of 1,500 to 2,000 lbs/hr.

20. Distill the inerts to the receiver until the amount collected is greater than one-half the amount of Charge B charged. Normal amount stripped is 6,500 lbs for BC-34.
21. When the inert strip is complete, set the reactor temperature set point to 100 oF. Isolate reactor from receiver.
22. The inerts in the receiver will be split into three portions:
 - The first 4,800 lbs will be pressure transferred to the bulk FM-3160 inert storage tank.
 - The next 1,200 lbs will be drained to drums and used as Charge F later in this batch.
 - The final amount contains water and inerts, and will be left in the receiver. Charge D will be added to the receiver and mixed with this material.

Verify the outside inert storage tank level is less than 96%, and that the pressure in the storage tank is less than 40 psia. If the level is greater than 96%, the inerts will have to be drummed. Refer to by-product draining section of FOP for drum to use. If the storage tank pressure is greater than 40 psia, vent the storage tank through the reactor overhead system with condenser water on at 55 oF.

If the storage tank pressure is less than 20 psia, and the automatic nitrogen block valve is open, investigate the status of the tank. If necessary, perform a

pressure test on the tank and transfer lines before initiating the transfer of inerts.

An automatic valve is on the inert charge line on top of the outside storage tank. If the tank level is above 98%, or if the tank pressure is above 50 psia, the charge valve will automatically close. Verify that the valve is open, and open if necessary.

Use nitrogen to pressure the receiver to 30 psig. **Do not exceed 35 psig of nitrogen pressure.** Pressure transfer the distillate as FM-3160 to the outside inert storage tank. **Stop the transfer when 4800 lbs have been pushed to the outside storage tank.** Making this transfer first ensures that the tank is not pressured up with excess nitrogen pressure. Also, there can be an upper water phase in the receiver. Water should not go to the outside inert storage tank. Watch for the water phase in the receiver during the inert transfer.

PPE Note: *Refer to attachment 1 for the PPE requirements for handling F-6002 and FM-3160.*

Drain two drums, 1,200 lbs, of the FM-3160 inerts from the receiver to drums. Save for later use as Charge F in this batch.

Watch for a water phase. Stop if water phase is encountered. Leave water phase in receiver. Charge D will be added to the receiver and mixed with this water phase.

When the inert transfer/draining is complete, vent the receiver pressure slowly through the reactor overhead.

Record both the FM-3160 bulk inerts and the drummed inerts on the Byproduct yield section of the yield card. Refer to the Byproduct section for more details.

VI. REACIDIFICATION

- 23. Verify the batch temperature is less than 130 oF. (Do not wait for temperature to reach 100 oF). The overhead valving should be total take-off with condensate routed to the reactor. The condenser water control should remain on cooling at 55 oF. Increase agitation to 80 rpm.
- 24. Isolate the reactor from the receiver. Prepare to vacuum Charge **D1 (41-2600-6002-9)** Water/Ammonium salts, and/or **D2 (11-0000-0995-8)**, Water to the receiver.

PPE Note: Refer to attachment 1 for the PPE requirements for handling F-6002.

Add Charge D to the receiver. Break vacuum with nitrogen and pressure transfer Charge D from the receiver to the reactor.

Note: Total of Charge D1 and D2 is 3,400 lbs for a normal batch size in BC-34.

- 25. Isolate the reactor from the receiver. Prepare to vacuum Charge **E (11-0000-3048-3)**, Sulfuric Acid (93%), to the receiver.

PPE Note: Refer to attachment 1 for the PPE requirements for handling RM-3048.

Add Charge E to the receiver. Break the vacuum with nitrogen and pressure to 15 - 20 psig with nitrogen. Slowly pressure transfer the sulfuric acid to the reactor. Control the addition rate to keep the batch temperature below 200 oF.

If the reactor pressure increases above 10-15 psig, or if the batch temperature increases above 200 oF, verify that reflux cooling has started. If necessary, to start reflux cooling, carefully vent reactor to scrubber through equalizing line above the receiver, leaving the equalizing line block valve on the top of the receiver closed.

This transfer can usually be done in 15 to 20 minutes. The acid will convert the sodium salt in the reactor to acid.

- 26. Carefully and slowly vent any remaining pressure on the reactor from the cold side of the condenser through the receiver equalization line to the scrubber. Verify overhead is set for total take-off with condensate routed to the reactor with scrubber vent open.

27. Set the batch temperature setpoint to 200 oF, the jacket high limit to 260 oF, and set the delta T setting to 100 oF. When inerts start to reflux back to reactor, reduce the delta T limit to 50 oF. Adjust the delta T limit as necessary to maintain a steady atmospheric reflux for 30 minutes.

28. When the reflux hold is complete, set the batch temperature set point at 130 oF. When the batch temperature reaches 130 oF, turn off the agitator and let the batch phase split for 90 minutes.
29. After the 90 minute phase split, transfer the bottom product phase to ST-15, the 3,000 gallon Hastelloy storage tank. The transfer line to ST-15 ties into the BC-34 reactor drain line.
- Check the load cell reading on ST-15 to make sure this batch will fit. The maximum capacity of ST-15 is **40,000 lbs**. Do not fill the tank beyond this unless the tank has been visually checked and it has been determined that the batch will fit. Expect a yield of about one-half of the amount of Charge B. Check the temperature of ST-15. The agitator on ST-15 should be on at 40 rpm. ST-15 temperature should be maintained at 150 oF (± 10 deg F).
 - Transfer the lower product phase with nitrogen pressure. Use 10 psi more than the pressure on ST-15. The bottom product phase in the reactor consists of perfluorooctanoic acid and inerts. The top phase is sulfuric acid and water. Expect a small amount of interphase dirt between the two phases. Stop the transfer when the top phase appears in the sight glass. Expect a yield of about one-half of the amount of Charge B.

What?

Caution: Do not drain the top phase to the sewer at this time. Top phase contains sulfuric acid, water, and a small amount of C8 acid.

- Do not vent the pressure from ST-15 unless the pressure is above 40 psig. Any venting should be done only through the BC-34 condenser to prevent loss of inerts.
- Record the net amount of the transfer (based on the change of the load cell readings of ST-15) on the yield section of the Draining Page.

Vii. inert extraction

30. Pull vacuum on the receiver and isolate. Prepare to vacuum Charge F (41-2700-3160-6) Crude C8 inert mixture to the receiver. (Charge F was drained to drums earlier in the run.)

PPE Note: Refer to attachment 1 for the PPE requirements for handling FM-3160.

Add Charge F to the receiver. Break vacuum with nitrogen and pressure transfer Charge F from the receiver to the reactor.

31. Mix the reactor for 15 minutes at 60 rpm and 130 oF. After the 15 minute mix turn the agitator off and allow the batch to phase split for 45 minutes.
32. After the 45 minute phase split, transfer the bottom product phase to ST-15, the 3,000 gallon Hastelloy storage tank. The transfer line to ST-15 ties into the BC-34 reactor drain line.

- Check the load cell reading on ST-15 to make sure this batch will fit. The maximum capacity of ST-15 is **40,000 lbs**. Do not fill the tank beyond this unless the tank has been visually checked and it has been determined that the bottom phase will fit. Expect to transfer about 1200 lbs.
- Transfer the lower product phase with nitrogen pressure. Use 10 psi more than the pressure on ST-15. The bottom product phase in the reactor consists of perfluorooctanoic acid and inerts. The top phase is sulfuric acid and water. Expect a small amount of interphase dirt between the two phases. Stop the transfer when the top phase appears in the sight glass. Expect to transfer about 1200 lbs.

Caution: Do not drain the top phase to the sewer at this time. Top phase contains sulfuric acid and water.

- Blow the transfer line empty with nitrogen to ST-15. Do not vent the pressure from ST-15 unless the pressure is above 40 psig. Any venting should be done only through the BC-34 condenser to prevent loss of inerts.
 - Record the net amount of the transfer (based on the change of the load cell readings of ST-15) on the yield section of the Draining Page. Record this transfer as FM-3206.
33. Prepare to drain the top sulfuric acid phase in the reactor to the sewer after

determining that the product yield is normal.

Caution: Top phase contains sulfuric acid and water. Refer to attachment 1 for PPE.

Set up a water hose in the sewer to help flush the sewer. Be sure the exhaust ventilation on the sewer is working. Carefully drain top phase to the sewer.

VIII. post-run

34. No cleaning is required between lots of FM-3206 or prior to FM-3256 Fractionation.
35. If cleaning is necessary, clean with hot caustic and water.

PPE Note: Refer to attachment 1 for the PPE requirements for handling RM-244.

A hot caustic water flush of the system followed by a water rinse is required. Vacuum charge two drums of **11-0000-0244-1**, 50% Sodium Hydroxide, to the reactor. Start the agitator at 60 rpm. Fill the reactor with water.

Caution: When flushing the reactor and overhead system with caustic and water, do not bump material through the carbon packing. The carbon packing is very brittle and bumping may damage the packing.

Set batch setpoint to 250 oF, the jacket high limit to 350 oF and the delta T limit to 300 oF. Set the jacket mode to direct steam and heat the water. When the reactor temperature reaches 205 - 210 oF, start adding water to the reactor at a controlled rate, maintaining a batch temperature of 200 - 210 oF. Overflow the reactor through the packed column, overhead condenser and allow to flow to the receiver. After collecting 8000 lbs of water in the receiver, cool to 130 oF. Put jacket in neutral and drain overhead lines, reactor, and receiver to the sewer. Refill the reactor with water, and repeat the flush through entire system with water until 8000 lbs of water is collected in the receiver. Drain overhead lines, receiver and reactor to the sewer.

Open the reactor and receiver sightglasses and inspect. Contact supervisor if additional cleanup is necessary.

36. If the system will be down, shut off the vacuum jets, water to the overhead condenser, water to reactor jacket, water to receiver jacket, and agitators in reactor and receiver.

DRAINING INFORMATION:

Container: 1st Choice: Drain to ST-15 Bulk FM-3206 Storage Tank
2nd Choice: Recycle polyoverpaks (34-7010-1156-0) from FM-3256/F-8281/F-8282
3rd Choice: New polyoverpak (34-7039-5732-3)

Filter: None

Label(s): Bulk - None required
If drummed: 41-2700-3206-7, Lot ____, Net ____,
Inside.

Weight Per Container: ST-15 maximum is 40,000 lbs. (700 lbs net if
drummed)

Draining Temperature: 120 - 150 oF (Maintain tank at 140 to 160 oF)

Draining Pressure: 0 - 40 psig as required. If draining to drums use 0-3
psig.

Special Draining Instructions:
Product will solidify at room temperature

Special Handling Instructions:
Bulk: Maintain the ST-15 above 140 deg F.
Drummed material must be put in a 150 deg F Hot Room prior to use.

Final Sample Requirements:
QC Lab: One 8-ounce - When requested by Engineer or
Chemist
Customer: None
Storage: ST-15

BY-PRODUCT DRAINING INFORMATION:

Step: 22

Code: **41-2700-3160-6**

Description: Distilled Crude C8F160 Inert Mixture

Container: 1. None, pressure transfer bulk FM-3160 tank
2. 1st Alternate: Green polyoverpak drum
(34-7029-4109-6)
3. 2nd Alternate: Blue polyoverpak drum
(34-7002-2745-6)

Label(s): Drums only: 41-2700-3160-6, Lot___, Net___, Inside

Weight Per Container: 600 lbs net (for drums)

Amount: 6,000 lbs (4,800 to bulk tank & 1,200 to drums)

Draining Temperature: 100 oF or less

Draining Pressure: 0 - 20 psig

Special Draining Instructions:

Check the volume and pressure on the bulk tank on the 1st floor readout. The volume should be less than 98%. Pressure the distilled inert mixture from the receiver to the bulk tank (15-98) with nitrogen pressure of 20 psig or as required. Watch carefully for an upper water phase near the end. Tank pressure reads out in psia. Subtract 14.7 to get normal gauge pressure. Both readings are on 1st floor next to BC-34 new receiver.

Final Sample Requirements: None

Storage: Outside bulk FM-3160 tank (15-98)

PRE-SERIES CLEAN-UP:

Hot caustic and water flush per Step 4 if required.

BETWEEN RUNS:

None required.

CLEANING AFTER LAST LOT OF SERIES:

Normally no cleaning required as FM-3256 fractionation will follow. If cleaning is required, refer to Step 4 for a hot caustic and water flush.

WASTE DISPOSAL:

Step No. 4
Description: 5% Sodium Hydroxide in water solution
Waste Stream Code: None
Container: None
Disposition: Drain to Phase I chemical sewer
Amount: 10,000 lbs each 7th lot (if required)

Step No. 33
Description: Sulfuric acid and water solution
Waste Stream Code: None
Container: None
Disposition: Drain to Phase I chemical sewer
Amount: ~ 7,500 lbs

AUTHOR: Manufacturing Engineer: _____
Brian T. Reski

APPROVED BY: Product Chemist: _____
Robert T. Beskar

Product Manager/Team Leader: _____

Health & Safety Eng.: _____ Yes _____ No

Environmental Coordinator: _____ Yes _____ No

COPIES TO: (Reference SOP 408-007)

SMD/FP&TC, Frank W. Klink, 53-6S-02
btr/fm3206.r04

Attachment 1

PERSONAL PROTECTIVE EQUIPMENT REQUIREMENTS FOR PRODUCT NUMBER: 41-2700-3206-7

Engineer: Brian Reski

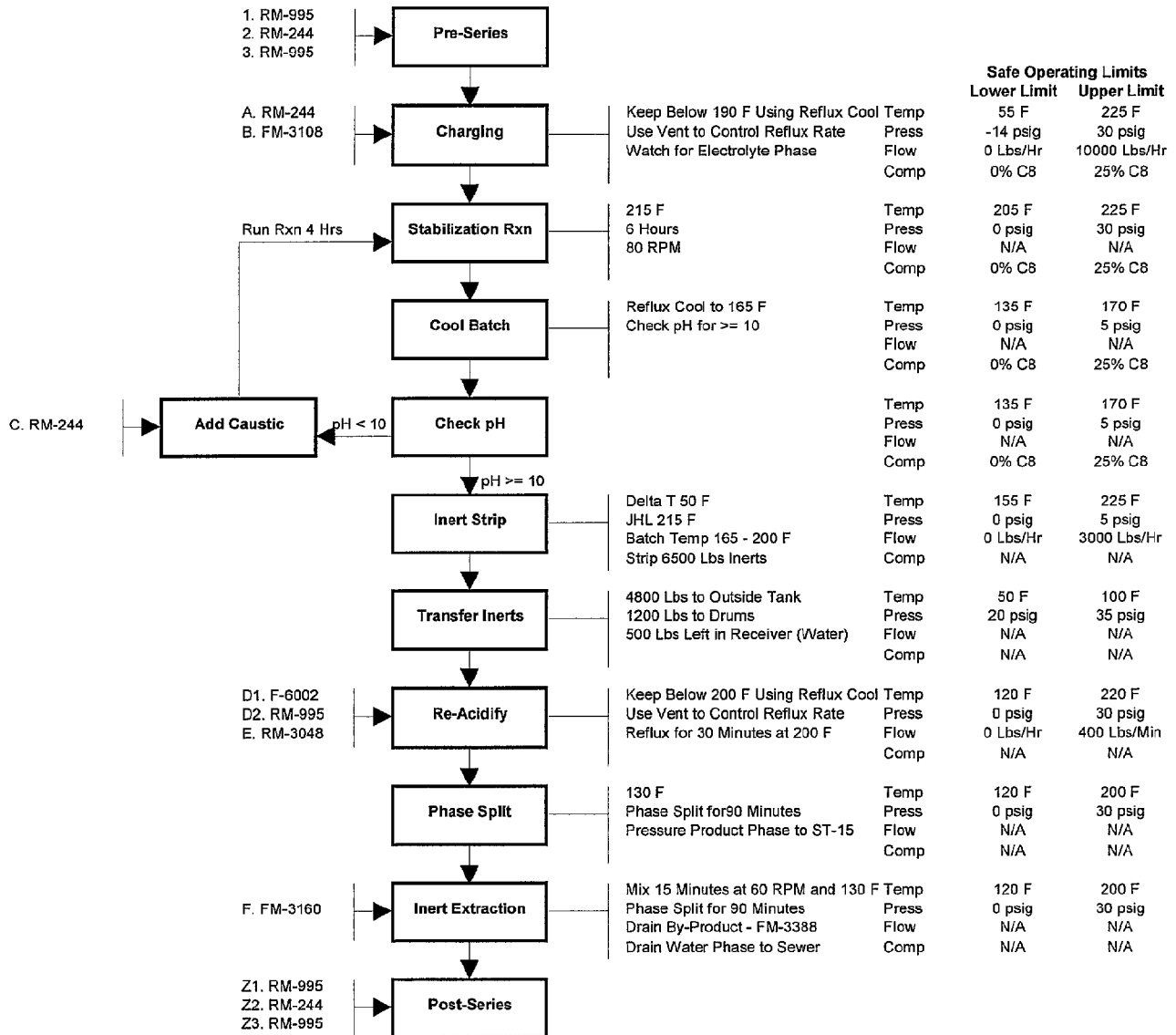
Hazard Level Definitions:

- E=Extreme: Likely to cause irreversible tissue damage, serious illness or death from contact or exposure with a very small amount.
- H=High: May cause extreme irritation, irreversible tissue damage, serious illness or death from single or repeated contact or exposures.
- M=Medium: May cause moderate to severe irritation; or may cause allergic reaction (sensitization); or may cause reversible systemic effects.
- L=Low: May cause mild temporary irritation on contact or exposure; no cumulative effects are expected from repeated contact or exposures.

NA = Not Applicable

11-Digit Code	Material Name	PHYSICAL DATA		HAZARD LEVEL			EYES		SKIN				LUNGS		Comments		
		Physical State	Flammable (or handled above flashpoint)*	Flashpoint (oF)	Eyes	Skin	Lungs	Goggles	Face shield	Gloves	Rubber jacket	Rubber suit & boots	Spot ventilation	Dust mask		Cartridge respirator	Fresh air mask / Helmet
11-0000-0244-1	Sodium hydroxide	L	NA	NA	H	H	M	X	X	Neoprene	X						
41-2700-3108-5	C8 ACID FLUORIDE CELL CRUDE	L	NA	NA	H	H	H	X	X	Neoprene	X	X		Yellow			1
41-2600-6002-9	C8 AMMONIUMSALT DISSOLVED IN WATER	L	NA	NA	M	M	L			Neoprene							
11-0000-3048-3	Sulfuric Acid	L	NA	NA	H	H	H	X	X	Neoprene	X	X		Yellow			
41-2700-3160-6	CRUDE C8 INERTS	L	NA	NA	M	M	M	X	X	Neoprene		X					1
41-2700-3206-7	STABILIZED PERFLUORO-OCTANOIC ACID IN INERTS	L	NA	NA	H	H	M	X	X	Neoprene	X	X					2
Sample 1	Sample for pH Check	L	NA	NA	H	H	M	X	X	Neoprene	X	X					
Comment 1	Handled with closed system. Use specified PPE if handling open containers.																
Comment 2	Solid at room temperature. Liquid above 120 F. Handled with closed system. Use specified PPE if handling open containers.																

Attachment 2 - Safe Operating Limits FM-3206 C8 Acid Stabilization Process



**3M COTTAGE GROVE PLANT
CHEMICAL PLANT**

FACTORY OPERATING PROCEDURE

Effective Date: **September 1, 1997**
Superceding: **March 10, 1997**

CODE NUMBER: 41-2700-3256-2 REVISION NO.: R02

SUBJECT: FRACTIONATION OF PERFLUOROOCCTANOIC ACID

YIELD:

41-2700-3256-2 Perfluorooctanoic Acid 7,500 - 10,500 lbs.

ESTIMATED MACHINE TIME: 90 - 110 Hours

EQUIPMENT:

1. Dept. 3060, BC-34 (1250 gallon Hastelloy reactor system).
2. Dept. 3060, BC-33 (1000-gallon Hastelloy reactor system) only with approval of Mfg. Engineer.

ITEMS NEEDING SPECIAL ATTENTION:

1. FM-3206, FM-3210, FM-3257, and F-4169 are Hot Room charges. Store in hot room 48 hours before changing.
2. FM-3206 should be as free of water as possible. Decant before charging, if necessary.

REASON FOR CHANGE:

(Revise Product Structure? ___ Yes x No)(New or Changed Emissions? ___ Yes x No)

1. To add Attachment 1, PPE Requirements for FM-3256, and to add references to the PPE attachment in the body of the FOP.
2. To update the instructions for transferring FM-3160 to the outside inert storage tank. A maximum of 35 psig of nitrogen pressure can be used for the pressure transfer of inerts to the outside storage tank.
3. To add inspection of sightglasses to pre-series preparation section of FOP.
4. To change the by-product 41-2700-3388-3 draining. Instead the inert flush of the column will be transferred to ST-15 and mixed with the FM-3206 product.

SHUTDOWN INSTRUCTIONS:

1. This run can be shutdown at any time without causing a safety or quality problem except as noted below. If the process is shut down for short periods (less than 8 hrs) the batch should be left on total reflux. This will avoid the time lost in cool down and heatup. If the fractionation is interrupted the column head temperature must be allowed to stabilize on total reflux before resuming takeoff to assure proper separation of components.

ATTACHMENTS:

1. PPE Requirements for FM-3256.

CHARGE CALCULATION:

1. Charge A = 13,600 lbs of FM-3206
2. Charge B = 100 lbs of RM-3179
3. Charge C = 25,600 lbs of FM-3206
4. Charge D = Optional Charge of FM-3210 if Specified
5. Charge E = Optional Charge of FM-3257 if Specified
6. Charge F = 500 lbs of RM-3048
7. Charge G = Optional Charge of F-4169 if Specified
8. Charge H = Optional 1200 lbs of FM-3160 for Column Flush if FM-3206 or FM-3256 does not follow.

REFERENCE:

1. 41-2700-3256-2 Factory Operating Standard, 3/10/97, Revision 1.

MFG. FOR:

1. Customer Division: SCD
2. Customer Plant Contact: NA
3. Customer Lab Contact: Marylee Maendler - 236-2A-01
4. SMD/FP&TC Contact: Lloyd White - 53-6S-02

END PRODUCT USE:

FC-118, FC-126 and FC-143 surfactants used in teflon manufacture; FC-26 which is sold as the acid.

PROCESS TOLERANCE:

1. Unless otherwise specified, record data for the process variables as requested in the FOP a minimum of once per hour.
2. Unless otherwise specified, time intervals specified are a minimum time. To obtain consistent process conditions the Operator should continue processing at the specified time interval.
3. Unless otherwise specified, maintain process variations within the tolerances listed below. If unable to operate within the acceptable tolerances, contact the Supervisor, or Mfg. Engineer for instructions. Note all additional verbal instructions on the data card. Attach all written instructions to the Production Reports.
4. Unless otherwise specified in the procedure, use stencil weights for charging.

<u>PROCESS VARIABLES</u>	<u>ACCEPTABLE PROCESS TOLERANCES</u>		<u>RECORD Y or N</u>
	(+)	(-)	
Agitator speed (RPM)	5	5	Y
Temperature (°F)			
batch	5	5	Y
jacket	5	5	Y
head	2	2	Y
condenser water	5	5	Y
Pressure (psig)	5	5	Y
Column <i>D.P.</i> (mm Hg)	5	5	Y
Vacuum (mm Hg)	2	2	Y
(in Hg)	0.5	0.5	N
Charge weight	5	5	Y
Draining weight	2	2	Y
Rates	Target and tolerances are specified in the procedure.		

OPERATING PROCEDURE:

I. PRE-RUN PREPARATION

1. The packed column must be in service; blank the open column if necessary.

The sight glasses on the reactor and receivers need to be protected from fluoride attack with a thin liner of Kel-F™ plastic on the inside. Inspect and replace any sightglass that has been etched from fluoride attack.

2. The reactor, overhead, receiver, and all transfer lines must be free from contamination of other products. No cleanup is required if this lot follows FM-3206, FM-3256, or FM-4569. If this lot follows any other lot, a hot caustic water flush of the system followed by a water rinse is required.

PPE Note: Refer to attachment 1 for the PPE requirements for handling RM-244.

Vacuum charge two drums of 11-0000-0244-1, 50% Sodium Hydroxide, to the reactor. Start the agitator at 60 rpm. Fill the reactor with water.

Caution: When flushing the reactor and overhead system with caustic and water, do not bump material through the carbon packing. The carbon packing is very brittle and bumping may damage the packing.

Set batch setpoint to 250 °F, the jacket high limit to 350 °F and the delta T limit to 300 °F. Set the jacket mode to direct steam and heat the water. When the reactor temperature reaches 205 - 210 °F, start adding water to the reactor at a controlled rate, maintaining a batch temperature of 200 - 210 °F. Overflow the reactor through the packed column, overhead condenser and allow to flow to the receiver. After flushing the overhead for 1 hour, cool to 130 °F. Put jacket in neutral and drain overhead lines, reactor, and receiver to the sewer. Refill the reactor with water, and repeat the flush through entire system with water for 1 hour. Drain overhead lines, receiver and reactor to the sewer.

Open the reactor and receiver manholes and inspect. Contact supervisor if additional cleanup is necessary.

3. Pressure the reactor system including overhead and receiver to 40 psig with nitrogen. The pressure loss should be less than 1 psig after 30 minutes. Find and repair leaks as required. Use water to hydrostat system if necessary to find leaks.
4. Test the vacuum control system. It must be able to control at 10 mm or less absolute pressure before proceeding.

5. Check the D.P. lines to make sure they are open and not plugged:
 - a. Put the control mode to D.P. and make sure the reactor is either on vacuum or at 0 psig and vented.
 - b. Set the jacket to Circ Water. (The conditions in 5(a) and 5(b) will open the shutoff valves to the D.P. lines. The valves will be shut under the following conditions, jacket control in neutral, Batch control with either Circ Water or Steam, and when reactor pressure exceeds 7 psig).
 - c. Go to 3rd floor and observe the small rotameters on the purge lines to the D.P. lines. There should be flow of nitrogen visible. Adjust the rotameters to midscale if necessary. Observe the rotameters for several minutes to make sure the readings are stable.
 - d. If there is no flow or if there was flow initially and the flow stopped it means the corresponding D.P. line is plugged. High pressure D.P. line means the line on the bottom of the column. Low pressure line means the line to the top of the packed column.
 - e. If there is flow through the rotameters one more test is required to determine if the lines are clear and there is not a leak in the tubing going to the D.P. lines. Put the jacket control in neutral to close the solenoid valves to the D.P. lines. Now watch the rotameters. If flow continues through the rotameters then there is a leak in the tubing. If the flow stops--(it may take a couple of minutes for the longer high pressure side D.P. line)--then the line is OK.
 - f. If the D.P. lines do not pass this test take steps to blow nitrogen or steam through the lines to clear the lines. If you cannot clear the lines call maintenance and have a fitter clean/repair the line. The most likely place for a plug is in the lower section of the line on the top of the reactor.
 - g. The D.P. control will not work if the lines are not clear.

6. Valve the overhead to by-pass the decanter. Open the by-pass valve in the liquid seal loop in the reflux return line. Close valves in condensate return line to the receiver. Condensate return should be routed to reactor for startup.

II. INERT STRIP *

7. Put the reactor jacket in circulating water with batch setpoint of 120°F, a jacket high limit of 350°F and a delta T limit of 100°F.

Put the receiver in emergency cooling. Turn on condenser water to "COOLING" and set water exit temperature setpoint at 55°F.

8. Add Charge A (41-2700-3206-7), Stabilized Cell Product, to the reactor. Use nitrogen pressure to transfer Charge A from Storage Tank 15.

PPE Note: Refer to attachment 1 for the PPE requirements for handling FM-3206.

9. Vent reactor to scrubber. Open reactor sightglass and add Charge B (11-0000-3179-6), Filter Cel, to the reactor. Close and tighten down sightglass. Turn on agitator and adjust speed to 70 RPM.

PPE Note: Refer to attachment 1 for the PPE requirements for handling RM-3179.

10. Set the system for distillation as follows:
- Set the overhead for atmospheric distillation of inerts. Route condensate takeoff to return to reactor during heatup.
 - Verify the jacket is in "CIRC-WATER" and set the batch control mode to D.P.
 - Set the ΔT at 50°F. **Adjust** ΔT as required to maintain a 1,200-1,800 lb/hr take-off rate.
 - Set the **D.P.** setpoint to 100 mm. This will *record* the D.P., but the ΔT will be the limiting set point.
 - Set the jacket high limit at 350°F.
 - Set the reflux timers for **10 seconds takeoff** and **5 seconds reflux**. (Reflux ratio 0.5 to 1.) Set the splitter valve to REFLUX/TAKEOFF.
11. Distill off FM-3160 inerts to the receiver at a rate of 1,200-1,800 lbs/hr. Adjust the ΔT setpoint to maintain the takeoff rate. Increasing ΔT will increase the jacket temperature, boil up rate and take-off rate. Decreasing ΔT will decrease jacket temperature and boil up rate.

12. Allow at least 10,000 - 12,000 lbs. (4,000 lbs for BC-33), of FM-3160 distillate to collect in the receiver before draining.

Verify the outside inert storage tank level is less than 96%, and that the pressure in the storage tank is less than 40 psia. If the level is greater than 96%, the inerts will have to be drummed. Refer to by-product draining section of FOP for drum to use. If the storage tank pressure is greater than 40 psia, vent the storage tank through the reactor overhead system with condenser water on at 55 °F.

If the storage tank pressure is less than 20 psia, and the automatic nitrogen block valve is open, investigate the status of the tank. If necessary, perform a pressure test on the tank and transfer lines before initiating the transfer of inerts.

An automatic valve is on the inert charge line on top of the outside storage tank. If the tank level is above 98%, or if the tank pressure is above 50 psia, the charge valve will automatically close. Verify that the valve is open, and open if necessary.

Use nitrogen to pressure the receiver to 30 psig. **Do not exceed 35 psig of nitrogen pressure.** Pressure transfer the distillate as FM-3160 to the outside inert storage tank. **Leave a 1,000 lb heel of inerts in the receiver** so the tank is not pressured up with excess nitrogen pressure. Also, there can be an upper water phase in the receiver. Water should not go to the outside inert storage tank. Watch for the water phase in the receiver during the inert transfer.

When the inert transfer is complete, vent the receiver pressure slowly through the reactor overhead.

Record the amount of FM-3160 inerts drained on the Byproduct Yield Card, whether drained to the bulk tank or to drums. If the tank is full or not operational, drain from the receiver to polyoverpaks but maintain a 1,000 lbs. heel in the receiver to allow for phase separation of any water. Drain any water phase as By-Product 41-2600-6002-9 and remove.

PPE Note: Refer to attachment 1 for the PPE requirements for handling F-6002 and FM-3160.

13. As each 1,200-2,400 lbs of FM-3160 is distilled, add 1,200-2,400 lbs of Charge C (41-2700-3206-7) to the reactor. (Add what you distill). Smaller more frequent additions are preferable to large slugs.

PPE Note: Refer to attachment 1 for the PPE requirements for handling FM-3206.

14. Continue to distill inerts to the receiver and add additional Charge C to the reactor until the reactor is full or until storage tank 15 is empty and all scheduled FM-3206 has been added. If there is an amount of Charge D scheduled, charge after all of Charge C has been added. Add Charge D (41-2700-3210-9) by first charging to storage tank 15 followed by pressuring to the reactor.

PPE Note: Refer to attachment 1 for the PPE requirements for handling FM-3210.

Increase the ΔT as necessary to maintain a take off rate of **1,200-1800 lb/hr**. At rates below 1200 lb/hr, the column efficiency decreases.

When the **head** temperature reaches **210 °F**, set the reflux timers for **5 sec takeoff** and **10 sec reflux**.

When the **batch** reaches **240-250°F**, drain the jacket, and switch the jacket to **direct steam**.

15. When the **reactor** temperature reaches **285°F** and no more Charge C or D remains to be charged (or the reactor is full), set the jacket to CIRC-WATER and BATCH-AUTO and cool the batch to 150°F.
16. While cooling to 150°F, maintain splitter valve settings, but route distillate to return to reactor.
17. Drain the balance of the receiver. Drain the bottom inert phase as FM-3160. If the next scheduled run is **not** FM-3206 or FM-3256, save 1,200 lbs of FM-3160 for later use as Charge H. Drain the top water phase as F-6002. See By-Product Draining Information.

PPE Note: Refer to attachment 1 for the PPE requirements for handling F-6002 and FM-3160.

III. FIRST PRE-CUT (100 mm Hg up to 245 °F head temperature)

NOTES:

- o The first pre-cut will be taken as a total cut in the receiver and then drummed.*
 - o Sample the precut as FM-3256, Dr ____, **using consecutive drum numbers** throughout the fractionation. Do not include drums of F-6002 drained when numbering drums of pre-cut. Discard all unused labels.*
 - o Label each drum according to C8 and HB content as specified on the QC page. If the C8 content is less than 10%, the QC lab will only report the C8 value. Drums with less than 10% C8 are scrap.*
 - o While draining the receiver, maintain vacuum on reactor and column, and continue to operate column with the same reflux timer settings, routing take-off to the reactor. When draining is complete, use single stage vacuum to pull vacuum on receiver, before switching back to BC-34's vacuum on the receiver. After re-establishing the normal vacuum level and a steady head temperature, switch take-off back to receiver.*
18. While cooling to 150°F, maintain splitter valve settings, with distillate routed to reactor. Pull vacuum on reactor system for first precut. Set the vacuum loop to AUTO and lower the vacuum to **100 mm Hg** in **50 mm** increments over a 30 minute period. Vacuum must be lowered slowly to prevent boilovers.
19. When the vacuum is **100 mm Hg** or less, start heating the batch as follows:
- a. Put the jacket in "CIRC WATER".
 - b. Set the control mode to "COL DP".
 - c. Set the ΔT at 50-100°F. Increase as necessary to maintain 300-400 lb/hr takeoff rate.
 - d. Set the **D.P.** setpoint to 100 mm. This will record the **D.P.**, but ΔT will be the limiting set point.
 - e. Set the jacket high limit at 350°F.
 - f. Set the splitter for **5** seconds takeoff and **20** seconds reflux (Reflux ratio 4:1) but route takeoff back to reactor to keep the line clear.

20. When the head temperature is stable for 10 minutes, start takeoff to the receiver. Leave the splitter set at 5 seconds takeoff and 20 seconds reflux. Monitor take-off rate and adjust the delta T setpoint to maintain a take-off rate of **300-400 lbs/hr**.

When the batch reaches 240-250°F, drain the jacket, and switch the jacket to "STEAM" mode.

21. Distill precut to the receiver until the head temperature reaches **245°F at 100 mm Hg**. Expect 1,500 to 3,500 lbs of distillate in the receiver for normal batches.
22. When the precut is complete, switch vacuum directly to reactor, through condenser and packed column. Continue to operate column with the same reflux timer settings, routing take-off to the reactor. Isolate receiver from reactor and break vacuum on the receiver with nitrogen. Pressure receiver to 20 psig with nitrogen.

PPE Note: Refer to attachment 1 for the PPE requirements for handling FM-3210, F-6002, and Sample 1.

Drain the precut to polyoverpak drums, watching for an upper water phase during draining. Take one, 2-ounce (in a 4-ounce bottle) in-process sample. Label as FM-3256 Lot __, Dr __, "**SAMPLE #1**".

If there is a water phase, drain it as byproduct 41-2600-6002-9 as in Step 12. Do not include the F-6002 byproduct as part of the Drum numbers in the fractionation.

When draining is complete, use single stage vacuum to pull maximum vacuum on receiver.

Label the drums based on QC results (as specified at the end of the Maincut Draining Information).

23. After draining the precut, add any scheduled Charge E (41-2700-3257-0), 2nd Precut, to the receiver by vacuum. The maximum charge that will fit is 3,000 - 4,000 lbs.

PPE Note: Refer to attachment 1 for the PPE requirements for handling FM-3257 and RM-3048.

Add Charge F (11-0000-3048-3), Sulfuric acid to the receiver by vacuum. Pressure receiver to 20 psig and transfer to the reactor. Again use single stage vacuum system to pull maximum vacuum on the receiver. Isolate receiver from single stage vacuum system then switch back to BC-34's vacuum system on the receiver. Use delta T setpoint of 15°F while vacuum pulls down and column D.P. levels. Reset delta T setpoint back to 100°F after column D.P. levels out.

IV. SECOND PRECUT (10 mm Hg up to Product cut purity))

NOTES:

- o The second precut will be taken off in 2 drum drainings until 96% C8. When the C8 is greater than 96%, the product will be taken as one cut until near the end. The maincut should be drained before getting near the end. This will eliminate getting too many high boilers in the maincut. The key variables to watch for ending the main cut are a dropping D.P. and an increase in steam pressure.*
 - o Feel temperature of reflux return line once per hour and note. This line must remain warm while in intercut and main cuts. If a line plugs with C8, blow it out with nitrogen or melt it.*
 - o While draining the receiver, maintain vacuum on reactor and column, and continue to operate column with the same reflux timer settings, routing take-off to the reactor. When draining is complete, use single stage vacuum to pull vacuum on receiver, before switching back to BC-34's vacuum on the receiver. After re-establishing the normal vacuum level and a steady head temperature, switch take-off back to receiver.*
24. Lower the vacuum set point to **10 mm Hg**. Set the reflux splitter to **5** seconds takeoff and **30** seconds reflux (Reflux ratio 6: 1).
 25. Turn on steam to the tracing. Set condenser water setpoint to **80°F**. Put the receiver in BATCH AUTO and set to WARM-WATER. Set batch setpoint to 130°F and the jacket high limit to 200°F. Monitor receiver temperature hourly to verify that the temperature is holding close to 130°F.
 26. Verify reactor jacket is set to DIRECT STEAM and the control mode to D.P.. Raise the jacket high limit to 380°F. Set the ΔT setpoint at 100°F.
 27. Adjust the D.P. to 50-80 mm Hg to establish a steady and strong take-off. Maintain take-off to the reactor until the head temperature is stable for 10 minutes.
 28. When the head temperature is stable, start take-off to the receiver. Raise the condenser temperature slowly throughout the intercut until the condenser temperature reaches 120 °F.

Condenser water note:

Raising the condenser water temperature too fast may make it impossible to attain the desired 10 mm Hg vacuum. A good guide to raising the condenser water temperature is: Condenser water temperature should be no higher than (Column head temperature - 90 °F). For example, if the head temperature is 190 °F, the condenser water temperature should not be higher than 100 °F.

29. Continue the second precut distillation to the receiver. Monitor take-off rate and adjust the column D.P. setpoint to maintain a take-off rate of **400-500 lbs/hr**.

Drain the receiver every two drums per instructions below until 96% purity is reached.

Switch vacuum directly to reactor, through condenser and packed column. Continue to operate column with the same reflux timer settings, routing take-off to the reactor. Isolate receiver from reactor and break vacuum on the receiver with nitrogen. Pressure receiver to 20 psig with nitrogen.

PPE Note: Refer to attachment 1 for the PPE requirements for handling FM-3257, F-8281, FM-3256, and Sample 1.

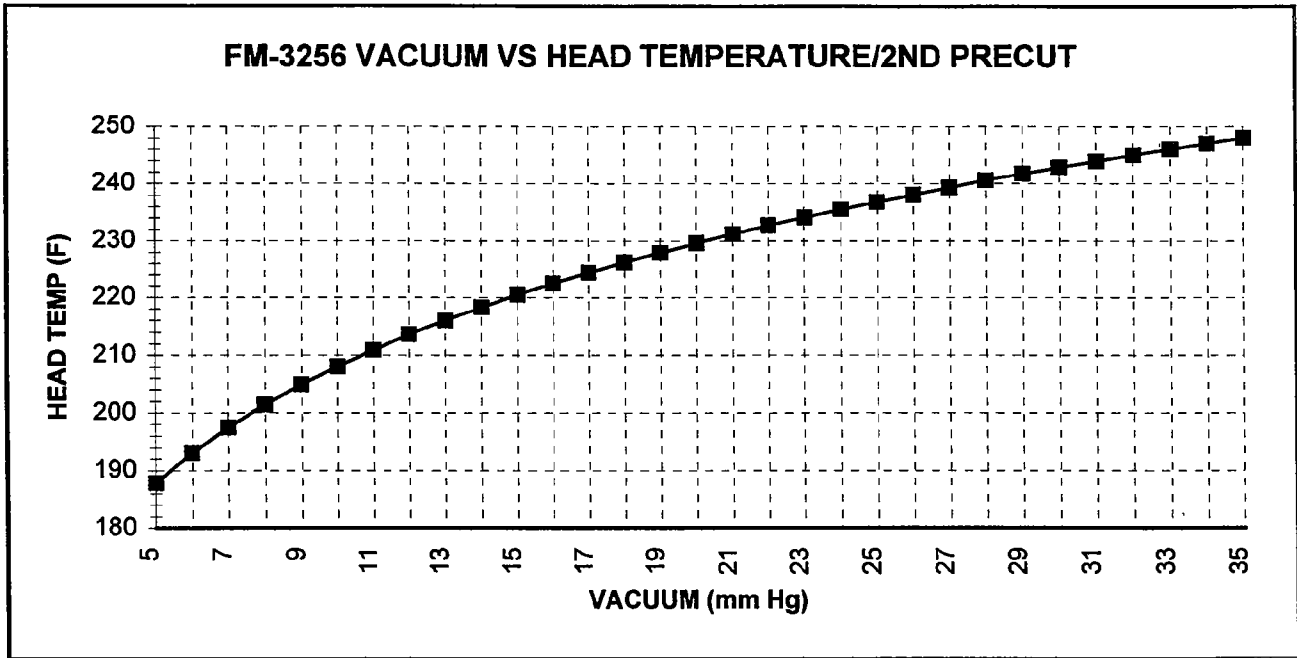
Drain the precut to **new** polyoverpak drums. Take one, 2-ounce (in a 4-ounce bottle) in-process sample. Label as FM-3256 Lot ___, Dr ___, "**SAMPLE #1**".

When draining is complete, use single stage vacuum to pull maximum vacuum on receiver. Isolate receiver from single stage vacuum system then switch back to BC-34's vacuum system on the receiver. Use delta T setpoint of 15^oF while vacuum pulls down and column D.P. levels. Reset delta T setpoint back to 100^oF after column D.P. levels out.

Label the drums based on QC results (as specified at the end of the Maincut Draining Information).

When the % C8 of the last draining is 96% or greater, proceed to Product Cut.

The following graph shows the head temperature of high purity C8 versus the vacuum. If the insulation on the column vapor line is good this line will approximate the head temperature at the end of the 2nd precut and beginning of the product cut.



V. PRODUCT CUT

NOTES:

- o The product cut will be taken as a total cut to the receiver and then drummed. **However, do not wait so long in draining that you get HB into the maincut. It is imperative that the operator monitor the jacket steam pressure and the D.P. to determine the first sign of the end of the maincut. End the maincut as soon as any one of these signals the end of the cut.***
- o Use D.P. control to fractionate the inter and main cuts. The ΔT set point can be used to limit the jacket temperature and swings in steam pressure.*
- o Feel temperature of reflux return line once per hour and note if it is cooled. If a line plugs with C8 blow it out with nitrogen or melt it.*
- o While draining the receiver, maintain vacuum on reactor and column, and continue to operate column with the same reflux timer settings, routing take-off to the reactor. When draining is complete, use single stage vacuum to pull vacuum on receiver, before switching back to BC-34's vacuum on the receiver. After re-establishing the normal vacuum level and a steady head temperature, switch take-off back to receiver.*
- 30. Set the splitter for 5 seconds takeoff and 30 seconds reflux. (Reflux ratio 6:1). Set the ΔT setpoint at 100°F.
- 31. Adjust the D.P. to 50-80 mm Hg to establish a steady and strong take-off. Maintain take-off to the reactor until the head temperature is stable for 10 minutes.
- 32. When the head temperature is stable, start take-off to the receiver. The condenser temperature should remain at 120 °F.

33. To maintain the proper take-off rate (and good fractionation) it will be necessary to make adjustments in the *D.P.* setpoint. Maintain a product takeoff rate of **400-500 lb/hr**. Rate will drop below this near the end of the cut.

Continue the product cut distillation to the receiver until:

- a. The jacket steam pressure rises sharply and remains above 90 psig, or
- b. The jacket temperature rises to 300°F, or
- c. The *D.P.* drops off from its steady state value.

Any one of these three items can signal the end of the large product cut where you should drain the receiver.

When the receiver requires draining, switch vacuum directly to reactor, through condenser and packed column. Continue to operate column with the same reflux timer settings, routing take-off to the reactor. Isolate receiver from reactor and break vacuum on the receiver with nitrogen. Pressure receiver to 20 psig with nitrogen.

PPE Note: Refer to attachment 1 for the *PPE* requirements for handling *FM-3256*, and *Sample 1*.

Drain the product cut to new polyoverpak drums. Take one, 2-ounce (in a 4-ounce bottle) in-process sample. Label as *FM-3256* Lot ___, Dr ___, "***SAMPLE #1***".

When draining is complete, use single stage vacuum to pull maximum vacuum on receiver. Isolate receiver from single stage vacuum system then switch back to *BC-34*'s vacuum system on the receiver. Use delta T setpoint of 15°F while vacuum pulls down and column *D.P.* levels. Reset delta T setpoint back to 100°F after column *D.P.* levels out.

34. Continue on product cut fractionation, draining in two drum increments until the high boilers in the product exceeds 3.0%. Drain receiver per step 33.

When the high boilers in the product exceeds 3%, proceed as follows. If an amount of Charge G is specified on the run card, proceed to section **VI. FIRST POST CUT**. Otherwise, proceed to section **VII. SECOND POST CUT (TOTAL TAKEOFF)**.

VI. FIRST POST CUT

NOTES:

- o This cut is only done if F-4169 is added to the reactor. Normally you will continue on maincut until end of the maincut is signalled in Step 34 and then go to total takeoff per step 40.*
 - o Feel temperature of reflux return line once per hour and note if not hot . Clear the line with nitrogen or by melting if necessary.*
 - o The first post cut will be taken to the receiver and drummed as each 600 lb is collected.*
 - o While draining the receiver, maintain vacuum on reactor and column, and continue to operate column with the same reflux timer settings, routing take-off to the reactor. When draining is complete, use single stage vacuum to pull vacuum on receiver, before switching back to BC-34's vacuum on the receiver. After re-establishing the normal vacuum level and a steady head temperature, switch take-off back to receiver.*
35. Isolate the receiver from the reactor and switch vacuum to reactor overhead. Maintain splitter valve settings with distillate routed to the reactor. Add any scheduled Charge G (41-2600-4169-8), Postcut, to the receiver by vacuum. Pressure receiver to 20 psig and transfer to the reactor. Use single stage vacuum system to pull maximum vacuum on the receiver. Isolate receiver from single stage vacuum system then switch back to BC-34's vacuum system on the receiver. Use delta T setpoint of 15°F while vacuum pulls down and column D.P. levels. Reset delta T setpoint back to 100°F after column D.P. levels out.
- PPE Note:** Refer to attachment 1 for the PPE requirements for handling F-4169.
36. Set the splitter for 5 seconds takeoff and 30 seconds reflux. (Reflux ratio 6:1). Set the ΔT setpoint at 100°F.
37. Adjust the D.P. to 50-80 mm Hg to establish a steady and strong take-off. Maintain take-off to the reactor until the head temperature is stable for 10 minutes.
38. When the head temperature is stable, start take-off to the receiver. Maintain the condenser temperature at 120 °F.

39. To maintain the proper take-off rate (and good fractionation) it will be necessary to make adjustments in the *D.P.* setpoint. Maintain a product takeoff rate of **275-350 lb/hr**. Rate will drop below this near the end of the cut.

When 600 lbs of distillate has been collected in the receiver, switch vacuum directly to reactor, through condenser and packed column. Continue to operate column with the same reflux timer settings, routing take-off to the reactor. Isolate receiver from reactor and break vacuum on the receiver with nitrogen. Pressure receiver to 20 psig with nitrogen.

PPE Note: Refer to attachment 1 for the PPE requirements for handling F-8282, and Sample 1.

Drain the product to new polyoverpak drums. Take one, 2-ounce (in a 4-ounce bottle) in-process sample. Label as FM-3256 Lot____, Dr____, "**SAMPLE #1**".

When draining is complete, use single stage vacuum to pull maximum vacuum on receiver. Isolate receiver from single stage vacuum system then switch back to BC-34's vacuum system on the receiver. Use delta T setpoint of 15°F while vacuum pulls down and column D.P. levels. Reset delta T setpoint back to 100°F after column D.P. levels out.

40. Continue fractionation, draining in one drum increments until the high boilers in the product exceeds 3.0%. Drain receiver per step 39.

When the high boilers in the product exceeds 3%, proceed to section **VII. SECOND POST CUT (TOTAL TAKEOFF)**.

VII. SECOND POST CUT (TOTAL TAKEOFF)

NOTES

- o The second post cut cut will be taken as a total cut to the receiver and then drummed.*
- o Feel temperature of reflux return line once per hour and note if not hot. Clear the line with nitrogen or by melting if necessary.*
- 41. Set the vacuum to 1 mm to get maximum vacuum. Set the splitter for total takeoff. Set the ΔT setpoint at **100°F**.
- 42. Adjust the *D.P.* to 50-80 mm Hg to establish a steady take-off. Maintain take-off to the reactor until the head temperature is stable for 10 minutes.
- 43. When the head temperature is stable, start take-off to the receiver. Maintain the condenser temperature at 120 °F.
- 44. Distill to get as much C8 Acid/HB out of the bottoms as possible. When the take-off rate falls below 30 lbs per hour and the batch temperature rises to 350°F with maximum vacuum, stop the distillation. Isolate receiver from reactor and break vacuum on the receiver with nitrogen. Pressure receiver to 20 psig with nitrogen.

PPE Note: Refer to attachment 1 for the PPE requirements for handling F-4169, and Sample 1.

Drain the product to polyoverpak drums. Take one, 2-ounce (in a 4-ounce bottle) in-process sample. Label as FM-3256 Lot __, Dr __, "**SAMPLE #1**".

VIII. FLUOROCARBON FLUSH

NOTE: Omit steps 45-49 if FM-3206 or FM-3256 follows this lot.

45. After ending the postcut, switch jacket to circulating water, batch control mode to batch auto, and start cooling the batch to 200°F.
46. Isolate system at full vacuum. Add Charge H (41-2700-3160-6) to the receiver and pressure transfer to the isolated reactor.

PPE Note: Refer to attachment 1 for the PPE requirements for handling FM-3160.

47. Break vacuum on system with nitrogen. Leave condenser water temperature at 120°F. Put 50°F water on the receiver jacket. Open receiver vent to scrubber.
48. Set overhead valving for total take-off to receiver. Set jacket high limit to 350°F, and delta T limit to 50°F.
49. Distill the flush to the receiver at a rate of 400-600 lbs/hour. End distillation when take-off rate is 30 lbs/hr or less. Transfer the inert flush to ST-15 using nitrogen pressure. If the storage tank is not available, drain to polyoverpak drums as FM-3388. Only record as by-product FM-3388 if drained to drums.

IX. DRAINING BOTTOMS

50. Turn on cooling water to the reactor and cool batch to 150°F. Break vacuum with nitrogen and pressure to 10 psig. When the temperature has dropped to 200 °F, slowly add water to the reactor. At first, it will exotherm, then will cool. Fill reactor half full with water. Open the drain valve and flush the sulfuric acid bottoms to the sewer with water.

XI. POST RUN CLEANUP

NOTE: No cleanup is necessary if a FM-3206 or FM-3256 is to follow.

51. **PPE Note:** *Refer to attachment 1 for the PPE requirements for handling RM-244.*

Vacuum charge two drums of **11-0000-0244-1**, 50% Sodium Hydroxide, to the reactor. Start the agitator at 60 rpm. Fill the reactor with water.

Caution: *When flushing the reactor and overhead system with caustic and water, do not bump material through the carbon packing. The carbon packing is very brittle and bumping may damage the packing.*

Set batch setpoint to 250 °F, the jacket high limit to 350 °F and the delta T limit to 300 °F. Set the jacket mode to direct steam and heat the water. When the reactor temperature reaches 205 - 210 °F, start adding water to the reactor at a controlled rate, maintaining a batch temperature of 200 - 210 °F. Overflow the reactor through the packed column, overhead condenser and allow to flow to the receiver. After flushing the overhead for 1 hour, cool to 130 °F. Put jacket in neutral and drain overhead lines, reactor, and receiver to the sewer. Refill the reactor with water, and repeat the flush through entire system with water for 1 hour. Drain overhead lines, receiver and reactor to the sewer.

Open the reactor and receiver manholes and inspect. Contact supervisor if additional cleanup is necessary.

MAINCUT DRAINING INFORMATION:

Containers: New black poly overpacks (34-7039-5732-3)
Labeling: 41-2700-3256-2, Lot, ___Net___, Drum___, Inside
Storage: Hold in Bldg. 15 Hot Room for F-7164.
Filter/Alternate: None
Weight Per Container: 600 lbs.
Draining Temperature: 120-140°F
Draining Pressure: 0 to 2 psig

Special Draining Instructions:

To avoid C8 set up in the drain line, blow the drain line clear from the bottom drain valve to the draining drum. Then suck through the drain line to the receiver briefly before shutting drain valve.

Sample Requirements:

Inerts: 41-2700-3160-6 None
Precut: 41-2700-3210-9 1, 2-ounce in-process sample from each drum or draining.
Intercut: 41-2700-3257-0 1, 2-ounce in-process sample from each drum or draining.
Maincut: 41-2700-3256-2 1, 2-ounce in-process sample from each 41-2600-8281-7 or 41-2600-8282-5 drum or draining.
Postcut: 41-2600-4169-8 1, 2-ounce in-process sample from each drum or draining.

Labeling Information For All Cuts:

<u>% C8</u>	<u>% HB</u>	<u>Label as:</u>	<u>Description:</u>
0.0 - 9.9	0	13-0017-0281-3	Scrap
10.0 - 69.9	0	41-2700-3210-9	First Precut
70.0 - 89.9	0	41-2700-3257-0	Second Precut
<u>Product Cuts</u>			
90.0 - 96.0	0	41-2600-8281-7	Product Cut
96.1 - 100.0	0.0 - 0.5	41-2700-3256-2	Product Cut (Heart cut)
97.0 - 99.4	0.6 - 3.0	41-2600-8282-5	Product Cut
<u>Postcut</u>			
10.0 - 96.9	3.1 - 89.9	41-2600-4169-8	Postcut
0.0 - 10.0	90-100	13-0017-0281-3	Scrap

BY-PRODUCT DRAINING INFORMATION:

Step: 12, 17

Description: C8 Cyclic Inert Mixture

Labeling: Outside inert tank: None (Record byproduct yield on production card).
Drums: 41-2700-3160-6, Lot ____, Net ____, Dr ____, Inside.

Wt per Container: 600 lbs net for drums

Container/Disposition: Pump/pressure to outside inert tank. If drummed use new or remanufactured poly overpak drums (Refer to list of suitable recycle drums at end of ByProduct Section). Remove drums to warehouse.

Amount: 20,000 - 30,000 lbs.

Step: 12, 17

Description: Water phase from inerts

Labeling: 41-2600-6002-9, Lot ____, Net ____, Dr ____, Inside.

Wt per Container: 400 lbs net.

Container/Disposition:

1. Recycle poly overpak drums (34-7010-1156-0) (Refer to list of suitable recycle drums at end of ByProduct Section).
2. Remanufactured poly overpak drums (34-7029-4109-6)
3. New poly overpaks (34-7002-2745-6)
/Remove to warehouse at end of series

Amount: Up to 4,000 lbs.

BY-PRODUCT DRAINING INFORMATION: (continued)

Step: 22

Description: First Precut

Labeling: 41-2700-3210-9, Lot ____, Net ____, Dr ____

Wt per Container: 600 lbs net

Container/Disposition:

1. Recycle poly overpak drums (34-7010-1156-0) (Refer to list of suitable recycle drums at end of ByProduct Section).
2. New black poly overpak drums (34-7039-5732-3)
/Remove to warehouse at end of series

Amount: 1,800-2,400 lbs

Step: 29

Description: Second Precut

Labeling: 41-2700-3257-0, Lot ____, Net ____, Dr ____, Inside

Wt per Container: 600 lbs net

Container/Disposition:

1. New Black Polyoverpaks (34-7039-5732-3)
2. Used FM-3256 drums
3. New poly overpaks (34-7002-2745-6)
/Remove to bldg 15 hot room.

Amount: 1,200-1,800 lbs.

BY-PRODUCT DRAINING INFORMATION: (continued)

Step: **33, 39**

Description: First post cut

Labeling: **41-2600-8282-5**, Lot ___, Net ___, Dr ___, Inside

Wt per Container: 600 lbs net

Container/Disposition:

1. New Black Polyoverpak drums (34-7039-5732-3)
2. Used FM-3256 poly overpak drums
2. New poly overpaks (34-7002-2745-6)
/Remove to bldg 15 hot room.

Amount: Up to 1,200 lbs.

Step: **44**

Description: Second Postcut

Labeling: **41-2600-4169-8**, Lot ___, Net ___, Dr ___, Inside.

Wt per Container: 600 lbs net

Container/Disposition:

1. Recycle poly overpak drums (34-7010-1156-0) (Refer to list of suitable recycle drums at end of ByProduct Section).
2. New poly overpaks (34-7039-5732-3)
/Remove to warehouse at end of series.

Amount: Up to 1,200 lbs.

BY-PRODUCT DRAINING INFORMATION: (continued)

Step: 22 or 44

Description: Pefluorooctanoic Acid (< 10% C8) in Inerts for Scrap Disposal

Labeling: 13-0017-0281-3, (Refer to Waste Stream Profile)

Wt per Container: 600 lbs net

Container/Disposition: 1. Recycle poly overpak drums (34-7010-1156-0) (Refer to list of suitable recycle drums at end of ByProduct Section).
2. Remanufactured poly overpak drums (34-7029-4109-6)
3. New poly overpaks (34-7002-2745-6)/Incinerator

Amount: Up to 1,200 lbs.

Step: 49

Description: Fluorocarbon flush

Labeling: 41-2700-3388-3, Lot ____, Net ____, Dr ____, Inside.

Wt per Container: 600 lbs net

Container/Disposition: 1. Recycle poly overpak drums (34-7010-1156-0) (Refer to list of suitable recycle drums at end of ByProduct Section).
2. Remanufactured poly overpak drums (34-7029-4109-6)
3. New poly overpaks (34-7002-2745-6)

Amount: 1,200 lbs

RECYCLE DRUMS FOR BY-PRODUCTS

BY-PRODUCT FM-3160 (if drummed)	FOR FM-3206 FM-3210, F-4169 or Scrap
USE DRUMS FROM LIST BELOW	USE DRUMS FROM LIST BELOW
1st Choice - New Drums	
2nd Choice - Remanufactured Drums	FM-3206
3rd Choice from list below	FM-3210
FM-3129	FM-3256
FM-3719	FM-3257
FM-3160	F-4169
FM-3144	F-8281
F-6566	F-8282
F-6567	F-8420--> F-8424

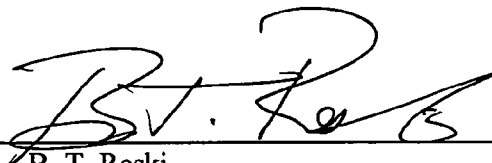
WASTE DISPOSAL:

Step No.: **50**
 Description: Sulfuric Acid Bottoms
 Stenciling/Labeling: None
 Container/Disposition: Drain to chemical sewer
 Amount: 500 lbs.

Step No.: **51**
 Description: Dilute Sodium Hydroxide Solution
 Stenciling/Labeling: None
 Container/Disposition: Drain to chemical sewer
 Amount: Up to 8,000 lbs.

AUTHOR:

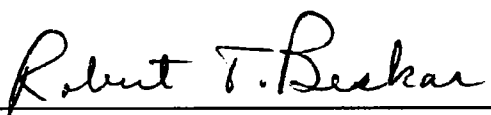
Manufacturing Engineer:



B. T. Reski

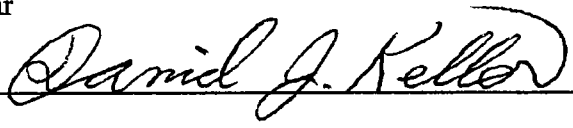
APPROVED BY:

Quality Representative:



R. T. Beskar

Product Manager\Technical Supervisor:



Health & Safety Eng.: Yes _____ No

Environmental Coordinator: _____ Yes No _____

br\fm3256.r02
Copies:
G. A. Groeneveld - 208-1C-01

PERSONAL PROTECTIVE EQUIPMENT REQUIREMENTS FOR PRODUCT NUMBER: 41-2700-3256-2

Engineer: Brian Reski

Hazard Level Definitions:

- E=Extreme: Likely to cause irreversible tissue damage, serious illness or death from contact or exposure with a very small amount.
- H=High: May cause extreme irritation, irreversible tissue damage, serious illness or death from single or repeated contact or exposures.
- M=Medium: May cause moderate to severe irritation; or may cause allergic reaction (sensitization); or may cause reversible systemic effects.
- L=Low: May cause mild temporary irritation on contact or exposure; no cumulative effects are expected from repeated contact or exposures.

NA = Not Applicable

11-Digit Code	Material Name	PHYSICAL DATA		HAZARD LEVEL			EYES		SKIN			LUNGS			Comments
		Physical State	Flammable (or handled above flashpoint)* Flashpoint (oF)	Eyes	Skin	Lungs	Goggles	Face shield	Gloves	Rubber jacket	Rubber suit & boots	Spot ventilation	Dust mask	Cartridge respirator	
41-2700-3206-7	STABILIZED PERFLUORO-OCTANOIC ACID IN INERTS	S	NA	H	H	M	X	X	Neoprene	X	X				3
11-0000-3179-6	Celite 500 (Filter cell; Diatomaceous earth)	S	NA	L	L	M			Cloth		X	X			1
41-2700-3160-6	CRUDE C8 INERTS	L	NA	M	M	M	X	X	Neoprene		X				3
41-2600-6002-9	C8 AMMONIUM SALT DISSOLVED IN WATER	L	NA	M	M	L			Neoprene						
41-2700-3210-9	PRECUT FROM FM-3256	L	NA	H	H	M	X	X	Neoprene	X	X				
41-2700-3257-0	INTERCUT FROM FM-3256	S	NA	H	H	M	X	X	Neoprene	X	X				2
41-2600-8281-7	FRACTIONATED C8 ACID - MAIN CUT	S	NA	H	H	M	X	X	Neoprene	X	X				2
41-2700-3256-2	FRACTIONATED C8 ACID - MAIN CUT	S	NA	H	H	M	X	X	Neoprene	X	X				2
41-2600-8282-5	FRACTIONATED C8 ACID - MAIN CUT	S	NA	H	H	M	X	X	Neoprene	X	X				2
41-2600-4169-8	POST-CUT FROM FM-3256	S	NA	H	H	M	X	X	Neoprene	X	X				2
11-0000-3048-3	Sulfuric Acid	L	NA	H	H	H	X	X	Neoprene	X	X		Yellow		
11-0000-0244-1	Sodium hydroxide	L	NA	H	H	M	X	X	Neoprene	X					
Sample 1	FRACTIONATED C8 ACID	S	NA	H	H	M	X	X	Neoprene	X	X				2
Comment 1	HMIS 0-0-0														
Comment 2	Solid at room temperature. Melts at 120 F.														
Comment 3	Normally handled as a liquid.														
	Normally handled with closed system. Use specified PPE if handling open containers														

**3M COTTAGE GROVE PLANT
CHEMICAL PLANT**

FACTORY OPERATING PROCEDURE

Effective Date: November 17, 1997
Superseding: March 1, 1996

CODE NUMBER:

~~41-2600-7164-6~~

REVISION NO.: R01

SUBJECT:

ONE-PLATED PERFLUORO-OCTANOIC ACID

YIELD:

2850 Lbs

ESTIMATED MACHINE TIME: 30 Hours

EQUIPMENT:

1. BC-32, Dept. 3060, 300-gallon Hastelloy kettle with glass overhead and 200 gallon glass-lined receiver.

ATTACHMENTS:

1. BC 32 Reactor Flowsheet
2. Automatic Distillation Operation for BC -32
3. PPE requirements for the F-7164 process.
4. Safe operating limits for the F-7164 process in BC -32

ITEMS NEEDING SPECIAL INSTRUCTIONS:

1. Distillation rates over 600 lbs/hr should be considered bump-overs and redistillation must be conducted.
2. Bottoms must be drained per Waste Disposal section. Do not drain to chemical sewer if bottoms contain dichromate from last one-plate distillation.
3. Do Not boil DI water through the glass-lined receiver. This may cause premature glass failure. Follow the cleanup instructions included in the cleanup section.
4. In order to help prevent vacuum system plugs, do not vacuum charge Dichromate (charge C) or Filter cell (charge B). These charges should be slowly added through the BC 32 sight glass and mixed into the batch before restarting the vacuum system.

REASON & DOCUMENTATION OF CHANGE:

(Revise Product Structure? ___ Yes X No); (New or Changed Emissions? ___ Yes X No)

MOC Required: X Yes ___ No: (Reference SOP 400-012)

Review Team required (Job Function):

- X Operations: Rick Pechacek
- ___ Engineering
- ___ Maintenance
- X Process Engineering - Dean Graham
- ___ Safety
- ___ Other

1. a. Description of Change:
Revise cleanup procedure to include hot water flushing of the receiver followed by a DI water flush. To also include DI water boils through the overhead, through the reflux return line, and back to the kettle bypassing the receiver.
- b. Basis for Change:
Boiling DI water through the receive is suspected of damaging the glass-lining.
- c. Consequences (negative) of Change:
No negative consequences are foreseen.
2. a. Description of Change:
To specify not to vacuum charge the Potassium Dichromate and Filter cell charges. These charges will be added to the reactor through the sight glass.
- b. Basis for Change:
Vacuum charging these materials can contribute to plugging the vacuum ejectors.
- c. Consequences of Change:
Increased mixing time of the filter cell. Since these charges are powder, operator exposure may also be increased.
- d. Safeguards to prevent negative consequences:
Operators are instructed to wear proper PPE during charging these materials and to mix the batch for one hour prior to restarting the vacuum system.
3. a. Description of Change:
Add safe operating limits.
4. a. Description of Change:
Add PPE generator table

REFERENCE:

1. 41-2600-7164-6, Factory Operating Procedure dated 3/1/96.
2. Meetings with Building 15 Supervisors and BC-32 Operators.

END PRODUCT USE: Ammonium salt surfactants

CHARGE CALCULATIONS:

1. **Charge A:** Maximum Charge A is 3000 Lbs.

The composite of Charge A must meet the following requirements:

C8 Acid: 96.5% Minimum, Target 98%
C7 Acid: 1.4% Maximum
C6 Acid: 1.4% Maximum
High Boilers: 0.5% Maximum

2. **Charge B:** Standard Charge is ~~10~~ lbs of RM-3179.
20 lbs 1/27/96
3. **Charge C:** Standard Charge is 60 Lbs of RM-8510.
4. **Charge D:** Standard Charge D is 440 Lbs of RM-2706.

If %H₂O in Charge A exceeds 1%, increase amount of Charge D as follows:

$$\text{Additional Charge D} = (0.00334) \times (\%H_2O \text{ in Charge A}) \times (\text{Charge A})$$

If Charge A includes powder, increase amount of Charge D as follows:

$$\text{Additional Charge D} = (0.440) \times (\text{Pounds of Charge A Powder})$$

5. **Manufacturing Engineer** will provide spreadsheet printout with charge calculations and quantities.

PROCESS TOLERANCE:

1. Unless otherwise specified, record data for the process variables listed below a minimum of once per hour.
2. Unless otherwise specified, time intervals specified are a minimum time. To obtain consistent process conditions the Operator should continue processing at the specified time interval.
3. Unless otherwise specified, maintain process variations within the tolerances listed below. If unable to operate within the acceptable tolerances, contact the Supervisor or Manufacturing Engineer for instructions. Note all additional verbal instructions on the data card.

Attach all written instructions to the Production Report.

4. Unless otherwise specified in the procedure, use stencil weights for charging.

	<u>ACCEPTABLE PROCESS TOLERANCES</u>		<u>RECORD DATA</u>	
	(+)	(-)	(Y)	(N)
Agitator Speed (RPM)	5	5	Y	
Temperature (°F)				
batch	5	5	Y	
jacket	5	5	Y	
head	5	5	Y	
condenser water outlet	5	5	Y	
Pressure (psig)	5	5	Y	
Vacuum				
(mm Hg)	10	10	Y	
Charge Weight	1%	1%	Y	
Draining Weight	1%	0%	Y	

Rates

Target and tolerances are specified in the procedure.

EMERGENCY SHUTDOWN INSTRUCTIONS:

1. The process (reactor, receiver, and agitators) can be shutdown any time during the operation of the equipment by turning off the agitator, shut off vacuum system, put the receiver and reactor jackets in neutral, and completely isolate the system.
2. Once confirming that the acid in the receiver or reactor is melted, the system can be restarted at any time by following the remainder of the FOP. Contact manufacturing engineer prior to restarting the operation.

Note: Once the jackets on the receiver and reactor are put into neutral any acid contained in these vessels will begin to solidify and harden. Remelting the acid may take up to a couple of days. Confirming that the acid is melted prior to starting up the agitator is essential to prevent agitator damage.

OPERATING PROCEDURE:

I. PRE-SERIES PREPARATION

1. The operator will need to be familiar with the Provox CRT and how it is used to operate BC-32. If questions exist contact the Supervisor.
2. A general flowsheet of the BC-32 process piping is attached. The operator should review it and use it as reference during the remainder of this standard.
3. The open column will be used for this run. If not already done, valve in the open column and blank off both top and bottom of packed column.
4. Reactor, overhead condenser, large receiver, and transfer lines normally do not require cleaning since this unit is dedicated to the distillation of C8 acid. However, a clean up will need to be performed if when following an F-7117 run. If additional cleaning is required it will be requested by the Manufacturing Engineer. See instructions in PRE-SERIES CLEANUP section in the back of this FOP.

II. CHARGING *Refer to Deviation*

5. Verify the steam tracing to overhead piping and to BC-32 drain lines is hot. The steam supply valve for overhead tracing is located directly behind the overhead condenser. The steam supply valve for the BC-32 drain lines is located on first floor against the wall underneath the BC-32 reactor. Verify the steam pressure is at 7 to 10 psig.
6. Close manual valve for liquid trap in reflux return line. Open all manual valves in takeoff line leading to large receiver. Open manual valve on top of large receiver located between receiver and automatic vacuum and vent valves. Using the CRT, close the vent valve on large receiver. Using the CRT, open the vacuum valve on large receiver. Using the CRT close the cold side vent and vacuum block valve on the overhead condenser. Verify BC-36 and BC-37 are isolated from the BC-32 vacuum system.
7. Verify both water and steam to the shell of the overhead condenser are on and adjust setpoint for condenser water to 130°F.
8. Bring up the provox CRT BC32OH screen and put the large receiver jacket in "condwatr" position. Using the same CRT screen verify automatic valve #14 is in the closed position. This allows the heated water from the outlet of the overhead condenser to flow through the receiver jacket.
9. Verify the splitter valve is in manual mode and put in total take-off position. Open large receiver drain valve and open return valves to top of BC-32 reactor.
10. Adjust vacuum set point to ⁷⁰20 mm Hg with all three stages on (Use the Auto Stage setting to ^{adequate} initiate all three stages). Verify vacuum block valve on large receiver is open and manual vent valve ^{vacuum} on top of BC-32 reactor is closed in preparation for vacuum charging. *stage 3. if stage 3 does not yield adequate vacuum use stage 1-3.*
11. Wear rubber jacket, rubber gloves, face shield and position local exhaust ventilation at the bungs while adding Charge A. Add Charge A (Perfluorooctanoic Acid), into the reactor through the bottom drain line by vacuum using dedicated 1" charge hose. Pull vacuum through the large receiver and leave vacuum on while adding Charge A. Refer to Attachment 3 for PPE requirements when handling Charge A (FM-3256 and F-8281). *rubber pants + rubber boots 12-21/17 M.E. 12/15/17 BC*

^{a JB 12/21/17}
Add Charge A powder (Ammonium Perfluorooctanoate) after the liquid Charge A is added and after adding half of Charge D (Sulfuric Acid) per step 14. (The balance of Charge D serves as a charge line flush and helps prevent the charge hose from plugging.) Add the Charge A powder at a slow rate (about 150 lbs/30 minutes) so the salts have an opportunity to mix into the batch. Refer to Attachment 3 for PPE requirements when handling Charge A powder (F-6514)
Gloves should be washed thoroughly after powder handling, before removing.

- NOTE:**
- When charging powder by vacuum, charge slowly and pull vacuum through the large receiver. This will reduce the amount of powder reaching the jets and causing plugging problems.
 - Manhole charging is acceptable. This is usually easier for fiber cartons with poly bags. Be sure to use local exhaust when charging through manhole.

12. With the vacuum system off, slowly add Charge B (11-0000-3179-6), Filter Cell, to reactor through sight glass. Wear a dust mask and safety glasses at a minimum, and position local exhaust ventilation when handling Charge B. Refer to Attachment 3 for PPE requirements for handling Charge B (RM-3179)
13. With the vacuum system off, add Charge C (11-0000-8510-7), Potassium Dichromate, slowly through the sight glass. Refer to Attachment 3 when handling Charge C (RM-8510)
14. Once Charge A, Charge B, and Charge C are added, agitate the batch at 90 rpm, with the vacuum system off, for one hour ~~before adding charge D~~ TB 12/23/97

Note: The filter cell and Potassium Dichromate must be fully mixed into the batch before restarting the vacuum system to prevent vacuum system plugging.

15. Refer to Attachment 3 for PPE requirements when handling Charge D (RM-2706). Position local exhaust ventilation at the drum bungs while charging. Check if part of Charge D was added during step 11, prior to charging Charge A powder. Vacuum charge the balance of Charge D (11-0000-2706-7), Sulfuric Acid, to the reactor through the bottom drain line.

Adding Charge D last serves to flush the dedicated 1" charge line. When finished charging, disconnect the 1" charge line and flush with water to the sewer.

16. Put reactor in "batch" control and jacket in circulating water position. Set batch set point at 165°F, jacket hi-limit at 400°F, delta-t at 350°F, and agitator at 75 rpm. For Automatic control, refer to Attachment 2, Automatic Distillation Operation of BC-32.

II. DICHROMATE OXIDATION

17. Heat the batch to 165°F and hold for four hours. Record batch temperature, agitator speed and hold time hourly.
18. After the four hour hold at 165°F, set jacket selector to drain position. Wait 2 minutes. Set jacket selector to direct steam. Set batch setpoint to 265°F and start heating to distillation temperature.

IV. VACUUM DISTILLATION

19. The batch will be distilled manually. Before initiating vacuum distillation, all hand valves must be placed in the positions specified below and stay in these positions for the remainder of this section of this run.

-	FLOOR LEVEL	
	BC-32 Reactor Drain Valve	Closed
-	FIRST DECK	
	BC-32 Reactor Vent	Closed
	BC-32 Reflux Return Valve	Closed
	BC-32 Receiver Return Valves	Opened
	BC-32 Receiver First Floor Drain Valve	Closed
	2 valves in transfer line between BC-32 Receiver and BC-36 Reactor	Closed
	Vacuum Block Valve on top of BC-36	Closed
	Vacuum Block Valve on top of BC-37	Closed
-	SECOND DECK	
	Large Receiver Drain Valve	Opened
-	THIRD DECK	
	Large Receiver Vacuum/Vent Isolating Valve	Opened
	Reflux Trap Valve	Closed
	Takeoff Valve to Large Receiver	Opened
-	FOURTH DECK	
	Steam Tracing (5-7 psig)	On
	KO Pot Isolating Valve to Receivers	Opened

Typically, the manual valves providing steam and water for reactor jackets, condensers, vacuum jets, and receiver jackets are open. The status of these valves should be confirmed if this run follows either a weekend shutdown, any BC-32 maintenance work, or any other unusual BC-32 activity.

20. While distilling and collecting material in the large receiver, the following distillation data should be recorded.

- Reactor temperature
- Condenser head temperature
- Reactor jacket temperature
- Reactor jacket steam pressure
- Agitator speed
- Reactor vacuum
- Condenser water outlet temperature
- Large receiver gross weight
- Take-off rate
- Accumulated take-off
- Operator Comments, QC Results & Special Instructions

21. With all the valves in the correct positions, vacuum distillation can be initiated. When the batch temperature levels at 265°F, begin lowering the vacuum to 70 mm Hg (use Auto Stage to initiate all three stages). When the vacuum reaches 70 mm Hg, monitor the condenser head temperature and the condenser product temperature. Continue lowering the vacuum in 2-3 mm Hg increments (do not go below 60 mm Hg), until the condenser head temperature reaches 240°F and the condenser product temperature reaches 170°F. The condenser water outlet temperature should also be about 7-8°F warmer than the condenser water inlet temperature. Reflux at these conditions through the large receiver and back to the BC-32 reactor for 30 minutes.

22. After refluxing through the receiver and back to the BC-32 reactor for 30 minutes, verify that the condenser head temperature remains above 240°F. If necessary, adjust vacuum level. Once a steady condenser head temperature above 240 is established, close the large receiver bottom valve and begin collecting product. Adjust the vacuum setpoint in 2-3 mm Hg increments so a take-off rate of 200-400 lbs/hr is established.

To use the head temperature automatic control:

- a. Start the one-plate manually using vacuum set point to control head temperature.
- b. Set vacuum limits to 70 mmHg and 40 mmHg.
- c. Set vacuum control selector (DSR # 18) to HEADTMP-AUTO (DSR # 19)
- d. Set head temperature set point to an appropriate value to achieve a take-off rate between 200 and 400 lbs/hr.

only if following an out of spec lot of F7164 or if following FC-126 or
23. Collect 800-1,200 lbs of acid in large receiver. Drain the collected material back to the reactor and FC 26
resume vacuum distillation and collect product in large receiver.

if following an inspec lot of F7164 only collect 400 lbs on the first
NOTE: Collecting 800-1,200 lbs of C8 acid in the large receiver is necessary to remove excess *Distillation*
water and clean the system. If this is the first lot following a water boilout, repeat step *run.*
21 for a total of two 800-1200 lb overhead flushes. *3/3/96*
DC

23

24. After 1 hour, check the large receiver weight and lower the vacuum as necessary to maintain a takeoff rate of about 200-400 lbs/hr. When the vacuum is at 40 mm Hg and the takeoff rate drops, increase the batch temperature setpoint to 285°F. Once the batch temperature is at 285°F and the takeoff is less than 20 lbs/hr, and the condenser head temperature is less than 200°F, the vacuum distillation is complete.

Shut off the vacuum system and release vacuum with nitrogen. Set the jacket selector to circulating water and set the batch setpoint to 150°F

NOTE: If the take-off rate exceeds 600 lbs/hr, stop the distillation. Drain the large receiver to the BC-32 reactor. The acid must be redistilled because it will be contaminated with sulfuric acid and metals.

V. SAMPLING

25. Refer to Attachment 3 for PPE requirements for handling F-7164. Position local exhaust ventilation at the sight glass. Sample the receiver by isolating and breaking vacuum on the receiver using nitrogen. Do not use the receiver vent valve to break vacuum as dirt could get sucked back into the vessel. Dip a 4-ounce in-process sample out of the receiver and send sample to lab. Label as "SAMPLE #1".

NOTE: The sample should be placed in a 4-ounce poly bottle to provide additional safety during handling and to avoid metal contamination from the cover.

26. Based on the QC results from the previous step, do one of the following:
- If the product is in specification, proceed to next step.
 - If the product fails specifications, contact the Manufacturing Engineer. A resample will be required.

VI. DRAINING

27. Once specifications are met, the large receiver can be drained directly to BC-36. Be sure the drain line steam trace is on and a new 0.5-micron filter (26-1005-3164-4) is installed in transfer line filter housing.
28. Isolate the large receiver and release vacuum with nitrogen. Use the nitrogen regulator to put 2-5 psig pressure on the receiver. Verify BC-32 large receiver to BC-36 transfer valves are set to correct positions. Also verify that manual valves to BC-32 reactor are closed. When the F-6050 process is ready, begin transferring to BC-36.
29. Verify that the reactor jacket selector is set to circulating water and the batch temperature is at 150°F.

30. DO NOT DRAIN BOTTOMS CONTAINING DICHROMATE TO CHEMICAL SEWER.
DRAIN BOTTOMS PER WASTE DISPOSAL SECTION AND RECORD FOR EACH LOT.

DO NOT Drain Bottoms to used F6002 Drums.

NOTE: These bottoms contain chrome which can not be disposed of in chemical sewers.

Verify drums are empty prior to draining.

31. Shut down the reactor and overhead system if another lot is not scheduled. Otherwise, return to beginning of FOP and prepare for next lot of series.

DRAINING INFORMATION:

Container/Alternate: N/A
Filter: 1/2 micron baked glass filter (26-1005-3164-4)
Packaging Supplies: N/A
Customer Use Labels: N/A

Labels: N/A
Weight Per Container: N/A
Draining Temperature: 120-150°F
Draining Pressure: 0 to 5 psig

Special Draining Instructions:

Verify BC-36 is ready. Verify that all transfer valves are in correct positions for transfer directly to BC-36.

Final Sample Requirements:

See Quality Report.

Storage: N/A

Special Handling Instructions: N/A

BY-PRODUCT DRAINING INFORMATION: N/A

VII. CLEANUP

PRE-SERIES CLEAN-UP:

Add 2000 lbs of High Purity DI Water to BC-32. Set the splitter valve at the outlet of the overhead condenser to direct flow back to the reactor bypassing the receiver. Open cold side condenser vent, and close vacuum block valve. Set jacket to circulating water. Set batch setpoint to 230°F and jacket high limit to 260°F. Set agitator speed to 100 rpm. Heat to boiling and allow to boil for two hours. Cool to 150°F and drain to chemical sewer. Inspect interior of reactor for cleanliness. If required, repeat water boil as described above.

Using the hot water hose connection on the third floor, add 1600 lbs of hot potable water to the receiver (use the load cell readouts to monitor the amount of water added). Agitate at 100 rpm for one hour and drain to the chemical sewer. Using the receiver bypass drain line, add 1600 lbs of DI water to the receiver. Agitate at 100 rpm for approximately 15 minutes, and drain to the chemical sewer.

Note: Leaving DI water in the receiver for longer than 15 minutes, or charging hot DI water to the receiver may cause premature glass failure.

Dry entire system with 200°F jacket heat, full vacuum, and slow nitrogen purge through bottom of BC-32 reactor. Dry system for 2 hours under these conditions.

BETWEEN RUNS:

Since this unit is dedicated to the work up of C8, no between run clean up is required. Drain the distillation bottoms containing dichromate as described in the waste disposal section and record for each lot.

CLEANING AFTER LAST LOT OF SERIES:

Since this unit is dedicated to the work up of C8, no end of series clean up is required. Drain the distillation bottoms containing dichromate as described in the waste disposal section and record for each lot.

WASTE DISPOSAL:

Step:	30
Description:	Distillation Bottoms from F-7164
Labeling:	13-0084-3752-0
Container:	<i>use 34-7039-5732-3 Black poly lined or 34-7002-2745-6 Blue poly lined drums</i>
Disposition:	Incinerator <i>9/8/98 DG</i>
Amount:	About 600 lbs. Net (600 lbs/drum)

AUTHOR: Manufacturing Engineer: Dean Graham
Dean Graham

APPROVED BY: Product Chemist: Robert T. Beska
Bob Beska

Product Manager/Technical Supervisor: Bin T. Park For DK
Dan Keller.

Health & Safety Eng.: Yes No

Environmental Coordinator: Yes No

Management of Change Approvals:

Approval for Start-up (Operations): _____

Approval for Start-up (Safety): _____

COPIES TO: (Reference SOP 408-007)

- Dean Graham
- For Fluorochemical Products: (except Electrofluorination)
- SMD/FP&TC, Frank Klink, 53-6S-02
- SCD, Chemical Specialties/Environmental: Dale Neuman, 53-6S-02

MANAGEMENT OF CHANGE CHECKLIST

Management of Change Checklist									
Must do before start-up (initial)	Must do for closure (initial)		Date Cmp	By (init)	Must do before start-up (initial)	Must do for closure (initial)		Date Cmp	By (init)
		1. Consult with:					7. Environmental Concerns:		
		engineering					emissions requirements		
		operations					hazardous waste rules		
		maintenance					obtain permit		
DC		process control					other:		
		product engineering	11/18	DC					
		safety					8. Miscellaneous:		
		environmental					review for Maintenance		
		industrial hygiene					Conscious Engineering		
RTR		chemist	11-18	RTR			check utility loading		
		2. Conduct either:					evaluate impact of		
DC		informal hazard analysis or	1/18	DC			organizational change		
		Proc. Hazard Analysis (PHA)					other:		
		3. Review Engineering Standards:					9. Update Documentation:		
		piping					elementaries/flow diagrams		
		pressure vessels					emergency procedures		
		HVAC design					SOPs/FOPs		
		fire protection					safety interlock/alarms listings		
		machine guarding					process control information		
		hoist design					equipment files		
		electrical (area/class/group)					maintenance procedures		
		other:					other:		
		4. Evaluate Process Change:					10. Complete training of:		
		process chemistry					operations	11/18	DC
		process conditions					maintenance		
		special equipment preparation					contractors		
		downstream process impact					other:		
		adjacent process impact							
DC		quality issues	11/18	DC			11. Conduct PSSR or complete		
		other:					required prestart-up checks indicated below:		
		5. Occupational Health & Safety:					Prestartup Safety Review		
		conduct Job Safety Anal. (JSA)					structural check		
		evaluate noise levels					electrical check		
		review ergonomics					mechanical check		
		assess personal protect. equip.					instrumentation check		
		update MSDS information					performance check		
		other:					safety check		
		6. Mechanical Integrity Standards:					calibration		
		update PMs					lockout/tagout check		
		order spare parts/set up stock					12. Punchlist on action items:		
		Intrinsic Safety (IS) approval					action items section(s):		
		identify critical devices							
		other:							

Approval for MOC closure.

Originator: Dean Dahan Date: 11/18/97

Original Form to remain with signature copy of FOP
 Sign on Approval Section of FOP

32



Specialty Materials Division

**3M Cottage Grove
FACTORY OPERATING PROCEDURE**

Goto Ref Dev/Exp

PRODUCTION NUMBER:

41-2600-7164-6

REVISION: 1

DESCRIPTION: ONE PLATED C8 ACID (100%)

Superseding Date: April 29, 1998
Effective Date: 12/01/99

PROCESS TYPE: Standard Process

3M PROCESS HAZARD CLASSIFICATION:

2. Moderate

PHA REVALIDATION DATE: PHA REF. #

MOC #:

DEPARTMENT: 3060

BURDEN CENTER: 1532

REASON FOR REVISION:

To Move FOP to MDI system and to update procedure to run the distillation in Delta T control. Change format of the procedure.

AUTHOR:

Dean Graham/US-Corporate/3M/US

Status: Effective

Production Comments/Suggestions (indicate your name if you wish feedback on your ideas):

EQUIPMENT:

1. BC-32, Dept. 3060, 300-gallon Hastelloy reactor with hastelloy overhead and 200 gallon glass-lined receiver.

ATTACHMENTS:

1. BC 32 Reactor Flowsheet
2. Safe operating limits for the F-7164 process in BC -32
3. PPE requirements for the F-7164 process.

ITEMS NEEDING SPECIAL INSTRUCTIONS:

1. Distillation rates over 600 lbs/hr should be considered bump-overs and redistillation must be conducted.
2. Bottoms must be drained per Waste Disposal section. The distillation bottoms contain Chrome and must not be drained to the Chemical sewer
3. Do Not **boil** DI water through the glass-lined receiver. This may cause premature glass failure. Follow the cleanup instructions included in the cleanup section.
4. In order to help prevent vacuum system plugs, do not vacuum charge Dichromate (charge D) or Filter cell (charge C). These charges should be slowly added through the BC 32 sight glass and mixed into the batch before restarting the vacuum system.

REASON & DOCUMENTATION OF CHANGE:

(Revise Product Structure? ___ Yes X No); (New or Changed Emissions? ___ Yes X No)

MOC Required: X Yes ___ No: (Reference SOP 400-012)

Review Team required (Job Function):

- X Operations: Pam Gotz
- ___ Engineering
- ___ Maintenance
- X Process Engineering - Dean Graham
- X Safety/Hygiene - Gerri Mirkin
- ___ Other

Description of Change:

1. To update the Format of the FOP.
2. To move the FOP to the MDI system.
3. To include instructions for running the distillation using ΔT control, and eliminate the practice of using the vacuum set point to control the distillation rate.

Possible Negative Consequences:

1. No negative safety consequences identified for change #1.
2. No negative safety consequences identified for change #2.
3. Possible to over heat the reactor. Safeguards include Jacket High limit set to 330 F with a batch temperature set point of 285F.

REFERENCE:

1. 41-2600-7164-6, Factory Operating Procedure dated 04/29/98
2. Reviews with Building 15, BC-32 Operators.

END PRODUCT USE: F-7164 is an intermediate in the production of ammonium salt surfactants/emulsifiers. These surfactants/emulsifiers are sold to external companies, and are used as additives in the production of fluorochemical polymers such as Teflon.

CHARGE CALCULATIONS:

1. **Charge A1-A7:** Maximum Charge A is 3000 Lbs.

The composite of Charge A must meet the following requirements:

C8 Acid:	96.5% Minimum	Target 98%
C7 Acid:	1.4% Maximum	
C6 Acid:	1.4% Maximum	
High Boilers:	0.5% Maximum	

2. **Charge A8-A9:** Spray dried C8 powder charges FC143
3. **Charge B1:** Standard Charge B is 440 Lbs of RM-2706.

If %H2O in Charge A exceeds 1%, increase amount of Charge B as follows:

$$\text{Additional Charge B} = (0.00334) \times (\% \text{H}_2\text{O in Charge A}) \times (\text{Charge A})$$

4. **Charge B2:** Additional acid for C8 powder charges, A8 and A9 FC143

If powder charges A8 or A9 are added, Charge B2 is calculated as follows:

$$\text{Charge B2} = (0.440) \times (\text{Charge A8} + \text{Charge A9 powder charges})$$

5. **Charge C:** Standard Charge is 20 lbs of RM-3179.
6. **Charge D:** Standard Charge is 60 Lbs of RM-8510.

Manufacturing Engineer will provide spreadsheet printout with charge calculations and quantities.

EMERGENCY SHUTDOWN INSTRUCTIONS:

1. The process (reactor, receiver, and agitator) can be shutdown any time during the operation of the equipment by turning off the agitator, shut off vacuum system, put the reactor jacket in drain, and completely isolate the system.
2. Once confirming that the acid in the receiver or reactor is melted, the system can be restarted at any time by following the remainder of the FOP. If shut down for more than 4hrs and the overhead condenser water temperature has fallen below 120 degrees F, verify that the acid in the receiver is melted.

Note: Once the jacket on the reactor is put into neutral any acid contained in this vessel will begin to solidify and harden. Remelting the acid may take up to a couple of days. Confirming that the acid is melted prior to starting up the agitator is essential to prevent agitator damage.

OPERATING PROCEDURE:

I. PRE-SERIES PREPARATION

1. The operator will need to be familiar with the Provox CRT and how it is used to operate BC-32. If questions exist contact the Supervisor.
2. A general flowsheet of the BC-32 process piping is attached. The operator should review it and use it as reference during the remainder of this standard.
3. The open column will be used for this run. If not already done, valve in the open column and blank off both top and bottom of packed column.
4. Reactor, overhead condenser, receiver, and transfer lines normally do not require cleaning since this unit is dedicated to the distillation of C8 acid. If additional cleaning is required the Manufacturing Engineer will request it. Refer to the Clean Up section in the back of this standard if a clean up is to be preformed.
5. Verify the steam tracing to overhead piping and to BC-32 drain lines is hot . The steam supply valve for overhead tracing is located directly behind the overhead condenser. The steam supply valve for the BC-32 drain line is located on first floor against the wall underneath the BC-32 reactor. The steam pressure should be about 5 to 10 psig.

II. CHARGING

6. Prepare to charge BC-32 reactor by setting the following:

Parameter or Valve	Desired setting for charging
Condenser water selector	Temp-H2O (opens water and steam valves) ✓
Condenser inlet water set point	130 F ✓
Receiver Jacket setting	Condwater with valve #14 closed. (Circulates the condenser outlet water through the receiver jacket). ✓
Reflux return line manual valve	Closed ✓
Manual valves in the take off line to the receiver (between the receiver and condenser)	All should be Open. ✓
Manual three way Splitter valve	Take off to receiver. (No longer an air operated valve) ✓
Manual vacuum/vent block valve on top of the receiver	Open ✓
BC-36 vacuum cross over manual valve	Closed or blanked off ✓
Cold side Vacuum/vent select	Isolate (closes vent and vacuum valves) ✓
Receiver Vacuum/vent Select	Vacuum (closes vent valve opens vacuum valve) ✓
Hot side Reactor manual vent valve	Closed ✓
BC-32 Reactor Jacket select	Neutral 120° ✓
BC-32 Agitator speed set point	75rpm ✓
Receiver Air operated Drain Valve	Closed for charging ✓

7. Verify the overhead condenser inlet water temperature is holding at 130°F.
8. Verify valve #14 on the receiver Jacket system is closed (console screen BC32OH).
9. Adjust vacuum set point to 100 mm Hg using stage three only (Using the Auto Stage setting will initiate all three stages). If stage three produces insufficient vacuum at any time during the operation, then change the vacuum status to stages 2-3 and then 1-3 or auto stage as needed to provide adequate vacuum levels.

Charging liquid C8 (Charges A1-A7) and Sulfuric Acid (Charge B1)

10. Wear neoprene rubber gloves, rubber jacket, rubber pants, rubber boots, face shield, safety goggles, and position local exhaust ventilation at the bungs while adding Charges A1-A7 (C8 acid) as specified in Attachment 3, PPE requirements. Add Charges A1-A7 (**liquid Perfluorooctanoic Acid**), into the reactor through the bottom drain line using 100mmHg continuous vacuum pulled through the open column, condenser, and through the receiver. Use the dedicated 1inch charge hose. Verify that the receiver drain valve is closed during charging.
11. Refer to Attachment 3 for PPE requirements when handling Charge B (RM-2706). Position local exhaust ventilation at the drum bungs while charging. Using 100mmHg of continuous add Charges B1 and B2 (11-0000-2706-7), Sulfuric Acid, to the reactor through the bottom drain line. Adding Charge B, Sulfuric acid, immediately after adding Charges A1-A7 serves to flush the dedicated 1" charge line. When finished charging, disconnect the 1" charge line and flush with water to the sewer.

Charging powder F-6514 (Charges A8-A9)

NOTE: Do not add F-6514 powder using vacuum. This can cause the powder to vaporize and be lost out the jets or plug the condenser. Add all F-6514 powder through the manhole. Be sure to use local exhaust when charging through manhole.

12. If Charges A8 and A9 are not included in this series, skip directly to the next step. Otherwise perform the following:
- a) Add powder Charges A8-A9 (**Ammonium Perfluorooctanoate F-6514**) after the liquid Charges A1-A7 and after adding Charges B1 + B2.
 - b) Change the vacuum select to Isolate and break vacuum with nitrogen.
 - c) Add the powder Charges A8 and A9 through the reactor man way at a slow rate (about 150 lbs/30 minutes) so the salts have an opportunity to mix into the batch. Refer to Attachment 3 for PPE requirements when handling Charge A powder (F-6514). Gloves should be washed thoroughly after powder handling, before removing .

Charging Filter Cell (Charge C) and Potassium Dichromate (Charge D)

13. With the vacuum system off (break vacuum using nitrogen) and with the receiver drain valve closed, slowly add Charge C (**11-0000-3179-6**), Filter Cell, to reactor through sight glass. Wear a dust mask and safety glasses at a minimum, and position local exhaust ventilation when handling Charge C. Refer to Attachment 3 for PPE requirements for handling Charge C (RM-3179)
14. With the vacuum system off (break vacuum using nitrogen) and with the receiver drain valve closed, add Charge D (**11-0000-8510-7**), Potassium Dichromate, slowly through the sight glass. Refer to Attachment 3 when handling Charge D (RM-8510)

*SET vacuum selector to isolate then turn jets off
7/17/00
00*

III. DICHROMATE OXIDATION

15. Prepare for the four hour hold by setting the following:

Parameter	4hr hold Desired setting
Jacket Control Select	"batch" control
Jacket mode	circulating water position
Batch Temperature set point	165°F
Jacket Hi-limit	340°F
Delta-T	100°F
Agitator speed setpoint	75 rpm

16. Heat the batch to 165°F and hold for four hours. Record batch temperature, agitator speed and hold time hourly.

RECEIVED
ALL STG
VACUUM / FLOOR LEVEL - ISOLATE

IV. VACUUM DISTILLATION

17. The batch will be distilled manually. Before initiating vacuum distillation, **all MANUAL valves must be placed in the positions specified below.**

FLOOR LEVEL	
BC-32 Reactor Drain Valve	Closed

FIRST DECK	
BC-32 Reactor Vent	Closed
BC-32 Reflux Return Valve	Closed
BC-32 Receiver Return Valves	Opened
BC-32 Receiver First Floor Drain Valve	Closed
2 valves in transfer line between BC-32 Receiver and BC-36 Reactor	Closed
Vacuum Block Valve on top of BC-36	Closed
Vacuum Block Valve on top of BC-37	Closed

SECOND DECK	
Large Receiver Manual Drain Valve	Opened

THIRD DECK	
Large Receiver Vacuum/Vent Manual Block Valve	Opened
Reflux Trap Valve	Closed
Takeoff Valve to Large Receiver	Opened

FOURTH DECK	
Steam Tracing (5-7 psig)	On
BC-36 & BC-37 vacuum cross over valve	Closed
KO Pot Isolating Valve to Receiver	Opened
Manual Splitter Valve	Take off to Receiver

Typically, the manual valves providing steam and water for reactor jackets, condensers, vacuum jets, and receiver jackets are open. The status of these valves should be confirmed if this run follows either a weekend shutdown, any BC-32 maintenance work, or any other unusual BC-32 activity.

18. While distilling and collecting material in the large receiver, the following distillation data should be recorded.
- Reactor temperature
 - Condenser head temperature
 - Reactor jacket temperature
 - Reactor jacket steam pressure
 - Agitator speed
 - Reactor vacuum
 - Condenser water outlet temperature
 - Large receiver gross weight
 - Take-off rate
 - Accumulated take-off
 - Operator Comments, QC Results & Special Instructions

19. With all the Manual valves in the correct positions, vacuum distillation can be initiated by setting the following:

Parameter	Desired setting for Vacuum Distillation
Condenser water selector	Temp-H2O (opens water and steam valves)
Condenser inlet water set point	130 F
Receiver Jacket setting	Condwater with valve #14 closed. (Circulates the condenser outlet water through the receiver jacket).
Receiver Vacuum/vent Select	Vacuum (closes vent valve opens vacuum valve)
Vacuum control select	HeadTemp-M (manual)
Vacuum Jet Selector	Stage 1-3
Vacuum Set point	40mmHg
BC-32 Reactor Jacket select	Switch from Circ water to Drain. Wait 2 minutes then set to direct steam.
Batch Temperature set point	285°F
Jacket Hi-limit	340°F
Delta-T	100°F
Air operated Receiver Drain Valve	Initially should be closed.

20. Once the batch temperature reaches 240 F then change the Delta T to ^{20 12/5/99 DC} 50. Continue to monitor the condenser head temperature. Once the head temperature reaches 240°F (the batch temperature should be about 250 F), then product should begin to collect in the receiver. The condenser product temperature should be near 170°F. The condenser water outlet temperature should be about 7-8°F warmer than the condenser water inlet temperature.

21. Verify that the condenser head temperature remains above 240°F and a strong take off rate is established. If necessary, adjust the Delta T to increase or decrease the take off rate. Change the Delta T by 5 or 10 degree increments. Increasing the Delta T will increase the heat put into the batch and will increase the take off rate. Decreasing the Delta T will decrease the take off rate.

22. Collect 400 lbs of acid in receiver. Drain the collected material back to the reactor then resume vacuum distillation and collecting product in receiver.

NOTE: Do not continuously reflux through the receiver, back to the reactor. The affect of the steady free falling liquid will erode the glass lining in the receiver. Collecting 400 lbs of C8 acid in the receiver is necessary to remove excess water and clean the system if material was bumped over during the initial heat up.

23. Check the large receiver weight hourly. Increase or decrease the Delta T as necessary to maintain a steady takeoff rate of approximately 400 lbs/hr (increasing the Delta T will increase the take off rate. Decreasing the Delta T will decrease the take off rate). Change the Delta T in 5 or 10 degree increments.

NOTE: If the take-off rate exceeds ^{700 163/hr} 600 lbs/hr, stop the distillation. Drain the receiver to the BC-32 reactor. The acid must be redistilled because it will be contaminated with sulfuric acid and metals.

24. Continue the distillation until the following occur then proceed to the next step:
- The batch temperature is at 285°F or greater with the vacuum level at 40 mmHg.
 - The takeoff rate drops to less than 20 lbs/hr
 - The condenser head temperature drops to less than 200°F
 - The Jacket temperature and pressure rises sharply from the steady state values (the steady state value for the jacket temperature should be between 280 F and 295 F. The steady state value for the jacket pressure should be between 35 psig and 45 psig)
25. Once the conditions of the previous step are met then end the distillation by performing the following:
- Place the jacket select to ~~NEUTRAL~~ ^{NEUTRAL} ~~THE DEAD~~, wait 2 minutes then switch to circulating water.
 - Set the Batch Temperature Set point to 150°F
 - Shut off the vacuum system and release vacuum with nitrogen.

V. SAMPLING

26. Refer to Attachment 3 for PPE requirements for handling F-7164. Position local exhaust ventilation at the receiver sight glass. Sample the receiver by isolating and breaking vacuum on the receiver using nitrogen. Do not use the receiver vent valve to break vacuum as dirt could get sucked back into the vessel. Using the sampling dipper, collect a 4-ounce in-process sample out of the receiver. Place the sample in a 4-oz poly bottle and send it to lab. Label as "SAMPLE #1".

NOTE: The sample should be placed in a 4-ounce poly bottle to provide additional safety during handling and to avoid metal contamination from the metal lids.

27. Based on the QC results from the previous step, do one of the following:
- If the product is in specification, proceed to next step.
 - If the product fails specifications, contact the Manufacturing Engineer. A resample will be required.

VI. RECEIVER DRAINING

28. Once specifications are met the material is ready for use. Determine which process this material is to be used in and perform one of the following:
- If the Acid is to be used in the F-1256 (FC-26) process, then go directly to the F-1256 operating procedure.
 - If the Acid is to be used in the F-6050 or F-8772 processes then transfer the acid to BC-36 per the following step when ready.
29. If this material is to be used in BC-36, the acid can be transferred from the receiver directly to BC-36 by performing the following:
- Be sure the drain line steam trace is on
 - Install a new 0.5-micron filter (26-1005-3164-4) in transfer line filter housing.
 - Isolate the receiver.
 - Use the nitrogen regulator to put 2-5 psig nitrogen pressure on the receiver.
 - Set all the valves in the transfer line between the BC-32 receiver and BC-36 to the correct positions to direct the flow from the receiver to the BC-36 reactor.
 - Verify that manual valves directing flow back to the BC-32 reactor are closed.
 - When the F-6050 or F-8772 process is ready, begin transferring to BC-36.

VII. DRAINING BOTTOMS

30. Verify that the reactor jacket selector is set to circulating water and the batch temperature is at 150°F.
31. The Distillation Bottoms should be drained to a used Charge A drum (black poly lined drum) per the waste disposal section. Verify all drums used to drain bottoms are empty. Do not drain bottoms into used F-6002 drums. A reaction will occur causing pressure to build in the drum. Do not drain the bottoms to the chemical sewer. These bottoms contain chrome, which can not be disposed of in chemical sewers.

VIII. CLEANUP

32. The reactor and receiver should be cleaned separately. In most cases the reactor will be the only vessel requiring cleaning. Clean up the reactor as follows:
 - a) Add 800 lbs of DI water to the reactor.
 - b) Using 100mmHg of vacuum add 1500 pounds (three drums) of RM-0244, Sodium Hydroxide. Refer to Attachment 3 for required PPE while charging RM-0244.
 - c) Inspect the inside of the reactor, and add enough DI water to bring the liquid level up to above the Dichromate residue, if required.
 - d) Set jacket to circulating water.
 - e) Set batch temperature setpoint to 160°F. **Do not boil water into the overhead. This can cause glass corrosion in the receiver. Verify that no water was trapped in the receiver. Drain any trapped water back to the reactor.**
 - f) Set the jacket high limit to 260°F and Delta T to 100
 - g) Set the agitator speed to 100 rpm.
 - h) Mix at 160°F for three hours.
 - i) After the three hr mix, Cool to the material to less than 100°F.
 - j) Since this cleaning solution contains Chrome the material should be drained to drums and sent to the incinerator per the waste disposal section. Refer to Attachment 3 for required PPE while draining the cleaning solution.
 - k) Flush out the reactor with DI water after the cleaning solution has been drained. Drain the flush water to the sewer.
 - l) Dry out the reactor with 200°F jacket heat, 40 mmHg vacuum, and slow nitrogen purge through bottom of the reactor. Dry system for 2 hours under these conditions.
 - m) Inspect interior of reactor for cleanliness.
33. If the receiver requires cleaning, then perform the following:
 - a) Set the receiver vacuum/vent select to vent and open the manual vacuum block valve.
 - b) Using the hot water hose connection on the third floor, fill the receiver with hot potable water (about 1600 lbs). Using a water hose, the receiver can also be flushed out through the sight glass if required.
 - c) Drain the water in the receiver to the sewer through the ground floor drain line. Be sure to blow the line clear with nitrogen. Do not leave water in the receiver for extended periods of time. This may cause glass damage. Be sure to drain immediately after flushing.
 - d) Repeat the water flush until the receiver is clean.
 - e) Dry out the receiver using Vacuum set at 40 mmHg, a slow nitrogen purge, and the receiver jacket mode set to condenser water with the condenser water inlet temperature to 130 F.

Note: Do not boil DI water into the receiver. This may cause glass corrosion.

BETWEEN RUNS:

Since this unit is dedicated to the work up of C8, no between run clean up is required. Drain the distillation bottoms containing dichromate as described in the waste disposal section and record for each lot.

CLEANING AFTER LAST LOT OF SERIES:

Since this unit is dedicated to the work up of C8, no end of series clean up is required. Drain the distillation bottoms containing dichromate as described in the waste disposal section and record for each lot.

DRAINING INFORMATION:

Container/Alternate: N/A
Filter: 1/2 micron baked glass filter (26-1005-3164-4)
Packaging Supplies: N/A
Customer Use Labels: N/A
Labels: N/A
Weight Per Container: N/A
Draining Temperature: 120-150°F
Draining Pressure: 0 to 5 psig

Special Draining Instructions:

Verify the run in BC-36 is ready for the acid transfer. Verify that all transfer valves are in correct positions for transfer directly to BC-36.

If this material is to be drained to jars for FC-26, then go directly to the F-1256 procedure for sampling and draining instructions.

Final Sample Requirements:

See Quality Report.

Storage: N/A

Special Handling Instructions: N/A

BY-PRODUCT DRAINING INFORMATION: N/A

WASTE DISPOSAL:

Step:	30
Description:	Distillation Bottoms from F-7164
Labeling:	13-0084-3752-0
Container:	34-7039-5732-3 used Black poly lined drum or 34-7002-2745-6 Blue poly lined drum
Disposition/Amount	Incinerator / About 600 lbs. Net (600 lbs/drum)

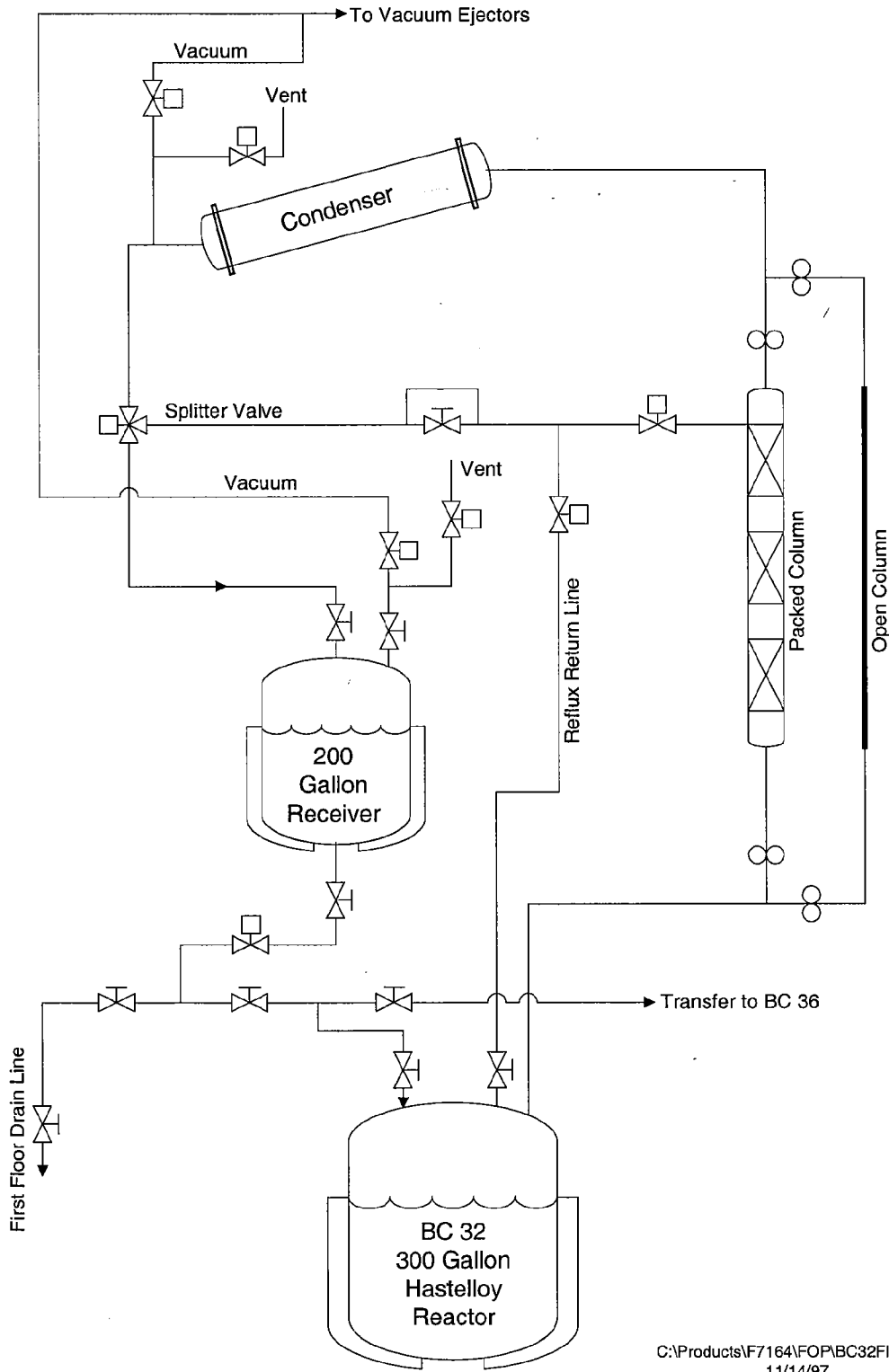
Step:	30
Description:	Sodium Hydroxide Cleaning Solution
Labeling:	13-0098-1435-4
Container:	34-7039-5732-3 used Black poly lined drum or 34-7029-4109-6 Reconditioned poly lined drum or 34-7002-2745-6 Blue poly lined drum
Disposition	Incinerator
Amount	About 2000 lbs. Net (400 lbs/drum)

MANAGEMENT OF CHANGE CHECKLIST

Management of Change Checklist									
Must do before start-up (initial)	Must do for closure (initial)		Date Cmp	By (init)	Must do before start-up (initial)	Must do for closure (initial)		Date Cmp	By (init)
		1. Consult with:					7. Environmental Concerns:		
		engineering					emissions requirements		
PJG		operations					hazardous waste rules		
		maintenance					obtain permit		
DG		process control			DG		other: WSP # for cleaning		
DG		product engineering							
GM		safety					8. Miscellaneous:		
		environmental					review for Maintenance Conscious Engineering		
GM		industrial hygiene					check utility loading		
RL		chemist					evaluate impact of organizational change		
							other:		
		2. Conduct either:							
DG		informal hazard analysis or Proc. Hazard Analysis (PHA)							
		3. Review Engineering Standards:					9. Update Documentation:		
		pipng					elementaries/flow diagrams		
		pressure vessels					emergency procedures		
		HVAC design			DG		FOPs		
		fire protection					safety interlock/alarms listi		
		machine guarding					process control information		
		hoist design					equipment files		
		electrical (area/class/group)					maintenance procedures		
		other:					other:		
		4. Evaluate Process Change:					10. Complete training of:		
DG		process chemistry			DG		operations		
DG		process conditions					maintenance		
		special equipment preparation					contractors		
DG		downstream process impact					other:		
DG		adjacent process impact							
DG		quality issues							
DG		other: Cleaning Procedure					11. Conduct PSSR or complete required prestartup checks indicated below:		
							Prestartup Safety Review		
		5. Occupational Health & Safety:					structural check		
		conduct Job Safety Anal. (JSA)					electrical check		
		evaluate noise levels					mechanical check		
		review ergonomics					instrumentation check		
		assess personal protect. equip.					performance check		
		update MSDS information					safety check		
		other:					calibration		
							lockout/tagout check		
		6. Mechanical Integrity Standards:					12. Punchlist on action items:		
		update PMs					action items section(s):		
		order spare parts/set up stock							
		Intrinsic Safety (IS) approval							
		identify critical devices							
		other:							

ATTACHMENT 1

BC-32 Reactor Flowsheet Department 3060

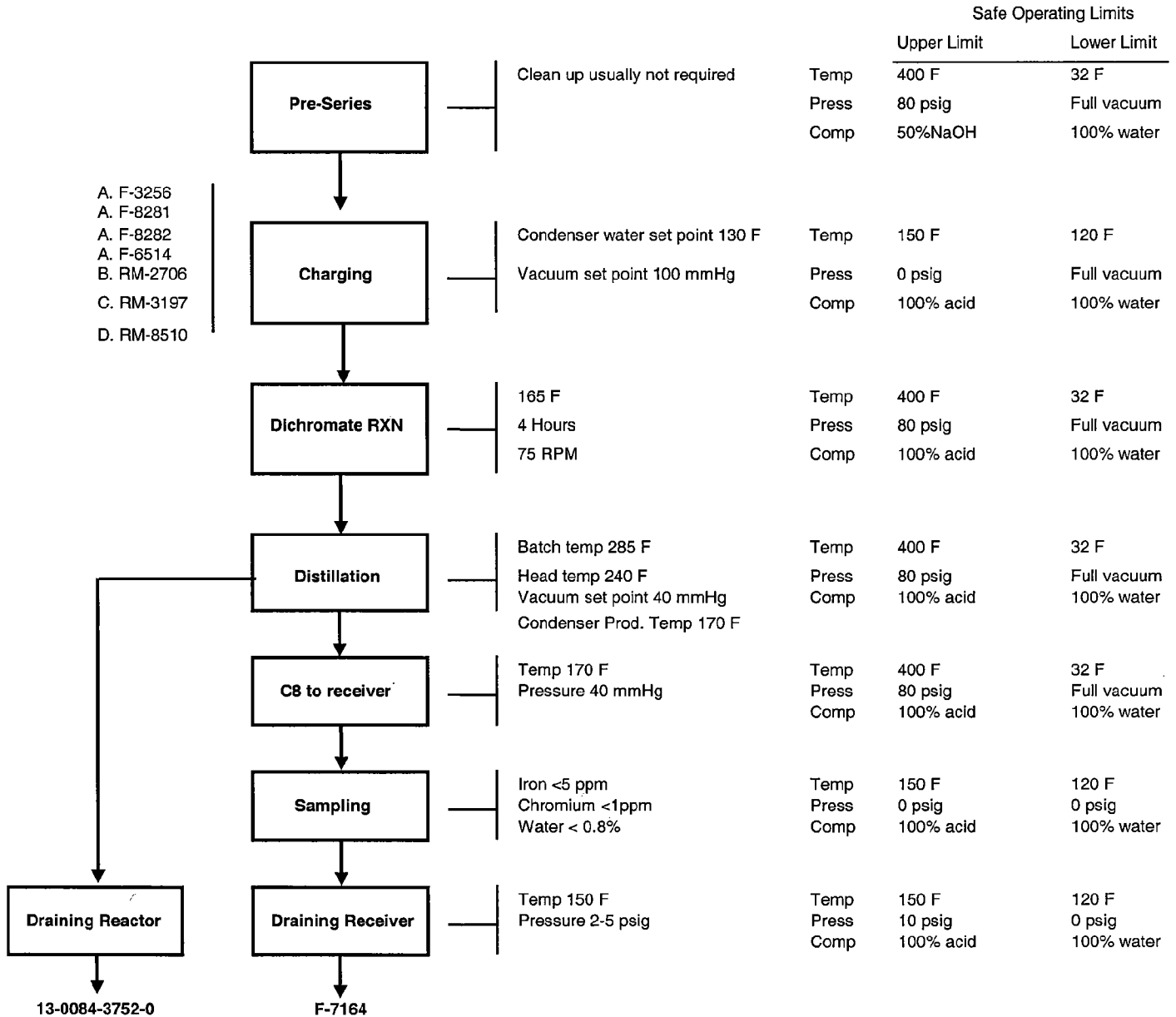


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11/14/97

ATTACHMENT 2

SAFE OPERATING LIMITS:

F-7164 One-Plated Perfluoro-Octanoic Acid Process



**ATTACHMENT 3
 PERSONAL PROTECTIVE EQUIPMENT REQUIREMENTS FOR PRODUCT NUMBER: F-7164**

Engineer: Dean Graham

Hazard Level Definitions:

- E=Extreme: Likely to cause irreversible tissue damage, serious illness or death from contact or exposure with a very small amount.
- H=High: May cause extreme irritation, irreversible tissue damage, serious illness or death from single or repeated contact or exposures.
- M=Medium: May cause moderate to severe irritation; may cause allergic reaction (sensitization); may cause reversible systemic effects.
- L=Low: May cause mild temporary irritation on contact; no cumulative effects are expected from repeated contact or exposures.

11-Digit Code	Material Name and Description	PHYSICAL DATA			HAZARD LEVEL			EYES		SKIN			LUNGS				Comments
		Physical state	Flammable (handled above)	Flash point	Flash point	Eyes	Skin	Lungs	Goggles	Face Shield	Gloves	Rubber Jacket	Rubber suit & boots	Spot ventilation	Dust mask	Cartridge respirator	
41-2700-3256-2	Fractionated C8 Acid - Main Cut	S		NA	H	H	M	X	X	Neoprene		X	X	X			1
41-2600-8281-7	Fractionated C8 Acid from FM-3256	S		NA	H	H	M	X	X	Neoprene		X	X	X			1
41-2600-8282-5	Fractionated C8 Acid from FM-3256	S		NA	H	H	M	X	X	Neoprene		X	X	X			1
11-0000-3179-6	Celite 500 (filter Cell, Diatomaceous)	S		NA	L	L	M			Cloth			X	X			
11-0000-8510-7	Potassium Dichromate	S		NA	H	H	H			Neoprene			X	X	Purple		2
11-0000-2706-7	Sulfuric Acid 96%	L		NA	H	H	M			Neoprene		X	X	X	Yellow		
41-2600-7164-6	Distilled C8 Acid	S		NA	H	H	M	X	X	Neoprene		X					1
11-0000-0244-1	Sodium Hydroxide 50%	L		NA	H	H	M	X	X	Neoprene		X	X	X			
Bottoms	Sulfuric Acid, Dichromate, Filter Cell	L		NA	H	H	M	X	X	Neoprene		X	X	X			
Cleaning solution	Dichromate, Sodium Hydroxide, Water	L		NA	H	H	M	X	X	Neoprene		X	X	X			
41-2600-6514-3	FC-143 (ammonium Perfluorooctanoate)	S		NA	M	L	M	X	X	Neoprene			X	X	Purple		2

Comments:

1. Solid at room temperature. Melts at 120 F. Usually handled as a liquid. Drums may be hot since the material is stored in the hot room.
2. The cartridge respirator with hepa filters can be upgraded to a supplied air respirator if desired.



**3M Cottage Grove
FACTORY OPERATING PROCEDURE**

Goto Ref Dev/Exp

PRODUCTION NUMBER: 41-2700-3256-2 REVISION: 1

DESCRIPTION: FRACT C8 ACID MAINCUT (>96%)

Effective Date:
10/20/2000

Expiration Date:
10/20/2006

PROCESS TYPE: Standard Process

3M PROCESS HAZARD CLASSIFICATION: SIGNIFICANT ENVIRONMENTAL ASPECT:
1. High

PHA REVALIDATION DATE: PHA REF. #

MOC #:

DEPARTMENT: 3060 BURDEN CENTER: 1534

REASON FOR REVISION:

To update procedure to include new safe handling instructions for charging, draining and sampling. To include new procedures for decontamination, area cleaning, and housekeeping to reduce exposure to C8 acid. To include new bottoms draining and reactor clean up procedures. To include instructions to help ensure the sample tap and drain line are cleared out after each use. To include instructions for ST. 15 operation. To include tables of operating parameters for each cut. To include instructions for Fractionating only recycled C8 acid pre cuts and post cuts.

Engineer Responsible for this Product:
Dean Graham/US-Corporate/3M/US

AUTHOR:
Dean Graham/US-Corporate/3M/US

Status: Effective

Production Comments/Suggestions (indicate your name if you wish feedback on your ideas):

TEXT:

EQUIPMENT:

1. Dept. 3060, BC-34 (1250 gallon Hastelloy reactor system)
2. Storage Tank 15 Stabilized C8 Acid/Inert Storage Tank

ATTACHMENTS:

1. Fractionation Labeling Information for all cuts
2. Temporary Inert Storage Procedures
3. Fractionation of recycled C8 acid pre-cuts and post cuts.
4. PPE Requirements for FM-3256.

ITEMS NEEDING SPECIAL ATTENTION:

1. C8 acid must be contained to within the reactor room. The most critical control parameter to ensure containment is the proper use of the PPE Donning and Doffing rooms/area. The proper PPE must be worn while in the room and this PPE must be properly removed in the Doffing room/area in order to prevent tracking the C8 acid out of the reactor room. Contact the shift supervisor for questions regarding PPE and for the proper use of the PPE Donning and Doffing room/area.
2. The recent C8 acid Industrial Hygiene Assessment of the reactor room has identified a number of exposure significant tasks which require special attention. **The most exposure significant tasks include Charging, Draining, Sampling, Draining Fractionation bottoms, and Maintenance.** Following each of these tasks the entire area should be washed down and cleaned. All tools and auxiliary equipment should be washed down and stored in the appropriate locations. During each of these tasks special attention should be given to good housekeeping practices to ensure the C8 acid material is properly contained and is not spread to non-process areas in the building.
3. FM-8749, F-0824, FM-4647, and all C8 acid recycle charges (FM-3210, FM-3257, F-8281, F-8282) are Hot Room charges. Store in hot room for 24 to 48 hours or until melted before charging. When opening drums from the hot room, extreme care should be taken to minimize exposure to C8 acid materials.

SHUTDOWN INSTRUCTIONS:

1. This run can be shutdown at any time without causing a safety or quality problem except as noted below.

- If the process is shut down for short periods (less than 8 hrs) the batch should be left on total reflux. This will avoid the time lost in cool down and heatup.
- If the fractionation is interrupted the column head temperature must be allowed to stabilize on total reflux before resuming takeoff to the receiver assure proper quality.

REFERENCE:

1. ~~41-2700-3256~~-2 Factory Operating Standard, July 26, 1999, Revision 3.

MANUFACTURED FOR:

1. Customer Division: SMMD
2. Customer Lab Contact: Mike Sierakowski
3. SMD/FP&TC Contact: Don Bloomdahl

END PRODUCT USE:

1. FM-3256 is a pure acid intermediate used in the production of FC-26, FC-118, FC-126 and FC-143 surfactants used in the manufacture of fluoropolymers and elastimers such as Teflon®.

PROCESS TOLERANCE:

1. Unless otherwise specified, record data for the process variables as requested in the FOP a minimum of once per hour.
2. Unless otherwise specified, time intervals specified are a minimum time. To obtain consistent process conditions the Operator should continue processing at the specified time interval.
3. Unless otherwise specified, maintain process variations within the tolerances listed below. If unable to operate within the acceptable tolerances, contact the Supervisor, or Mfg. Engineer for instructions. Note all additional verbal instructions on the data card. Attach all written instructions to the Production Reports.
4. Unless otherwise specified in the procedure, use stencil weights for charging.

PROCESS VARIABLES	ACCEPTABLE PROCESS TOLERANCES		RECORD HOURLY	
	(+)	(-)	(Y)	(N)
Agitator speed (RPM)	5	5	Y	

Batch Temperature (°F)	5	5	Y	
Jacket Temperature (°F)	5	5	Y	
Head Temperature (°F)	2	2	Y	
Condenser Water Temperature(°F)	5	5	Y	
Reactor Pressure/Vacuum (mmHg)	2	2	Y	
Column D.P. (mmHg)	5	5	Y	
Charge weight	5	5	Y	
Drain weight	2	2	Y	
Take Off / Charge / Draining Rates	Target and tolerances are specified in the procedure			

OPERATING PROCEDURE:

I PRE-RUN PREPARATION

1. The packed column must be in service; blank the open column if necessary.
2. Set up the valves in the overhead by performing the following:
 - a) Set up the valves to by-pass the decanter.
 - b) Open the by-pass valve in the liquid seal loop in the reflux return line to the column.
 - c) Close the valve in condensate return line to the New receiver.
Condensate return should be routed to reactor for startup.
3. The sight glasses on the reactor and receivers need to be protected from fluoride attack with a thin liner of Kel-F™ plastic on the inside. Inspect and replace any sightglass that has been etched from fluoride attack.
4. The reactor, overhead, receiver, and all transfer lines must be free from contamination of other products. **A Pre-Run Cleanup is required if this lot follows a product other than FM-3256.** Since this reactor is currently dedicated to C8 acid processes and a post run clean up is performed after each lot, a pre-run clean up is usually not required. If a pre run clean up is required then perform the steps specified in the Post Run Clean up Section of this FOP. If cleanup is not required, proceed to the next step.
5. Pressure the reactor system including overhead and receiver to 40 psig with nitrogen. The pressure loss should be less than 1 psig after 30 minutes. Find and repair leaks as required. Use water to hydrostat system if necessary to find leaks. Once the test is complete then vent off the pressure to the scrubber.
6. Check the D.P. lines to make sure they are open and not plugged by performing the following:
 - a) Set the jacket to Circ Water.
 - b) Put the control mode to D.P. and make sure the reactor is either under vacuum or at 0 psig and vented. This should open the shutoff valves in the D.P. lines. The D.P. valves will not open if the reactor pressure is greater than 7psig.
 - c) Go to the 3rd floor and observe the small rotameters on the nitrogen purge lines to the D.P. tubes. There should be flow of nitrogen visible. Adjust the rotameters to midscale if necessary. Observe the rotameters for several minutes to make sure the readings are stable.
 - d) If there is no flow or if there was flow initially and the flow stopped then the corresponding *D.P.* line is plugged or a valve is closed in the

tube.

- e) If a flow is visible and is stable then test to verify that there is not a leak in the D.P. tubing by putting the jacket control in neutral to close the valves to the D.P. lines. Now watch the rotameters. If flow continues through the rotameters then there is a leak in the tubing. If the flow stops--(it may take a couple of minutes for the longer high pressure side D.P. line)--then the line is OK.
 - f) If the D.P. lines do not pass the above tests then blow nitrogen through the D.P. tubes to clear the lines. If the lines cannot be cleared with nitrogen or steam then call maintenance and have a fitter clean/repair the line. The most likely place for a plug is in the lower section of the line on the top of the reactor.
 - g) The D.P. control will not work properly if the lines are partially plugged.
7. Test the vacuum control system. It must be able to control at 10 mmHg or less before proceeding.

II CHARGING

8. Set the following parameters to the desired settings in preparation for charging:

Parameter	Desired Setting
Reactor Jacket Select	Circulating Water
Reactor Jacket Mode	Jacket Auto
Reactor Jacket Set point	130 F (The reactor jacket will hold at 130 F and will prevent the C8 acid material from freezing on the walls of the reactor.)
Receiver Jacket Select	Full Cooling
Condenser Select	Cooling
Condenser Water Temp Set point	55 F
Over head Valve Select	New Receiver
ST 15 Steam Select	Steam On (See note Below)
ST 15 Jacket Mode	Batch Auto
ST 15 Jacket High Limit	150 F
ST 15 Batch Temp Set point	130 F (See not Below)
ST 15 Agitator	Turn on & set to run at 50 rpm

Note: Storage Tank 15 only runs on direct steam. Monitor the batch and jacket temperatures. If ST 15 overheats then shut off the steam supply valve to the jacket. Do not let the material in the storage tank to get hotter than 150 F.

9. Add the specified amounts of **Charge A1 (41-2600-8749-3), A2 (41-2600-0824-2) or A3 (41-2700-4647-1)** Stabilized Cell Product, to

Storage Tank 15 by performing the following steps. *If F-8749, F-0824, or FM-4647 are **not** scheduled for this run and the input charges are **only** recycled C8 acid pre-cuts or post cuts then follow the procedure in Attachment 3 for charging and Fractionation of recycled materials.*

- a) Pull continuous vacuum on the reactor through the receiver, and overhead, and through equalizing line. If using the single stage, pull down to the maximum vacuum level or if using the multi stage use a set point of 100 mmH.
 - b) Vacuum Charge A into the reactor. Occasionally FC salts solidify in the charge A drums and can not be vacuumed into the reactor. If the charge A drums can not be completely emptied then dispose of each drum as indicated in the waste disposal section.
 - c) Once the reactor is full then use nitrogen pressure to transfer the material into ST 15.
 - d) Continue filling the reactor and transferring to ST 15 until the tank is full (approximately 45,000 lbs). Once ST 15 is full, then begin filling the reactor. Leave the reactor full (approximately 13,000 to 17,000 lbs) in preparation for the inert strip step. At this point ST 15 and the reactor should be full of Charge A (approximately 58,600 lbs total).
10. Using the single stage vacuum, add **Charge B (11-0000-3179-6)**, Filter Cel to the reactor through the bottom drain line.

III INERT STRIP

11. Set the system to begin stripping inerts as follows:

Parameter	Desired Setting
Reactor Agitator	Turn On and set to run at 70 rpm
Reactor Jacket Select	Circulating Water until the batch temp reaches 220 F
Reactor Jacket High Limit	350 F
Delta T set point	50
Reactor Jacket Control Mode	DP with a set point of 100 mmHg. This will record the DP but the Delta T will be the limiting set point.
Condenser Select	Cooling
Condenser Water Temp Set point	55 F
Over head Valve Select	Rout the distillate to return to the reactor during heat up
Splitter Valve Setting	Reflux/Takeoff
Splitter Settings	For head temps less than 210 F –10 sec takeoff & 5 sec reflux. For head temps greater than 210 F – 5 sec takeoff & 10 sec reflux
Scrubber Vent	Open for Atmospheric Distillation of inerts
Receiver Jacket Select	Full Cooling

12. Once the head temperature rises and the DP levels out then switch the Over head valve select to take off to the new receiver.
13. Distill off FM-3160 inerts to the receiver. Adjust the Delta T set point to maintain a **takeoff rate of 1,200-1,800 lbs/hr**. At rates below 1200 lb/hr, the column efficiency decreases. Increasing Delta T will increase the jacket temperature, boil up rate and take-off rate. Decreasing Delta T will decrease jacket temperature and boil up rate.
14. When the **head** temperature reaches **210 °F**, set the reflux timers for **5 sec takeoff** and **10 sec reflux**.
15. When the **batch** reaches **210-220°F**, drain the jacket, and switch the jacket to **direct steam**.

PPE Note: Refer to Attachment 3 for the PPE requirements for handling F-6002 and FM-3160.

16. Drain the FM-3160 inerts at the end of each shift (do not fill receiver to more than 12,000 lbs before draining. At the end of each shift drain off what has been distilled). **The inerts should always be drained to drums.** Refer to the by-product draining information section for the appropriate drum number and draining specifications. If drums are not available then contact the manufacturing engineer. **Temporary** storage in totes or in a storage tank may be required. If **temporary** storage is required then refer to **Attachment 2** for instructions for draining the inerts.

17. As the FM-3160 inerts are stripped off, a portion of **Charge A** should be added to the reactor from Storage Tank 15. Fill up the reactor at least once per hour. Smaller more frequent additions are preferable to large slugs. Check the Batch temperature of ST 15. If ST 15 is over heated (greater than 160 F) then the material should be transferred very slowly to prevent overheating and bumping over the material in the Reactor.

18. Continue to distill inerts to the receiver and add additional Charge A to the reactor from ST 15, until the following occur:
 - a) Storage Tank 15 is empty and no more charge A is available or
 - b) Batch temperature equals **285 °F** and the reactor is near full. **Some head space must be left in the reactor in order to begin the fractionation. Two to three feet for space between the top of the liquid and the sight glass is adequate.**
19. When the above conditions are met, then set the reactor jacket mode to CIRC-WATER and set the jacket control to BATCH-AUTO with a set point of 170°F.
20. While cooling to 170°F, maintain splitter valve settings, but route distillate to return to reactor.

PPE Note: Refer to Attachment 3 for the PPE requirements for handling F-6002 and FM-3160.

21. Drain all of the material in the receiver. Drain the bottom inert phase to drums as FM-3160 per the by-product draining section. Drain the top water phase as F-6002 per the by-product draining section. If drums are not available then temporary storage may be required. Contact the manufacturing engineer and refer to Attachment 2 for temporary storage procedures for inerts.

IV FIRST PRE CUT (100 mm Hg up to 245 °F head temperature)

22. If there is any Charge C (FM-3210) scheduled to be added, charge to the receiver and transfer to the reactor after the inert strip is complete by performing the following:
 - a) Verify there is enough head space in the reactor for the additional charge C material.
 - b) Verify the batch has cooled to 170 F.
 - c) Isolate the receiver from the reactor.
 - d) Using the single stage vacuum, pull down to the maximum vacuum on the receiver and isolate.
 - e) Add **Charge C (41-2700-3210-9)** to the receiver using isolated vacuum. Isolated vacuum must be used to prevent loss of low boiling materials out the vacuum system.
 - f) Vent the reactor to the scrubber and use nitrogen to pressure transfer the material from the receiver to the reactor. Be sure to not over fill the reactor. Two to three feet of headspace is required in the reactor once

charging is complete.

23. With the batch temperature at 170°F, pull vacuum on reactor system for first precut. Verify the scrubber Vent is closed, Set the vacuum loop to "all stages" and lower the vacuum to **100 mm** Hg. Vacuum must be lowered slowly to prevent boilovers or bumping material into the packed column.

24. When the vacuum is 100 mm Hg, set the following to the desired settings:

Parameter	Desired Setting
Reactor Agitator	Turn On and set to run at 70 rpm
Reactor Jacket Select	Circulating Water until the batch temp reaches 220 F , then drain the jacket and set to Direct Steam (see step below)
Reactor Jacket High Limit	350 F
Delta T set point	50 increase as necessary until strong reflux is established. (see step below)
Reactor Jacket Control Mode	DP with a set point of 100 mmHg. This will record the DP but the Delta T will be the limiting set point.
Condenser Select	Cooling
Condenser Water Temp Set point	55 F
Over head Valve Select	Rout the distillate to return to the reactor during heat up
Splitter Valve Setting	Reflux/Takeoff
Splitter Settings	5 seconds takeoff and 20 seconds reflux (Reflux ratio 4:1)
Vacuum Level	100 mmHg
Receiver Jacket Select	Full Cooling

25. Increase the Delta T as necessary to establish a strong reflux back to the reactor
26. When the head temperature is stable for 10 minutes, start takeoff to the receiver. Monitor take-off rate and adjust the delta T to maintain a take-off rate of **300-500 lbs/hr**. With a reflux ratio of 4:1 the column efficiency will decrease if take off rate is less than 300 lbs/hr (i.e. the boilup rate should be between 1200 and 2000 lbs/hr).
27. When the batch reaches 210-220°F, drain the jacket, and switch the jacket to "STEAM" mode.
28. Distill precut to the receiver until the head temperature reaches 245°F at 100 mm Hg. Expect 1,500 to 3,500 lbs of distillate in the receiver for normal batches. **The first pre-cut will be taken as a total cut in the receiver and then drummed.**
29. When the precut is complete, switch vacuum directly to the reactor. Continue to operate column with the same reflux timer settings, routing take-off to the reactor.
30. Set the reactor jacket high limit to equal the current jacket temperature. (This will prevent the jacket from overheating and will maintain the current boil up rate while the receiver is being drained).
31. Isolate receiver from reactor.
32. Break vacuum on the receiver with nitrogen, and pressure the receiver to

20 psig with nitrogen.

PPE Note: Refer to Attachment 3 for the PPE requirements for handling FM-3210, F-6002, while sampling.

33. Drain the precut to polyoverpak drums. Label the drums based on the C8 and High Boiler QC results. **Label each drum with consecutive drum numbers (1, 2, 3...?) starting with the precut and continue through the post cuts.** If the C8 content is less than 10%, the QC lab may only report the C8 value. Drums with less than 10% C8 should be labeled as scrap (include scrap drums when numbering drums for the fractionation). Refer to **Attachment 1** for labeling information based on lab results. Watch for an upper water phase while draining. Drain any water phase as by-product 41-2600-6002-9. Do not include drums of F-6002 drums when numbering drums for the fractionation.

"SAMPLE #1".

Take one, 8-ounce in-process sample while draining. Label the sample as FM-3256 Lot ___, Dr ___

V SECOND PRE CUT (10 mm Hg up to Main Cut purity))

PPE Note: Refer to Attachment 3 for the PPE requirements for handling FM-3257, F-8281, F-8282

34. If there is any Charge D scheduled to be added, then an estimated amount will be listed on the run card. A list of specific drums and load ID's will also be provided. After draining the precut, add any **scheduled Charge D1 (41-2700-3257-0), Charge D2 (41-2600-8281-7) or Charge D3 (41-2600-8282-5)**, to the receiver and transfer to the reactor after the first pre-cut is complete by performing the following:

- a) Verify that there is enough head space in the reactor for the additional charge D material.
- b) Cool the batch to 170 F by setting the reactor jacket to circulating water with a set point of 170 F. (cooling down is needed since the charge D material can contain lower boiling materials and can be flashed off during vacuum charging).
- c) Isolate the receiver from the reactor.
- d) Using the single stage vacuum, pull down to the maximum vacuum on the receiver and isolate.
- e) Add the **Charge D material/s** to the receiver using isolated vacuum.
- f) Use nitrogen to pressure transfer the material from the receiver to the reactor. Be sure not to over fill the reactor. Two to three feet of headspace is required in the reactor once charging is complete.

PPE Note: Refer to Attachment 3 for the PPE requirements for handling RM-3048.

35. Add **Charge E (11-0000-3048-3)**, Sulfuric acid to the receiver then transfer to the reactor by performing the following:

- a) Verify that there is enough head space in the reactor for the Charge E

material.

- b) Cool the batch to 170 F by setting the reactor jacket to circulating water with a set point of 170 F. (cooling down is needed since the batch can still contain some lower boiling materials and can be flashed off during vacuum charging).
- c) Isolate the receiver from the reactor.
- d) Using the single stage vacuum, pull down to the maximum vacuum on the receiver.
- e) Add the **Charge E (11-0000-3048-3)**, Sulfuric acid to the receiver using vacuum.
- f) Use nitrogen to pressure transfer the material from the receiver to the reactor. Be sure not to over fill the reactor. Two to three feet of headspace is required in the reactor once charging is complete. If the entire amount of Charge E will not fit into the reactor then add the remaining portion once more of the pre-cut or second pre-cut is distilled off
- g) When the transfer is complete use single stage vacuum to pull maximum vacuum on receiver.
- h) Isolate the receiver from single stage vacuum system then switch back to BC-34's vacuum system on the receiver and open the equalizing line.

36. Begin the Second Pre-Cut Fractionation by setting the following parameters to the desired settings:

Parameter	Desired Setting
Reflux routing	Route the reflux back to the reactor while heating
Splitter settings	Reflux Ratio—6:1 [5 seconds takeoff and 30 seconds reflux]
Reactor Jacket Settings	Control Mode = DP control Jacket Select = Direct Steam High Limit = 350 F (Once the vacuum levels out set to 380 F)
Delta T Set point	100°F—[The Delta T set point can be used to limit the jacket temperature and swings in steam pressure.]
D.P. Set point	30 to 50 mmHg [Adjust to maintain a take off rate of 200 to 300 lbs/hr (see following steps)]
Condenser water Set point	Initially set to 80 F (see note below)
Steam Tracing	Turn on all overhead piping and receiver drain line steam tracing.
Take off rate	200 to 300 lbs/hr [rate will decrease as the fractionation continues]
Receiver Jacket Settings	Control Mode = Batch Auto Jacket Select = Circulating Water Jacket high limit = 175 F Batch Set Point = 130 F
Vacuum Set Point	10 mmHg
Receiver draining interval	Drain after every 600 lbs to 1200 lbs until Main-Cut purity

37. Adjust the *D.P.* to 30-50 mm Hg to establish a steady and strong take-off. Maintain take-off to the reactor until the head temperature is stable for 10 minutes.
38. When the head temperature is stable, change the overhead valve select to start take-off to the New receiver.
39. Raise the condenser water temperature set point slowly throughout the second precut up to 120 F. Use the column head temperature as the basis for setting the condenser water set point (see note below).

Condenser water note:

Raising the condenser water temperature too fast may make it impossible to attain the desired 10 mm Hg vacuum. The Condenser water temperature should not be higher than (Column head temperature - 90 °F) with the max at 120 F. Once the C8 acid content of the product is greater than 85% then the condenser water should be set at 120 F. Example: head temperature = 190 °F, the condenser water temperature should be set at 100 °F.

40. Continue the second precut distillation to the receiver. Monitor take-off rate and adjust the column *D.P.* setpoint to maintain a take-off rate of 300-400 lbs/hr. With a reflux ratio of 6:1 the column efficiency will decrease if take off rate is less than 300 lbs/hr (i.e. the boilup rate should be between 1200 and 2000 lbs/hr).
41. Check the steam trace at least once per shift to verify it is hot and functioning.
42. **Drain the receiver every 600 to 1200 pounds per steps 43 and 44 until 96% C8 acid purity is reached. When the C8 is 96% or greater then go to the Main Cut Section**

43. Perform the following when Draining the receiver:

- a) Switch vacuum directly to reactor.
- b) Set the reactor jacket high limit to the current Jacket temperature.
(This will prevent the jacket from overheating and will maintain close to the current boil up rate while the receiver is being drained).
- c) Continue to operate column with the same reflux timer settings, routing take-off to the reactor.
- d) Isolate receiver from reactor.
- e) Verify the drain line and sample tap are not plugged by first opening the receiver bottom drain valve and then open the sample tap valve, allowing a small amount of air to be sucked into the receiver while the receiver is still under vacuum. Special care must be taken when performing this to not loose vacuum and drain any material out. Re-close the valves when the test is complete.
- f) With the receiver drain valve closed, Pressure the receiver to 20 psig with nitrogen.

PPE Note: Refer to Attachment 3 for the PPE requirements for handling FM-3257, F-8281, FM-3256, F-8282, F-4169 and Sample 1.

- g) Drain the receiver to the appropriate containers as specified in the Draining Information Section.
- h) Collect one, 4-ounce in-process sample (fill an 8 oz jar ½ full). Label the sample as FM-3256 Lot __, Dr __, "**SAMPLE #1**".
- i) Label the drums based on the C8 and High Boiler QC results. **Label each drum with consecutive drum numbers (1, 2, 3...?) starting with the precut and continue through the post cuts.** Refer to Attachment 1 for labeling information based on lab results. .

44. When draining is complete perform the following:

- a) Use single stage vacuum to pull maximum vacuum on receiver.
- b) Briefly crack open the drain valves and allow a small amount of air to be sucked into the receiver (be sure to clear the sample tap out as well). If the drain valve/line is plugged than use nitrogen to blow out the line into the receiver. Once the line is cleared then close all of the valves in the drain line.
- c) Isolate receiver from single stage vacuum system then switch back to BC-34's vacuum system on the receiver.
- d) Reset the jacket high limit to 380°F after column D.P. levels out with the reactor at 10 mmHg.

- e) Maintain take-off routed to the reactor until the normal vacuum level, D.P. level, and a steady head temperature are re-established, then switch take-off to the receiver.
45. When the % C8 of the last draining is 96% or greater, proceed to the Main Cut section.

VI MAIN CUT

46. When the QC results for the last draining are greater than or equal to 96% C8 then stop draining at one drum intervals and begin collecting the main cut in the receiver.
47. Distill the Main Cut at the conditions listed below:

Parameter	Desired Setting
Vacuum level	10 mmHg
Splitter settings	Reflux Ratio—6:1 [5 seconds takeoff and 30 seconds reflux]
Delta T Set point	100°F—[The Delta T set point can be used to limit the jacket temperature and swings in steam pressure.]
<i>D.P.</i> Set point	30 to 50 mmHg [Adjust to maintain a take off rate of 200 to 300 lbs/hr]
Take off rate	200 to 300 lbs/hr [rate will decrease as the fractionation continues]
Condenser Temperature	120°F
Receiver draining interval	Only after the end point of the product/main cut refer to Step 48

Note: The Main Cut will be taken as a total cut to the receiver and then drummed. However, do not wait so long before draining that HB'ers get into the maincut. It is imperative that the jacket steam pressure and the D.P. be closely monitored to determine the first sign to end the maincut and drain the receiver.

48. Continue the Main Cut distillation to the receiver until one or more of the following occur:
- The D.P. drops off from its steady state value.
 - The jacket temperature rises to 300°F
 - The jacket steam pressure rises sharply
49. Once any of the above conditions are met then drain the receiver per steps 43 and 44. Collect the required sample and continue to the next step.
50. Continue with the Main Cut fractionation at the conditions specified above. Drain the receiver per steps 43 and 44 in 600 to 1200 pound increments until the high boilers in the product exceed 3.0%.
51. When the high boilers in the product exceed 3%, **and** if there is an amount of Charge F specified on the run card, proceed to the FIRST POST CUT Section. If No charge F is scheduled for this run then, proceed to the SECOND POST CUT Section.

VII FIRST POST CUT

Note: This cut is only done if F-4169 is added to the reactor. If F-4169 is not scheduled to be added during this run, then proceed to section VII. SECOND POST CUT.

PPE Note: Refer to Attachment 3 for the PPE requirements for handling F-4169.

52. If there is any Charge F scheduled to be added, then an estimated amount will be listed on the run card. Once the HB content in the product is greater than 3%, add any **scheduled Charge F (41-2600-4169-8)** directly to the reactor by performing the following:

- a) Verify that there is enough head space in the reactor for the additional charge F material.
- b) Cool the batch to 170 F by setting the reactor jacket to circulating water with a set point of 170 F. (cooling down is needed since the charge F material contains high concentrations of C8 and can be flashed off during vacuum charging at higher temperatures).
- c) Using vacuum on the reactor pulled through the receiver, overhead, and through the equalizing line add the **Charge F (F-4169)** to the reactor. Be sure not to over fill the reactor. Two to three feet of headspace is required in the reactor once charging is complete.

53. Once the charging is complete then begin the 1st Post Cut fractionation by setting the following:

Parameter	Desired Setting
Reflux routing	Route the reflux back to the reactor while heating
Splitter settings	Reflux Ratio—10:1 [5 seconds takeoff and 50 seconds reflux]
Vacuum Set Point	10 mmHg
Reactor Jacket Settings	Control Mode = DP control Jacket Select = Direct Steam High Limit = 350 F (Once the vacuum levels out set to 380 F)
Delta T Set point	Initially set to 15°F Then set to 100°F once the head temperature comes up and the DP levels out [The Delta T set point is used to limit the jacket temperature and swings in steam pressure.]
D.P. Set point	30 to 50 mmHg [Adjust to maintain a take off rate of 200 to 300 lbs/hr]
Condenser water Set point	120 F
Steam Tracing	Turn on all overhead piping and receiver drain line steam tracing.
Take off rate	200 to 300 lbs/hr [rate will decrease as the fractionation continues]
Receiver Jacket Settings	Control Mode = Batch Auto Jacket Select = Circulating Water Jacket high limit = 175 F

	Batch Set Point = 130 F
Receiver draining interval	Drain after every 600 lbs to 700 lbs

54. Adjust the *D.P.* to 30-50 mm Hg to establish a steady and strong take-off. Maintain take-off to the reactor until the head temperature is stable for 10 minutes.
55. When the head temperature and DP are stable, start take-off to the receiver.

56. To maintain the proper take-off rate (and good fractionation) it will be necessary to make adjustments in the *D.P.* setpoint. Maintain a product takeoff rate of **200-300 lb/hr**. Rate will drop below this near the end of the cut.

Note: Check the steam trace at least once per shift to verify that it is hot and functioning.

57. When 600 lbs of distillate has been collected in the receiver, switch vacuum directly to reactor and drain the receiver per steps 43 and 44.

PPE Note: Refer to Attachment 3 for the PPE requirements for handling F-8282, F-4169, and Sample 1.

58. Continue fractionation, draining in one drum increments until the high boilers in the product exceed 3.0%. Drain the receiver per steps 43 and 44.

59. When the high boilers in the product exceeds 3%, proceed to section VII. **SECOND POST CUT (TOTAL TAKEOFF).**

VIII SECOND POST CUT (total take off)

Note: The second post-cut will be taken as a total cut to the receiver then drummed.

60. The following parameters should be entered to begin the second post cut distillation:

Parameter	Desired Setting
Over head valve Select	Rout the distillate to the reactor while heating and pulling vacuum.
Splitter settings	Total take off
Vacuum level	1 mmHg
Delta T Set point	100°F
Jacket High Limit	395°F [The jacket high limit can be used to limit temperature swings and steam pressure when draining by setting to the current batch temp.]
D.P. Set point	30 to 50 mmHg [Adjust to maintain a strong take off rate]
Take off rate	200 to 300 lbs/hr [rate will decrease as the fractionation continues]
Condenser Temperature	120°F
Receiver draining intervals	Only at the end point of the distillation (refer to the following steps)

61. Maintain take-off to the reactor until the head temperature is stable for 10 minutes, then switch to take-off to the receiver.

62. Distill the batch to get as much C8 acid out of the bottoms as possible. Stop the distillation when **all** of the following occur:

a) Take off rate drops to below 30 lbs per hour.

- b) The batch temperature is at greater 300 °F
 - c) Jacket Temperature is greater than at 375 °F (target 390 °F).
 - d) The vacuum level is at maximum [target less than 5mmHg]
63. Isolate the receiver from the reactor and drain the receiver per steps 43 and 44. Collect the required sample and label the drums accordingly. Refer to **Attachment 1** for labeling information based on lab results.

IX. DRAINING BOTTOMS

Note: Once the fractionation is complete any material in the reactor should be drained to drums. Do not drain any fractionation bottoms to the sewer.

64. Cool the bottoms by setting the following:
- a) Set the reactor jacket control mode to Batch Auto
 - b) Set the reactor jacket select to circulating water.
 - c) Set the reactor batch temperature set point to 160 F
 - d) Set the Delta T to 50 F.

Note: If there is any delay in draining the reactor, then place the jacket in Circulating water with a batch set point of 200 °F.

65. When the batch temperature has dropped to 160 °F, slowly add about 500 lbs of water to the reactor. (if the total volume in the reactor after the water addition does not reach the agitator blades then add enough water to reach the blades and to allow some mixing.
66. Mix the water and bottoms solution for ½ hr.
67. Verify the bottom drain line is not plugged by:
- a) Pull vacuum on the reactor to about 100 mmHg (do not pull down to less than 100 mmHg or water may start to boil).
 - b) Crack open the drain valve and allow a small amount of air to be sucked into the reactor.
 - c) If the drain valve/line is plugged than use nitrogen to blow out the line into the reactor.
 - d) Once the line is clear then close the bottom drain valve and break vacuum with nitrogen.
68. Pressure the reactor to up to 10 psig with nitrogen.

PPE Note: Refer to Attachment 3 for the PPE requirements for draining

bottoms.

69. Use 10-20 psig nitrogen pressure to drain the water and bottoms solution to drums per the waste disposal section. Drain 600 lbs to each drum. Label each drum with the waste stream labels (13-0068-1432-4)
70. Once the bottoms are drained then clear out the drain line and drain hose by turning on the vacuum system and sucking air into the reactor through the drain line. Do not wash out the hose. This hose will be used to charge the methanol cleaning solution and will be cleaned out as part of the post run cleanup. Do not leave product in the hose or it will freeze and plug up the hose. If there is a delay in charging the methanol then the hose can be briefly flushed out with hot water if needed.

X. POST RUN CLEANUP

Note: No cleanup is necessary if another FM-3256 run is immediately following this run.

71. Once the fractionation bottoms have been drained, Prepare to add Methanol by inerting the reactor and receiver as follows:
- Use the single stage vacuum on the receiver to pull maximum vacuum on the receiver and on the reactor through the overhead.
 - Isolate the vessels from the vacuum system and break vacuum using nitrogen.
 - Re-pull vacuum on both vessels, isolate and break vacuum using nitrogen for a total of three times.

PPE Note: Refer to Attachment 3 for the PPE requirements for handling RM-3009.

72. After inerting the vessels, add Charge Z1, RM-3009, Methanol to the reactor by performing the following:
- Set the reactor agitator to run at 90 rpm
 - Set the receiver jacket to full cooling
 - Set the reactor jacket to full cooling.
 - Set the overhead condenser to cooling and set the water temp set point at 55 F
 - Use maximum **isolated** vacuum to add **Charge Z1 (RM-3009 Methanol)** to the reactor. Be sure to ground and bond all Methanol drums before opening and while charging.
 - Once all of the methanol has been added then break vacuum with nitrogen.
73. Add enough **Charge Z2 Water (RM-0995)** to the reactor so the liquid level reaches the agitator blades. In order to minimize waste, do not add too much water.
74. Boil the methanol through the overhead by setting the following:

Parameter	Desired Setting
Reactor Agitator	Turn On and set to run at 70 rpm
Reactor Jacket Select	Circulating Water
Reactor Jacket High Limit	300 F
Delta T set point	25
Reactor Jacket Control Mode	Batch Auto
Reactor Batch Temp Set Point	200 F
Condenser Select	Cooling
Condenser Water Temp Set point	55 F
Over head Valve Select	Route the distillate to the reactor (see following steps)
Splitter Valve Setting	50 sec take off 10 sec reflux (to clean out the reflux line)
Scrubber Vent	Open for Atmospheric Distillation

Receiver Jacket Select	Full Cooling
------------------------	--------------

75. Reflux the through the overhead and back to the reactor for one hr. Adjust the Delta T to establish a steady reflux.
76. After refluxing back to the reactor for one hr, switch the overhead valves to take off to the New Receiver.
77. When about $\frac{1}{2}$ to $\frac{3}{4}$ of the **methanol** has been collected in the receiver, set the batch temperature set point to 90 F.

78. Once the material has cooled to 120 F then use nitrogen pressure to transfer the material in the receiver back to the reactor. The material may exotherm. Control the transfer rate so the reactor batch temperature does not exceed 140 F.
79. Continue mixing the material in the reactor until the batch temperature is at 90 F.
80. Once the material is at 90 F, then transfer the methanol, water, and FC material to the Building 5 scrap solvent tank.
81. Fill the system with water, and flush the entire system (Reactor, packed column, overhead, and receiver, reflux return line to the reactor, and transfer line from the receiver to the reactor).
82. Drain flush water to the sewer. Be sure to drain all overhead lines, receiver, reactor and any transfer lines.
83. Open the reactor and receiver manholes and inspect. If additional cleanup is necessary, then repeat the methanol boil (be sure to inert the system) and contact the supervisor.

MAINCUT DRAINING INFORMATION:

Containers	New black poly overpack (34-7039-5732-3)
Labeling	Lot, ___ Net ___, Drum ___, [Refer to Attachment 1 for labeling information based on lab results.]
Storage	Hold in Bldg. 15 Hot Room for charging into F-7164
Filter	None
Weight per Container	600 pounds
Draining Temperature	120 – 140°F
Draining Pressure	0 to 5 psig

Special Draining Instructions:

To avoid C8 setting up in the drain line, while pulling vacuum, briefly suck air through the drain line into the receiver before shutting drain valve.

Sample Requirements:

- Inerts: 41-2700-3160-6 None
- Precut: 41-2700-3210-9 1 in-process sample each time the receiver is drained.
- Intercut: 41-2700-3257-0 1 in-process sample each time the receiver is drained.
- Maincut: ~~41-2700-3256-2~~ 41-2700-3256-2 1 in-process sample each time the receiver is

drained.

Postcut: 41-2600-4169-8 1 in-process sample each time the receiver is drained.

BY-PRODUCT DRAINING INFORMATION:

Step	16 & 21
Description	C ₈ Cyclic Inert Mixture
Container	1st choice --- Green remanufactured poly overpack drums 2nd choice --- Blue New poly overpack drums
Disposition	Remove drums to the warehouse
Labeling	Drums: 41-2700-3160-6 , Lot ____, Net ____, Dr ____ (Record byproduct yield on card)
Wt per Container	600 lbs net for drums
Amount	20,000 - 30,000 lbs.

Step	16 & 21
Description	Water phase from inert stip
Container	1. Recycle poly overpak drums (34-7010-1156-0) 2. Remanufactured poly overpak drums (34-7029-4109-6) 3. New poly overpaks (34-7002-2745-6)
Disposition	Remove to warehouse
Labeling	41-2600-6002-9 , Lot ____, Net ____, Dr ____
Wt per Container	400 lbs net
Amount	Up to 4,000 lbs.

Step	33
Description	First Precut
Container	New black poly overpak drums (34-7039-5732-3) or Used F-8281, F-8282, FM-3256, FM-3257, FM-3210 black poly overpak drums
Disposition	Remove to warehouse
Labeling	41-2700-3210-9 , Lot ____, Net ____, Dr ____
Wt per Container	600 lbs net
Amount	1,800-2,400 lbs

Step	42
Description	Second Precut
Container	New Black Polyoverpaks (34-7039-5732-3) or Used FM-3256 drums
Disposition	Remove to Bldg 15 or Bldg 3 hot room
Labeling	41-2700-3257-0 , Lot ____, Net ____, Dr ____
Wt per Container	600 lbs net
Amount	1,200-1,800 lbs

Step	42
Description	Second Precut
Container	New Black Polyoverpaks (34-7039-5732-3)
Disposition	Remove to Bldg 15 or Bldg 3 hot room
Labeling	41-2600-8281-7, Lot ____, Net ____, Dr ____
Wt per Container	600 lbs net
Amount	Up to 1,200 lbs

Step	57 & 58
Description	First post cut
Container	New Black Polyoverpaks (34-7039-5732-3)
Disposition	Remove to Bldg 15 or Bldg 3 hot room
Labeling	41-2600-8282-5, Lot ____, Net ____, Dr ____
Wt per Container	600 lbs net
Amount	Up to 1,200 lbs

Step	63
Description	Second Post-cut
Container	New Black Polyoverpaks (34-7039-5732-3) or Used F-8281, F-8282, FM-3256, FM-3257, FM-3210, F-4169 black poly overpak drums
Disposition	Remove to warehouse
Labeling	41-2600-4169-8, Lot ____, Net ____, Dr ____
Wt per Container	600 lbs net
Amount	Up to 1,200 lbs

WASTE DISPOSAL:

Step	33 & 63
Description	Pefluorooctanoic Acid (< 10% C8) for Scrap Disposal
Container	Remanufactured poly overpak drums (34-7029-4109-6) or New poly overpacks (34-7002-2745-6) Used Charge A drums are also acceptable to use if any are available
Disposition	Incinerator
Labeling	13-0017-0281-3 , (Refer to Waste Stream Profile)
Wt per Container	600 lbs net
Amount	Up to 1,200 lbs

Step	69
Description	Sulfuric Acid and Fractionation Bottoms and water
Container	Remanufactured poly overpak drums (34-7029-4109-6) or New poly overpacks (34-7002-2745-6). Used Charge A drums are also acceptable to use if any are available
Disposition	Incinerator
Labeling	13-0068-1432-4 (Refer to waste stream profile)
Wt per Container	500 to 600 lbs
Amount	2000 to 3000 lbs

Step	80
Description	Methanol, water, and Fluorochemical compounds
Container / Disposition	Transfer to Building 5 scrap solvent tank
Labeling	None
Amount	Up to 6000 lbs

Step	9
Description	Acid Salts, Inerts, and FC Acid residue in the Charge A drums
Container	Leave in the original drums which contained the Charge A material.
Disposition	Incinerator
Labeling	13-0028-8398-4 , (Refer to Waste Stream Profile)
Wt per Container	25 up to 100 lbs net
Amount	Up to 1000 lbs

Attachment 1 Labeling Information For All Cuts:

% C8	% HB	Label as	Description
Pre Cuts			
0.0 - 9.9	0	13-0017-0281-3	Scrap
10.0 - 69.9	0	41-2700-3210-9	First Precut
70.0 - 89.9	0	41-2700-3257-0	Second Precut
Note: If the high boiler content of any precut drums are greater than 0.6% then these drums should be labeled as F-8282.			
Main Cuts			
90.0 - 96.0	0.0 - 0.5	41-2600-8281-7	Main Cut
96.1 - 100.0	0.0 - 0.5	41-2700-3256-2	Main Cut (Heart cut)
97.0 - 99.4	0.6 - 3.0	41-2600-8282-5	Main Cut
Post-cut			
10.0 - 96.9	3.1 - 89.9	41-2600-4169-8	Postcut
0.0 - 10.0	90-100	13-0017-0281-3	Scrap

Sample Requirements:

- Inerts:.....41-2700-3160-6 None
- Precut:.....41-2700-3210-9 1 in-process sample each time the receiver is drained.
- Intercut:...41-2700-3257-0 1 in-process sample each time the receiver is drained.
- Maincut:~~41-2700-3256-2~~ 1 in-process sample each time the receiver is drained.
- Postcut:...41-2600-4169-8 1 in-process sample each time the receiver is drained.

Labeling Requirements:

Use consecutive drum numbers throughout the fractionation for all cuts.
Label each drum with consecutive drum numbers (1, 2, 3...?) starting with the precut and continue through the post cuts. Do not include FM-3160, or F-6002 drums, in the numbering sequence. If the C8 content is less than 10%, the QC lab may only report the C8 value. Drums with less than 10% C8 should be labeled as scrap (include scrap drums when numbering drums for the fractionation. These are usually the first 1 or 2 drum if any).

Attachment 2 Temporary inert storage procedures

FM-3160 inerts should always be drained to drums unless specified by the manufacturing engineer. The storage tank or totes should only be used as a last resort for temporary storage since only drummed material can be shipped. Using the storage tank or totes will require extra handling since the material will need to be re-charged into the reactor or receiver and drained to drums before shipping.

Use of the Out Side Inert Storage Tank. ST 98

Transferring inerts from bc-34 to St 98

1. Verify the bulk storage tank level is less than 96%. If the level is greater than 96%, the inerts will have to be drained to another alternative.
2. Verify that the pressure in the storage tank is between 40 psig and 20 psig. If the storage tank pressure is greater than 40 psig, vent the storage tank through the reactor overhead system with condenser water on at 55°F.
3. If the pressure is less than 20 psig investigate the status of the nitrogen line to the tank. The automatic nitrogen block valve should be open. This valve should automatically open when the pressure in the tank is less than 20 psig and will close if the pressure is greater than 40 psig. (the tank is not rated for vacuum).
4. Perform a pressure test on the tank and transfer lines before initiating the transfer of inerts.
5. Open the automatic valve in the charge line on top of Storage Tank 98. If the tank level is above 98%, or if the tank pressure is above 50 psig, the charge valve will automatically close.
6. Use nitrogen to pressurize the receiver to 30 psig. **Do not exceed 35 psig of nitrogen pressure.** Pressure transfer the inerts(FM-3160) to the bulk storage tank. Continue transferring until the receiver is empty or until a top water phase is detected. Avoid transferring any water to the bulk tank. Drain any water phase as By-Product 41-2600-6002-9 and remove.
7. Once the inert strip is complete and the receiver is empty, then blow out the inert transfer line to the top of the tank. **Do not let the tank pressure exceed 35 psig.** If the pressure exceeds 35 psig then vent off the excess pressure through the reactor overhead system with condenser water set at 55°F
8. When the inert transfer is complete, vent the receiver pressure slowly through the reactor overhead.
9. Record the amount of FM-3160 inerts transferred as Byproduct on the run card

Transfer inerts from st 98 to bc-34

1. Check the pressure reading of ST98. If the pressure is lower than 20 psig then open the automatic Nitrogen valve at the top of the tank and pressure the tank up to 35 psig.
2. The inerts can be transferred from ST 98 to the BC-34 receiver (an alternate choice is also BC-33 receiver if authorized by the shift supervisor and the manufacturing engineer). Open all necessary hand valves in the inert transfer line to direct the flow from the bottom of the storage tank to the bottom of the receiver.
3. Open the automatic valve on the bottom of the storage tank and begin transferring the desired amount to the receiver (up to 17,500 lbs for BC-34 receiver and 14,000 lbs max for BC-33 receiver).
4. Vent off any pressure on the receiver through the overhead system with the condenser set to Cooling with the inlet water set point of 55°F.
5. Once the desired amount has been transferred, then close the automatic valve on the bottom of the storage tank.
6. The inerts should now be drained from the receiver into drums for shipping as specified in the By-product draining section
7. When draining is complete then blow out the transfer line with nitrogen. Do not leave the transfer line liquid full for any extended period of time. Inerts can vaporize in the line and over pressure the line.

Use of totes for FM-3160

Note: Totes should only be used if drums are not available. The totes are not certified for shipping.

PPE Note: Refer to Attachment 3 for the PPE requirements for handling FM-3160.

1. The inerts should be drained to poly totes normally used for F-6057 from the spray dryer room. These are poly totes with blue steel framing. Generic stainless steel totes can also be used as an alternate if the poly totes are not available.
2. Continue Draining totes until the receiver is empty or until a top water phase is detected. Avoid transferring any water to the containers. Drain any water phase as By-Product 41-2600-6002-9 and remove.

Attachment 3

Fractionation Of Recycled C8 Acid Pre-Cuts & Post-Cuts

If the only charges for this run are recycled fractionation pre-cuts and post-cuts and no Charge A1, A2, or A3 material is to be added, then perform the required actions based on the following:

- 1 If FM-3210 is **scheduled** to be charged:
 - Perform the pre-run preparation
 - Skip the charging section
 - Skip the inert strip section
 - Add the scheduled amount of FM-3210 per step 22 (if the reactor is empty then the FM-3210 can be vacuumed directly into the reactor. Pull vacuum through the overhead when adding directly to the reactor).
 - Complete all remaining steps of the standard (steps 22 through 83)

- 2 If FM-3210 is **not** scheduled and FM-3257, or F-8181 or F-8282, or FM-3256 rework, or up to 2000 lbs of F-4169 are the only charges:
 - Perform the pre-run preparation
 - Skip the charging section
 - Skip the inert strip section
 - Skip the First pre-cut section
 - Add the scheduled amount/s of FM-3257, F-8281 or FM-3256 rework per step 34. (if the reactor is empty then the materials can be vacuumed directly into the reactor. Pull vacuum through the overhead when adding directly to the reactor).
 - Note:** if less less than 2000 lbs of F-4169 is scheduled than add it to the reactor with the above materials per step 34. If more than 2000 lbs of F-4169 is scheduled then wait to charge it until the First post cut.
 - Complete all remaining steps of the standard (steps 34 through 83)

- 3 If F-4169 is the only material scheduled to be charged:
 - Perform the pre-run preparation
 - Skip the charging section
 - Skip the inert strip section
 - Skip the First pre-cut section
 - Add in Charge E, RM-3048 Sulfuric acid per step 35 (if the reactor is empty then the materials can be vacuumed directly into the reactor. Pull vacuum through the overhead when adding directly to the reactor).
 - Skip the rest of the Second Pre-cut section
 - Skip the Main-cut Section
 - Add the scheduled amount of F-4169 per step 52. (if the reactor only

contains sulfuric acid then the F-4169 can be vacuumed directly into the reactor. Pull vacuum through the overhead when adding directly to the reactor).

Complete all remaining steps of the standard (steps 52 through 83)

**PERSONAL PROTECTIVE EQUIPMENT REQUIREMENTS FOR
PRODUCT NUMBER: FM-3256**

Hazard Level Definitions:

Engineer: Dean Graham

- E=Extreme: Likely to cause irreversible tissue damage, serious illness or death from contact or exposure with a very small amount.
 H=High: May cause extreme irritation, irreversible tissue damage, serious illness or death from single or repeated contact or exposures.
 M=Medium: May cause moderate to severe irritation; may cause allergic reaction (sensitization); may cause reversible systemic effects.
 L=Low: May cause mild temporary irritation on contact; no cumulative effects are expected from repeated contact or exposures.

11-Digit Code	Material Name and Description	PHYSICAL DATA			HAZARD LEVEL			EYES		SKIN			LUNGS				Comments
		Physical state	Flammable (handled at or above flash point)	Flash point (F)	Eyes	Skin	Lungs	Goggles	Face Shield	Gloves	Rubber Jacket	Rubber suit & boots	Spot ventilation	Dust mask	Cartridge respirator	Fresh air mask / Helmet	
41-2600-3206-7	Stabilized C8 Acid in Inerts	S		NA	H	H	H	X	X	Neoprene	X	X	X		X		
41-2600-8749-3	Stabilized C8 Acid [Antwerp]	S		NA	H	H	H	X	X	Neoprene	X	X	X		X		
41-2600-0824-2	Stabilized C8 Acid in Inerts [Antwerp]	L		NA	H	H	H	X	X	Neoprene	X	X	X		X		
41-2700-4647-1	Stabilized C8 Acid [Decatur]	S		NA	H	H	H	X	X	Neoprene	X	X	X		X		
11-0000-3179-6	Celite 500 (filter Cell, Diatomaceous)	S		NA	L	L	M			Cloth		X	X	X	X		1
41-2700-3160-6	Crude C8 Inerts	L		NA	H	H	H	X	X	Neoprene	X	X	X		X		
41-2600-6002-9	C8 Salts/Acids Dissolved in water	L		NA	H	H	H			Neoprene		X			X		
41-2700-3210-9	First Pre-Cut from FM-3256	L		NA	H	H	H	X	X	Neoprene	X	X	X		X		
41-2700-3257-0	Inter-cut from FM-3256	S		NA	H	H	H	X	X	Neoprene	X	X	X		X		2
41-2600-8281-7	Fractionated C8 Acid from FM-3256	S		NA	H	H	H	X	X	Neoprene	X	X	X		X		2
41-2700-3256-2	Fractionated C8 Acid - Main Cut	S		NA	H	H	H	X	X	Neoprene	X	X	X		X		2
41-2600-8282-5	Fractionated C8 Acid from FM-3256	S		NA	H	H	H	X	X	Neoprene	X	X	X		X		2
41-2600-4169-8	Post-Cut from FM-3256	S		NA	H	H	II	X	X	Neoprene	X	X	X		X		2
11-0000-3048-3	Sulfuric Acid 93%	L		NA	H	H	H			Neoprene	X	X	X		X		
Sample 1	Fractionated C8 Acid	S		NA	H	H	H	X	X	Neoprene	X	X	X		X		2
Bottoms	Fractionation Bottoms with H2SO4	L		NA	H	H	H	X	X	Neoprene	X	X	X		X		2
11-0000-3009-5	Methanol	L	X	52	M	L	L	X		Butyl		X	X		X		3

- HMIS 0-0-0
- Solid at room temperature. Melts at 120 F. Usually handled as a liquid. Drums may be hot since the material is stored in the hot room.
- Inhalation of high concentrations may cause permanent eye damage. Wear supplied air mask if using large concentrations. All equipment and drums must be grounded and bonded when handling flammable or combustible materials

Special PPE Requirements:

- For all materials charged or drained as part of this run a Cartridge Respirator must be worn with stacked yellow acid gas and hepa cartridges.
- C8 acid must be contained to within the reactor room. The most critical control parameter to ensure containment is the proper use of the PPE Donning and Doffing rooms/area. All PPE must be properly removed in the Doffing room/area in order to

prevent tracking the C8 acid out of the reactor room. Contact the shift supervisor for questions regarding PPE and for the proper use of the PPE Donning and Doffing room/area.

- The above listed PPE requirements are for handling specific compounds and may differ from the general room PPE requirements. If the above PPE differs from the general room PPE then the highest level should be worn.

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