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AMERICAN CYANAMID COMPANY

PIGMENTS DIVISION

PINEY RIVER PLANT

END LIQUOR CONCENTRATION

ACID RECOVERY

OPERATING PROCEDURE

VOLUME XI

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SECTION I

(I)

OPERATING PROCEDURE

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END LIQUOR CONCENTRATION

ACID RECOVERY

I. THE PURPOSE OF CONCENTRATING END LIQUOR IS TO RECOVER AND CONCENTRATE THE SULFURIC ACID CONTAINED IN END LIQUOR. THIS ACID MAY THEN BE REUTILIZED BY THE PLANT.

THE END LIQUOR IS CONCENTRATED IN A VACUUM EVAPORATOR, COOLED, AND THEN FILTERED OVER A ROTARY FILTER TO SEPARATE THE IMPURITIES.

II. STEPWISE OPERATING INSTRUCTIONS

A. EVAPORATOR START-UP

1. CHECK TO BE SURE ALL VALVES ARE CLOSED.
2. FILL SURGE TANK TO WITHIN 10" OF OVERFLOW WITH END LIQUOR.
3. TURN WATER ON RECIRCULATING PUMP GLAND TO 20 - 25 LBS. PRESSURE. START RECIRCULATING PUMP.
4. OPEN DISCHARGE VALVES ON RECIRCULATING LINES TO BE USED (VALVES AT HEAT EXCHANGER AND EVAPORATOR INLET). BE SURE TRANSFER VALVE IS CLOSED.
5. OPEN AIR OPERATED DISCHARGE VALVE ON EVAPORATOR. THIS IS DONE BY TURNING AIR CONTROL TO 15 LBS. PRESSURE ON GAUGE.
6. SLOWLY OPEN SUCTION SIDE VALVE OF RECIRCULATING PUMP.
7. GRADUALLY BUILD UP THE VOLUME OF SLURRY IN THE EVAPORATOR SO THAT THE LIQUOR LEVEL CAN BE SEEN IN THE LOWER SIGHT GLASS. THE VOLUME OF THE SLURRY IN THE SURGE TANK WILL BE INSUFFICIENT TO MAINTAIN A LEVEL IN THE SURGE TANK. THEREFORE, IT IS NECESSARY TO ADD END LIQUOR TO THE SURGE TANK AS THE LEVEL IS BEING BUILT UP IN THE EVAPORATOR. BUILD UP LEVEL IN EVAPORATOR BY GRADUALLY CLOSING THE DISCHARGE (AIR OPERATED) VALVE.

8. WHEN THE LEVEL IN THE EVAPORATOR CAN BE MAINTAINED AND THERE IS 2 FEET OF SLURRY ABOVE PUMP SUCTION, CLOSE END LIQUOR VALVE SO THAT THERE IS APPROXIMATELY 2 - 3 G.P.M. FLOW.

9. START BUILDING VACUUM IN THE EVAPORATOR BY OPENING WATER VALVE TO CONDENSER (OPERATE WITH CONTROLLER ON MANUAL), SO THAT APPROXIMATELY 350 GALS./MIN. (25" OF MANOMETER) IF ONE RECIRC. LINE IS USED, AND 500 GALS./MIN. IF BOTH RECIRC. LINES ARE USED.

   TURN WATER ON INTERCOOLER OF EJECTOR STAGES AND OPEN NO. 1 AND NO. 2 STAGES TO 120 LBS. STEAM PRESSURE ON EACH. NOTE: IF LIQUOR BUILDS UP IN EVAPORATOR DUE TO HIGH VACUUM, TURN OFF EJECTOR STAGES AND OPERATE AT LOW VACUUM UNTIL GRAVITY BUILDS UP AND DESIRED LEVEN IN THE EVAPORATOR CAN BE MAINTAINED.

10. TURN STEAM ON HEAT EXCHANGER VERY SLOWLY. MAINTAIN PRESSURE OF 20 - 40 P.S.I.G., OR AS SPECIFIED. BE SURE TO OPEN VALVES TO BOTH UPPER AND LOWER HEAT EXCHANGERS ON RECIRCULATION LINE BEING USED. DO NOT LET RECIRC. TEMP. EXCEED 105°C.

11. WHEN GRAVITY REACHES 1.770, OR AS SPECIFIED, SLOWLY INCREASE END LIQUOR FEED RATE TO MAINTAIN THIS SPECIFIC GRAVITY. DENSITY RECORDER IS TO BE USED AS SOON AS DESIRED GRAVITY IS REACHED AND TRANSFERRING HAS BEGUN.

12. WHEN SPECIFIC GRAVITY IS WITHIN LIMITS, TURN WATER ON HEAT EXCHANGER, OPEN TRANSFER VALVE AND TRANSFER AS CLOSELY AS POSSIBLE TO THE RATE OF EVAPORATION. THIS IS DONE BY MAINTAINING A CONSTANT LEVEL IN THE SURGE TANK (USE THE GAUGE FOR THIS). BE SURE LEVEL IS ALWAYS 2 FEET ABOVE PUMP SUCTION.

13. SET AUTOMATIC CONTROLLER TO MAINTAIN TAIL LEG WATER TEMPERATURE BETWEEN 90 - 100°F. WHEN VACUUM REACHED 28" Hg. IN SUMMER THE TAIL LEG TEMPERATURE MAY RISE ABOVE 100°F. INCREASING WATER FLOW WILL NOT LOWER THE TEMPERATURE, BUT ONLY FLOOD THE CONDENSER AND CAUSE LOSS OF VACUUM. IN THIS CASE, USE JUST ENOUGH WATER TO HOLD VACUUM DESIRED.

B. FILTER START-UP

1. WHEN FILTER FEED TANK IS WITHIN 30" (A.S.) OF TOP, OPEN VALVES ON SUCTION AND DISCHARGE SIDES OF FILTER FEED PUMP, TURN WATER ON GLAND TO 20 - 25 P.S.I.G., AND START PUMPING FILTER FEED SLURRY TO THE FILTER BOWL.
2. WHEN BOWL IS FULL (TO OVERFLOW LINE) START FILTER AGITATOR AND FILTER DRIVE. TO START DRIVE, THE STRONG FILTRATE PUMP MUST BE STARTED FIRST AS THE TWO ARE INTERLOCKED.
3. SET FILTER DRIVE AT FAST SPEED (No. 8) AND SLOWLY OPEN THE PICK-UP AND DEWATERING VACUUM VALVES. AS SOON AS DRUM IS COVERED WITH CAKE, CHANGE SPEED OF FILTER TO SPEED DESIRED.
4. TURN ON WATER SPRAY SO THAT VACUUM PICKS UP WATER AND DOES NOT DRAIN OFF DRUM INTO FILTER BOWL. APPROXIMATELY 1 G.P.M. IS REQUIRED.
5. OPEN AIR BLOW VALVE.

C. GENERAL INSTRUCTIONS

1. THE OPERATING PANEL BOARD HAS GAGES TO SHOW THE LIQUID LEVEL FOR THE SURGE TANK, THE FILTER FEED TANK, AND THE RECOVERED ACID TANK.
2. THE SURGE TANK SHOULD BE KEPT AT LEAST 2 FT. ABOVE THE PUMP SUCTION (33" A.S.). GAGE READING WILL VARY WITH SPECIFIC GRAVITY BUT SHOULD READ ABOUT 95 WHEN OPERATING AT SPECIFIC GRAVITY OF 1.770.

3. WHEN THE GAGE FOR THE FILTER FEED TANK SHOWS APPROX. 150 THE FILTER SHOULD BE STARTED AND FILTRATION BEGUN. (THERE SHOULD BE ROOM IN RECOVERED ACID (CONCENTRATED) ACID TANK BEFORE STARTING).

4. FEED RATE OF END LIQUOR WILL DEPEND UPON USAGE OF THE RECOVERED ACID AT DIGESTION. AT PRESENT, THE END LIQUOR FEED RATE IS NORMALLY 18 G.P.M. WHILE OPERATING BOTH RECIRCULATING LINES.

5. MAINTAIN A VACUUM OF 27 - 28" HG.

6. THE TEMPERATURE OF THE RECIRCULATED LIQUOR FROM THE POLYBLOC HEAT EXCHANGER SHOULD NOT EXCEED 105 °C. THE TEMPERATURE IS CONTROLLED BY STEAM PRESSURE TO THE HEAT EXCHANGER. NORMAL PRESSURE IS 20 - 40 P.S.I.G.

7. MAINTAIN LEVEL IN THE EVAPORATOR AT THE LOWER SIGHT GLASS.

8. GAGE LEVEL OF 200 INDICATES A FULL RECOVERED ACID TANK AT 1.490-1.500 SPECIFIC GRAVITY.

9. WATER FLOW TO THE BAROMETRIC CONDENSER IS AUTOMATICALLY MAINTAINED BY SETTING DESIRED TEMPERATURE ON THE CONTROLLER. IF CONTROLLER DOES NOT MAINTAIN THE TEMPERATURE, USE BY-PASS TEMPORARILY TO MAINTAIN THE TEMPERATURE (90 - 100°F). THERMOMETER IS LOCATED ON TAIL LEG AT 3RD MOORE LEVEL.

10. THE FILTER FEED AGITATOR IS INTERLOCKED WITH THE FILTER DRUM. THEREFORE, IF THE AGITATOR STOPS FILTRATION STOPS. ALSO, THE FILTER DRUM IS INTERLOCKED WITH THE FILTRATE PUMP. IF THE FILTRATE PUMP STOPS, THE FILTER WILL STOP. THE VACUUM MUST BE TURNED OFF THE FILTER MANUALLY.

11. A HEAT EXCHANGER IS INSTALLED IN THE TRANSFER LINE FROM THE EVAPORATOR TO THE FILTER FEED TANK. THE SLURRY IS COOLED TO 60°C TO PREVENT DAMAGE TO THE FILTER CLOTH. AN ALARM SOUNDS WHEN THIS TEMPERATURE IS EXCEEDED. DISCHARGE WATER FROM THE HEAT EXCHANGER IS USED TO FLUSH THE FILTER CAKE DOWN THE DRAIN.

12. AFTER PUMPING CONCENTRATED ACID TO THE DIGESTORS, BE SURE TO CLOSE VALVES TO THE STORAGE TANK SINCE THE SAME LINE IS USED TO PUMP BIRD FILTRATE TO DIGESTORS.

III. EMERGENCY PROCEDURE

A. IN CASE OF POWER FAILURE, SHUT OFF THE STEAM.

B. CLOSE EVAPORATOR DISCHARGE VALVE (BY AIR OPERATED VALVE).

C. WHEN POWER RETURNS, OPEN THE EVAPORATOR DISCHARGE VALVE, CONTINUE RECIRCULATING, AND PUT STEAM BACK ON HEAT EXCHANGER.

D. START OTHER EQUIPMENT.

IV. PROCEDURE FOR ANNUAL PLANT SHUTDOWN

WITH THE SHUTDOWN OF DIGESTION, THERE WILL BE NO NEED FOR THE PRODUCTION OF RECOVERED ACID AND THE ACID RECOVERY UNIT CAN START SHUTTING DOWN.

IF IT IS PLANNED TO CLEAN THE EVAPORATOR AND TANKS DURING THE SHUTDOWN THE FOLLOWING PROCEDURE IS RECOMMENDED:

1. EVAPORATOR

A. SHUT OFF THE STEAM TO THE HEAT EXCHANGERS.

B. SHUT OFF THE END LIQUOR FEED TO THE EVAPORATOR AND RECIRCULATING PUMPS.

C. TRANSFER THE SLURRY FROM THE SURGE TANK AND AT THE SAME TIME START EMPTYING THE EVAPORATOR BY OPENING THE DISCHARGE VALVE AND ALLOWING SLURRY TO DISCHARGE INTO THE SURGE TANK.

D. TRANSFER UNTIL THE EVAPORATOR HAS BEEN EMPTIED AND THE SURGE TANK HAS BEEN LOWERED TO TOP OF THE PUMP INLET.

E. FILL THE SURGE TANK WITH WATER AND START RECIRCULATING MAINTAINING LEVEL IN SIGHT GLASS. DO NOT USE THE BAROMETRIC CONDENSER OR STEAM EJECTORS.

F. BOIL FOR 4 HOURS OR UNTIL THE SYSTEM APPEARS TO HAVE ALL SOLIDS IN SOLUTION.

G. DRAIN THE SYSTEM AND FILL THE EVAPORATOR AND SURGE TANK WITH END LIQUOR.

2. EVAPORATOR FEED TANK

A. DRAIN SOLIDS FROM BOTTOM OF THE TANK UNTIL CLEAR.

B. FILL TANK AND LEAVE FULL OF END LIQUOR.

3. FILTER FEED TANK

A. CONTINUE FILTERING UNTIL THE TANK IS EMPTY.

B. DRAIN THE TANK AND WASH OUT WITH WATER. LEAVE EMPTY.

4. FILTER

A. WHEN FINISHED FILTERING, WASH DOWN THE FILTER, BOWL, AND GENERAL AREA.

B. REPORT ALL REPAIR WORK TO BE DONE.

IF THE SHUTDOWN IS FOR A SHORT PERIOD AND THE SYSTEM IS NOT TO BE CLEANED, THE FOLLOWING PROCEDURE IS RECOMMENDED.

1. EVAPORATOR

   A. SHUT OFF STEAM TO THE HEAT EXCHANGERS.

   B. SHUT OFF THE END LIQUOR FEED TO THE EVAPORATOR AND RECIRC. PUMPS.

   C. TRANSFER THE SLURRY FROM THE SURGE TANK AND AT THE SAME TIME START EMPTYING THE EVAPORATOR BY OPENING THE DISCHARGE VALVE AND ALLOWING SLURRY TO DISCHARGE INTO THE SURGE TANK.

   D. TRANSFER UNTIL THE EVAPORATOR HAS BEEN EMPTIED AND THE SURGE TANK HAS BEEN LOWERED TO TOP OF PUMP INLET.

   E. FILL THE SURGE TANK WITH END LIQUOR TO WITHIN 2 INCHES OF THE OVERFLOW.

   F. LEAVE EVAPORATOR EMPTY AND VALVE OPEN.

   G. LEAVE AGITATOR IN SURGE TANK RUNNING.

2. EVAPORATOR FEED TANK

   A. DRAIN SOLIDS FROM BOTTOM OF TANK UNTIL CLEAR.

   B. FILL TANK AND LEAVE FULL OF END LIQUOR.

3. FILTER FEED TANK

   A. CONTINUE FILTERING UNTIL THE LEVEL IS ABOUT 1 FOOT ABOVE THE AGITATOR SO SOLIDS WILL REMAIN SUSPENDED.

4. FILTER

   A. WHEN FINISHED FILTERING, WASH DOWN THE FILTER, BOWL, AND GENERAL AREA.

   B. REPORT ALL REPAIR WORK TO BE DONE.

V. SAFETY PROCEDURE

A. WEAR SAFETY GOGGLES AND RUBBER GLOVES WHEN TAKING SPECIFIC GRAVITY, SAMPLING ACID, AND WASHING DOWN FILTER.

B. IF CONTACTED WITH ACID, WASH OFF IMMEDIATELY AND REPORT TO DISPENSARY.

C. REPORT ALL INJURIES REGARDLESS HOW SLIGHT

D. REPORT ACID LEAKS IMMEDIATELY.

STEAM #2

ACID RECOVERY OPERATION
CONCENTRATION SECTION

DATE: 3-25-69

TIME	FED LIQUOR PSID		STEAM		RECIRC TEMP. °F.		LIQUID TEMP. °F.	EVAPORATOR		RECIRC. PUMP AMPS		SURGE TANK LEVEL IN	EVAP. LIQUID SP. GR.	REMARKS
	G.P.M.	SP. GR.	FLOW LBS/HR.	PRESS. PSIG	#1	#2		VAPOR TEMP. °F.	VACUUM IN HG.	#1	#2			
4:00	16	—	4900	09	88	90	49	49	29	11.5	8.5	92	1740	
9	14.5	—	5000	07	87	88	52	50	29	11.5	8.5	92	1745	
10	14.5	—	5000	07	88	89	52	50	29	11.5	8.5	92	1750	
11	14.5	—	4800	07	87	89	53	51	29	11.5	8.5	92	1750	
12	13.5	—	4800	07	87	88	53	51	29	11.5	8.5	92	1750	
1	13.5	—	4800	06	87	89	54	51	28	11.5	8.5	92	1750	
2	13.5	—	4700	06	87	88	54	51	29	11.5	8.5	91	1755	
3	13	—	4200	02	84	85	54	50	29	11.5	8.5	90	1760	
4:00	13	—	4200	02	84	85	54	50	29	11.5	8.5	90	1760	
5	13	—	4200	02	84	84	54	50	29	11.5	8.5	90	1760	
6	13	—	4200	02	84	84	54	50	29	11.5	8.5	90	1760	
7	13	—	4200	02	84	84	54	50	29	11.5	8.5	90	1760	
8	13	—	4200	02	85	84	52	49	29	11.5	8.5	90	1760	
9	13	—	4200	02	85	84	52	49	29	11.5	8.5	90	1760	
10	13	—	4200	02	85	86	52	49	29	11.5	8.5	90	1760	
11	13	—	4200	02	85	86	52	49	29	11.5	8.5	90	1760	
12:00	13	—	4200	03	85	86	52	49	29	11.5	8.5	91	1760	
1	13	—	4400	03	85	86	52	49	29	11.5	8.5	91	1760	
2	13	—	4400	04	85	86	52	49	29	11.5	8.5	91	1760	
3	13	—	4500	04	85	86	52	49	29	11.5	8.5	92	1760	
4	13	—	4500	04	85	86	51	48	29	11.5	8.5	92	1760	
5	13	—	4500	04	85	86	52	48	29	11.5	8.5	93	1760	
6	13	—	4500	04	85	86	51	48	29	11.5	8.5	93	1760	
7	13	—	4400	04	85	87	52	48	29	11.5	8.5	93	1760	

OPERATOR: A. [illegible]

B. [illegible]

C. Mays

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FORM 21-0

2-7

3-25-69

TIME	TANK PRESSURE	WATER SPRAY G.P.M.	AIR BLOW PSIG	FILTRATE SP. GR.	REMARKS	FILTRATE TROUBLE	SP. GR.	[illegible]	[illegible]
00									
Filter started at 12:15 PM & shifted						145"		112"	
33	25	1.0	20			118		120	
33	25	1.0	20			98		136	
33	24	1.0	20			82		154	
00 33	24	1.0	20			39		170	
5 33	24	1.0	20			20		180	
33	24	1.0	20			15		199	
Filter down at 615 full stage								208	
00									

Mays

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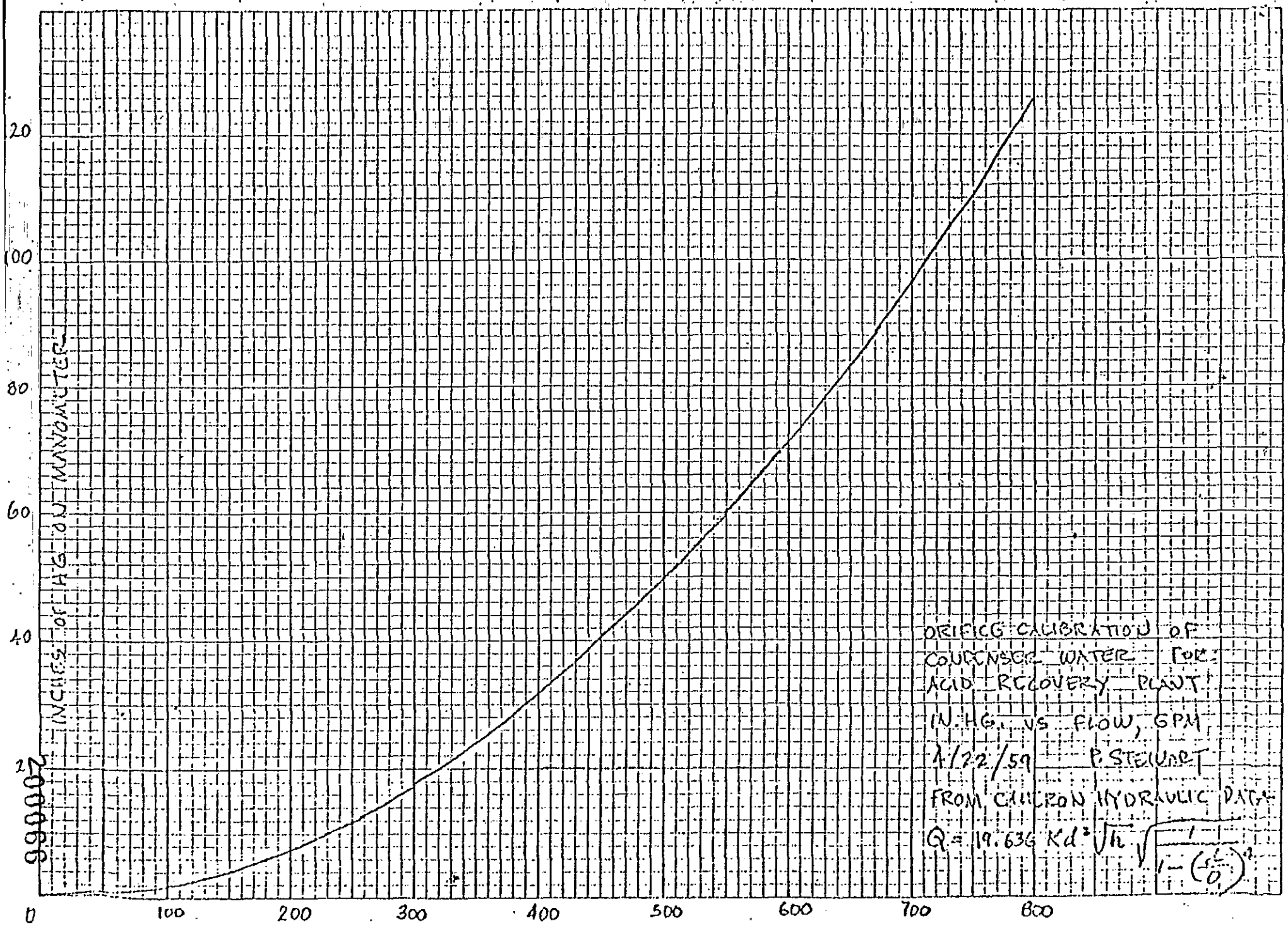
SECTION I

(2)

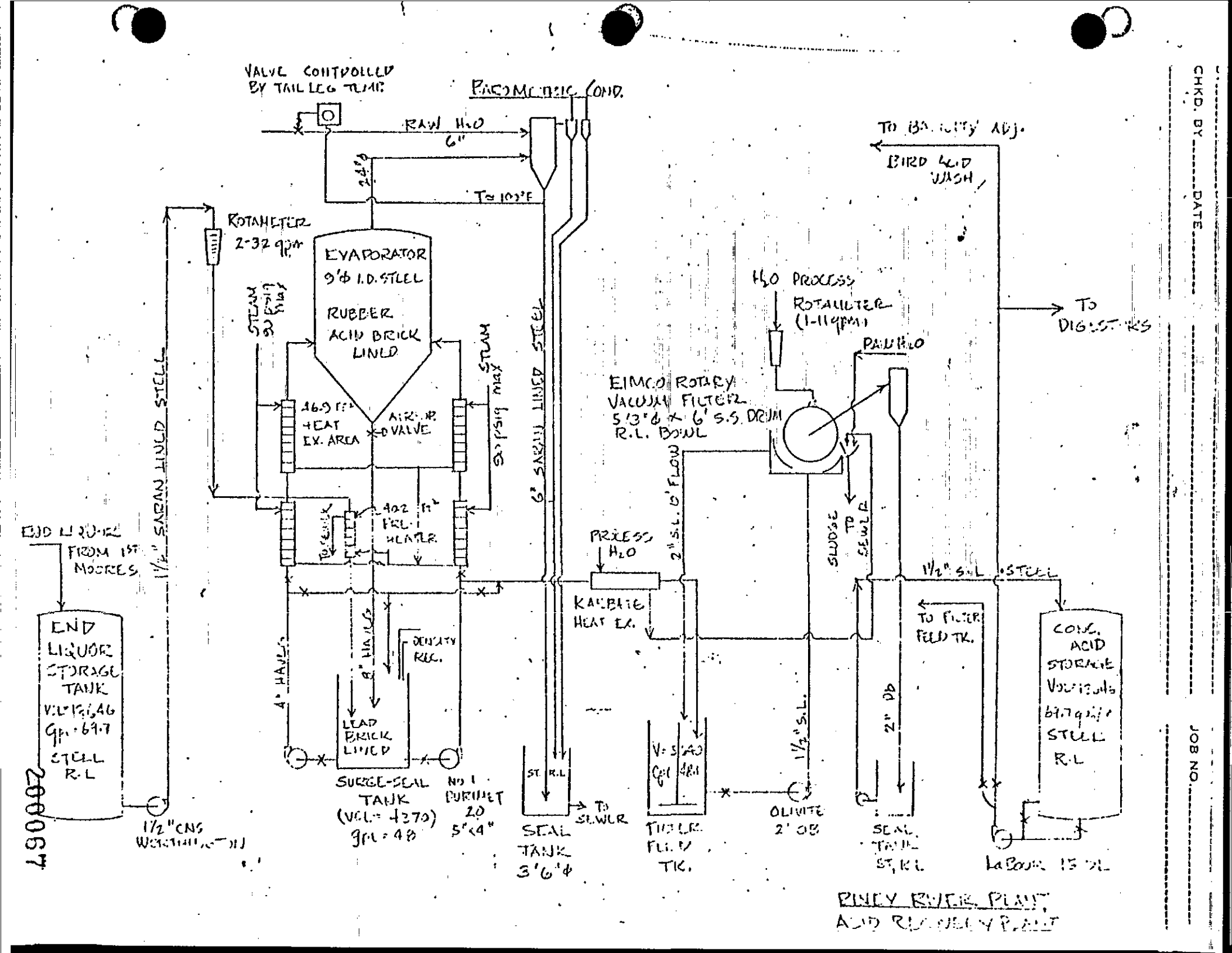
FLOWSHEET

200065

10 X 10 PER INCH
EUGENE DIETZGEN CO.
MADE IN U. S. A.



AR200066



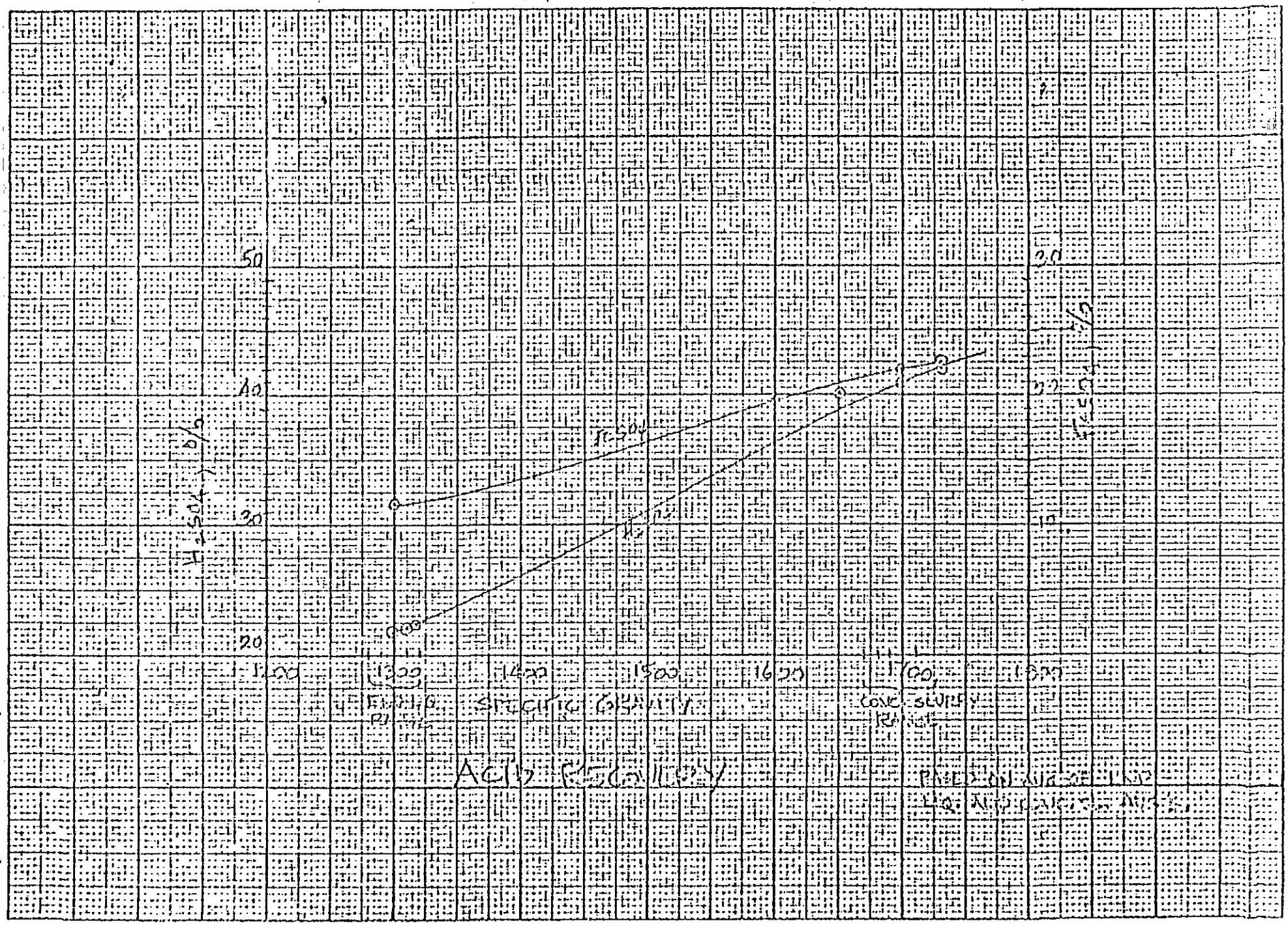
CHKD. BY _____ DATE _____

JOB NO. _____

2000067

NO. 340 -20 DIETZGEN GRAPH PAPER
20 X 20 PER INCH

EUGENE DIETZGEN CO.
MADE IN U. S. A.



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SECTION II

ANALYTICAL PROCEDURE

200069

MARCH 1969

ANALYTICAL INSTRUCTIONS FOR RECOVERED END LIQUOR*

DETERMINATION OF H_2SO_4

PRINCIPLE

TOTAL TITRATABLE SULFATE IS DETERMINED BY TITRATION OF THE HOT SOLUTION WITH NAOH AFTER OXIDATION OF FERROUS IRON WITH H_2O_2 TO INSURE COMPLETE PRECIPITATION AND FLOCCULATION OF THE IRON. THE END POINT IS TAKEN AT pH 7.0 USING BROMTHYANOL BLUE AS INDICATOR. AT MUCH HIGHER OR LOWER pH (E.G. METHYL ORANGE OR PHENOLPHTHALEIN END POINT) THE PRECIPITATE ABSORBS CONSIDERABLE ACID OR ALKALI WITH A CONSEQUENTLY LESS DEFINITE END POINT. THEORETICALLY, BECAUSE OF THE HIGH TEMPERATURE, THE END POINT SHOULD BE TAKEN AT A SLIGHTLY LOWER pH (ABOUT 6.2). SINCE THE pH CHANGE IS QUITE RAPID BETWEEN 6 AND 7, IT HAS NOT BEEN CONSIDERED NECESSARY TO CORRECT FOR THIS.

IRON IS DETERMINED ON A SEPARATE SAMPLE AND THE H_2SO_4 EQUIVALENT OF THE IRON (FOR $FeSO_4$) IS SUBTRACTED FROM THE TOTAL SULFATE CALCULATED AS H_2SO_4. THE DIFFERENCE IS REPORTED AS "ACTIVE ACID" OR BY CONVENTION SIMPLY AS "H_2SO_4".

IT IS UNCERTAIN JUST WHAT COMPOUNDS OF TITANIUM ARE PRESENT IN THESE SOLUTIONS. IN THE FOLLOWING EQUATIONS IT IS ASSUMED TO BE PRESENT AS TITANYL SULFATE.

$$TiOSO_4 + 2\ NaOH + H_2O = Ti(OH)_4 + Na_2SO_4$$

$$H_2SO_4 + NaOH = Na_2SO_4 + 2\ H_2O$$

$$2\ FeSO_4 + H_2SO_4 = Fe_2(SO_4)_3 + 2\ H_2O$$

$$Fe_2(SO_4)_3 + 6\ NaOH = 2\ Fe(OH)_3 + 3\ Na_2SO_4$$

*THESE ANALYTICAL INSTRUCTIONS WERE ADAPTED FROM ANALYTICAL INSTRUCTIONS NO. 3 (VOLUME II OF OPERATING INSTRUCTIONS, PINEY RIVER AND SAVANNAH PLANTS) SHEETS NO. 99, 101 A, 102 AND 104.

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PROCEDURE

PIPETTE 40 ML. SAMPLE INTO A 200 ML VOLUMETRIC FLASK. DILUTE, WITH SHAKING, TO THE MARK. MIX BY INVERTING AND SHAKING.

PIPETTE A 10 ML. PORTION OF THE DILUTED LIQUOR INTO A 400 ML. BEAKER. DILUTE TO APPROXIMATELY 200 ML. WITH BOILING WATER AND ADD 5 ML. OF 6% H_2O_2. BOIL 10 MINUTES. ADD 5 ML. OF 0.04% BROMTHYANOL BLUE SOLUTION AND TITRATE IMMEDIATELY WITH 0.51 N NAOH TO MATCH PH 7.0 STANDARD (LAMOTTE STANDARD). ADD TWO DROPS EXCESS NAOH. HEAT TO BOILING AND AGAIN TITRATE WITH 0.51 N NAOH TO JUST MATCH PH 7.0 STANDARD. IN BOTH TITRATIONS IT IS NECESSARY TO ALLOW THE PRECIPITATE TO SETTLE IN ORDER TO OBSERVE THE COLOR OF THE CLEAR SUPERNATANT LIQUID. RECORD TOTAL TITRATION.

IRON IS TITRATED WITH $Ti_2(SO_4)_3$ IN ANOTHER 10 ML. ALIQUOT OF THE SAME DILUTED SAMPLE. THIS PROCEDURE IS DESCRIBED BELOW UNDER THE TITLE "DETERMINATION OF $FeSO_4$".

VOLUME OF SAMPLE IN PORTION TITRATED = $\frac{10}{200} \times 40 = 2$ ML.

G.P.L TOTAL H_2SO_4 = $\frac{\text{NAOH TITRATION} \times 0.025 \times 1.000}{2}$

= NAOH TITRATION x 12.5

G.P.L H_2SO_4 COMBINED WITH FE =

$\frac{Ti_2(SO_4)_3 \text{ TITRATION} \times H_2SO_4 \text{ FACTOR OF } Ti_2(SO_4)_3 \times 1.000}{2}$

= $Ti_2(SO_4)_3$ TITRATION (H_2SO_4 FACTOR OF $Ti_2(SO_4)_3$ x 500)

(THE FACTOR INDICATED IN BRACKETS WILL BE POSTED ON THE TAG).

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IN THE ORIGINAL RECOVERED END LIQUOR SAMPLE ALL THE IRON IS PRESENT AS $FeSO_4$. THEREFORE,

$$\text{THE } H_2SO_4 \text{ FACTOR ON THE } Ti_2(SO_4)_3 = FeSO_4 \text{ FACTOR} \times \frac{\text{MOL. WT. } H_2SO_4}{\text{MOL. WT. } FeSO_4}$$

$$\text{THE } H_2SO_4 \text{ FACTOR ON THE } Ti_2(SO_4)_2 = FeSO_4 \text{ FACTOR} \times 0.646$$

G.P.L. "ACTIVE ACID" = G.P.L. TOTAL H_2SO_4 - G.P.L. H_2SO_4 COMBINED WITH FE

IF IT IS NECESSARY TO REPORT RESULTS AS % ACTIVE ACID, DETERMINE SPECIFIC GRAVITY BY HYDROMETER. BECAUSE OF THE DEEP COLOR OF THE RECOVERED END LIQUOR, THE TOP OF THE MENISCUS IS READ. THE DEGREE OF ACCURACY REQUIRED DOES NOT JUSTIFY APPLYING A MENISCUS CORRECTION.

$$\%\ \text{ACTIVE ACID} = \text{G.P.L. } \frac{\text{ACTIVE ACID} \times 100}{\text{SP. GR. ACTIVE ACID}} \times 100$$

DETERMINATION OF $FeSO_4$

PRINCIPLE

IRON IS DETERMINED BY OXIDATION WITH $KMnO_4$ AND TITRATION WITH $Ti_2(SO_4)_3$ USING NH_4SCN AS INDICATOR. NH_4SCN REDUCES THE SLIGHT EXCESS OF $KMnO_4$. A LARGE EXCESS OF $KMnO_4$ SHOULD BE AVOIDED AS IT MAY CAUSE HIGH RESULTS.

$$5\ Fe^{++} + MnO_4^- + 8\ H^+ \longrightarrow 5\ Fe^{+++} + Mn^{++} + 4\ H_2O$$

$$Fe^{+++} + Ti^{+++} \longrightarrow Fe^{++} + Ti^{++++}$$

PROCEDURE

USE SAME DILUTED LIQUOR AS IN "DETERMINATION OF H_2SO_4". PIPETTE A 10 ML. PORTION OF THE DILUTED LIQUOR INTO A 600 ML. BEAKER, ADD 1000 ML. OF

1 / 5 H_2SO_4 AND DILUTE TO APPROXIMATELY 300 ML. ADD APPROXIMATELY 0.5 N $KMnO_4$ FROM A BURETTE TO FIRST PERMANENT PINK. AVOID MORE THAN ONE OR TWO DROPS EXCESS. ADD 10 ML OF NH_4SCN (100 G.P.L.) SOLUTION AND TITRATE WITH STANDARDIZED APPROXIMATELY 0.1 N $Ti_2(SO_4)_3$ TO DISAPPEARANCE OF RED FERRIC THIOCYANATE COLOR. FINAL COLOR IS YELLOW DUE TO PRESENCE OF TITANIUM THIOCYANATE.

VOLUME OF SAMPLE IN PORTION TITRATED = 10/200 x 40 = 2 ML.

$$\text{G.P.L. } FeSO_4 = \frac{\text{TITRATION} \times FeSO_4 \text{ FACTOR OF } Ti_2(SO_4)}{2}$$

$$= \text{TITRATION} \times (FeSO_4 \text{ FACTOR OF } Ti_2(SO_4)_3 \times 500)$$

(THE FACTOR INDICATED IN BRACKETS WILL BE POSTED ON THE TAG).

SECTION III

(1)

EQUIPMENT LIST

END LIQUOR CONCENTRATION

ACID RECOVERY

EVAPORATOR, C. T. 53,287 -

USED TO CONCENTRATE END LIQUOR FROM 1ST MOORES.

CAPACITY: 39.3 GAL./IN., CONE VOL. = 381 T.V. = 8,740 GAL.

SIZE: 9' 0" I.D. STEEL (8' 11 5/8" I.D.) HEIGHT: 17' 8 5/8" ST. SECT., 6' 10 1/4" CONE

MATERIAL: 1/2" STEEL

LINING: 1/4" RUBBER, 1/8" NON-BURN ASBESTOS, 4 1/2" ACID BRICK

OUTLETS:
- TWO (2) - 5" DIA. SIGHT GLASS NOZZLES
- ONE (1) - 3" DIA. INSTRUMENT CONN. NOZZLE
- ONE (1) - 3" DIA. NOZZLE VACUUM BREAKER
- TWO (2) - 6" DIA. INLET NOZZLES
- TWO (2) - 6" DIA. INLET NOZZLE SPARES
- ONE (1) - 12" DIA. OUTLET @ CONE
- ONE (1) - 24" DIA. VAPOR OUTLET
- ONE (1) - 16" DIA. MANHOLE
- ONE (1) - 24" DIA. MANHOLE

INSTRUMENTATION: EQUIPPED WITH VACUUM GAGE AT VAPOR OUTLET, TEMPERATURE RECORDER FOR TOP AND BOTTOM OF EVAPORATOR, 0 - 250°F. THERMOMETER ON TAIL LEG OF CONDENSER

END LIQUOR STORAGE TANK, C. T. 53,318

USED TO STORE END LIQUOR AND ALSO AS EVAPORATOR FEED TANK.

CAPACITY: 69.7 GPI., CONE VOL. = 696 T.V. = 12,646

SIZE: 11' 11 1/4" I.D. HEIGHT: 14' 3 5/8" ST. SECT. TOTAL HEIGHT - 17' 7 7/8"

MATERIAL: 1/4" PLATE STEEL

LINING: 3/16" RUBBER

OUTLETS:
- ONE (1) - 6" DIA. NOZZLE FOR HEATER INSTALLATION
- ONE (1) - 4" DIA. NOZZLE DECANTING
- ONE (1) - 4" NOZZLE THERMOWELL
- ONE (1) - 4" DIA. NOZZLE DRAIN OUTLET
- ONE (1) - 6" DIA. NOZZLE TANK OVERFLOW
- ONE (1) - 4" DIA. TANK FILL NOZZLE
- ONE (1) - 6" DIA. NOZZLE HIGH LEVEL ALARM
- ONE (1) - 24" DIA. (O.D.) MANHOLE
- ONE (1) - 4" DIA. NOZZLE SPARE

INSTRUMENTATION: HIGH LEVEL ALARM

RECOVERED ACID (CONCENTRATED) STORAGE TANK, C.T. 53,317

SAME AS C.T. 53,318, EXCEPT EQUIPPED WITH KING LEVEL GAUGE, AND HAS NO HIGH LEVEL ALARM.

FILTER FEED TANK, C.T. 51,397

USED FOR FEED TANK FOR EIMCO FILTER

CAPACITY: 48.1 GAL./IN., VOL. - 5,640 GALS.
SIZE: 9' 11" I.D. HEIGHT: 117"
MATERIAL: 1/4" STEEL PLATE
LINING: 12# SHEET LEAD, 3" BRICK LINED BOTTOM
COVER: WOOD, TAR PAPER COVERED
OUTLETS: ONE (1) - 2 1/2" TANK OUTLET NOZZLE
AGITATION: ONE BLADE HAVEG COVERED AGITATOR, NETTCO DRIVE: C.T. 50,167, RATIO 54:1, 16 R.P.M.
INSTRUMENTATION: KING LEVEL GAUGE INSTALLED ONE FT. FROM BOTTOM OF TANK.

EIMCO ROTARY FILTER, C.T. 53,322

USED TO FILTER $FeSO_4$ FROM CONCENTRATED END LIQUOR.

SIZE: 5' 3" ø x 6', 100 FT.^{2} FILTER AREA
CONSTRUCTION: DRUM OF STAINLESS STEEL, WITH PLASTIC GRIDS, DOCTOR BLADE STAINLESS
DRIVE: REEVES VARI-SPEED MOTOR, C.T. 53,323, 1725 R.P.M. 3 PHASE 60 CY., SERIAL M10804 JR
INSTRUMENTATION: EQUIPPED WITH VACUUM GAGES, AND AIR PRESS. GAGE.

FILTRATE SEAL TANK, C.T. 53,339

USED TO MAINTAIN VACUUM WHILE WITHDRAWING FILTRATE FROM FILTER

CAPACITY: 4.275 GAL./IN. VOL. - 256
SIZE: 2' 11 1/2" I.D. HEIGHT = 5'.0"
MATERIAL: 1/4" MILD STEEL
LINING: 3/16" NEOPRENE
OUTLETS ONE (1) - 2 1/2" DIA. NOZZLE OVERFLOW
ONE (1) - 2" DIA. NOZZLE DRAIN
ONE (1) - 2 1/2" DIA. NOZZLE PUMP SUCTION
INSTRUMENTATION: HIGH LEVEL ALARM

FILTRATE RECEIVER TANK, C.T. 53,335

USED TO SEPARATE FILTRATE FROM VACUUM

SIZE: 24" O.D., HEIGHT: 4' 0"
MATERIAL: STEEL
LINING: 3/16" RUBBER
OUTLETS: ONE (1) - 4" BOTTOM OUTLET
ONE (1) - 4" SIDE NOZZLE
ONE (1) - 6" TOP OUTLET

SURGE AND SEAL TANK, C.T. 50,045

USED TO KEEP VACUUM SEAL WHILE RECIRCULATING END LIQUOR.

CAPACITY: 48.0 GAL./IN.; 81" OVERFLOW VOL. = 3,888 GALS.
SIZE: 9' 10 3/4" I.D. HEIGHT = 91" (TO O.F. = 81")
MATERIAL: 1/4" STEEL PLATE
LINING: 1/16" ASBESTOS, 12# SHEET LEAD, 4 1/2" DURO ACID BRICK
COVER: WOOD (PINE) WITH TAR PAPER LINED
OUTLETS: THREE (3) - 6" DIA. NOZZLE OPENINGS FOR PUMPS
ONE (1) - 6" NOZZLE OVERFLOW
ONE (1) - 6" DRAIN NOZZLE

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(CONT'D.)

AGITATION: NETTCO DRIVE 30.1 RATIO LEAD COVERED TURBO AGITATOR C.T. 50,706. MOTOR, C.T. 50,623 5 H.P., 870 R.P.M. MODEL SK 326 A 992
INSTRUMENTATION: KING LEVEL GAUGE

RECIRCULATION PUMP NO. 1, C.T. 53,219
USED TO RECIRCULATE SLURRY THROUGH HEAT EXCHANGERS AND EVAPORATOR.
TYPE: DURIRON 4 X 3
DRIVE: DIRECT COUPLED
MOTOR: C.T. 51,912
MAKE: STAR ELEC. CO., 15 H.P., 1760 R.P.M. TYPE NL 364

EVAPORATOR FEED PUMP, C.T. 52,596
USED TO PUMP END LIQUOR FROM STORAGE TANK THROUGH ROTAMETER AND PREHEATER TO SURGE TANK.
TYPE: WORTHINGTON 1 1/2 CNG 74 A, SER, A 9052 8" IMPELLER
DRIVE: DIRECT
MOTOR: C.T. 52021
MAKE: WAGNER ELEC. CO. 5 H.P., 1750 R.P.M. TYPE CP-1 3 PH

FILTER FEED PUMP, C.T. 53725
USED TO PUMP CONCENTRATED SLURRY TO EIMCO FILTER
TYPE: DURCO PUMP 2"
DRIVE: DIRECT
MOTOR: C.T. 51,384
MAKE: WESTINGHOUSE 3 H.P., 1150 R.P.M. TYPE CS STYLE 1071176

STRONG FILTRATE PUMP, C.T. 50,742
USED TO PUMP RECOVERED ACID TO STORAGE TANK
TYPE: LABOUR 15 DS 2 1/2"
DRIVE: DIRECT
MOTOR: C.T. 50,886
MAKE: GENERAL ELECTRIC "K" 5 H.P., 900 R.P.M., FRAME 326 MODEL 5 K 326A992

RECOVERED ACID (ACID STORAGE), PUMP C.T. 52,537
USED TO PUMP ACID TO DIGESTORS
TYPE: LABOUR 15 DL 1-ML SERIAL 9330 2"
DRIVE: DIRECT
MOTOR: C.T. 50,080
TYPE: GENERAL ELECTRIC "K" 10 H.P., 1760 R.P.M. SERIAL LH 1184, MODEL 5 K 326 B 997

HEAT EXCHANGER, C.T. 53,282; C.T. 53,281; C.T. 53,312: C.T. 53,311
USED TO HEAT END LIQUOR PRIOR TO ENTERING EVAPORATOR
SIZE: 13" DIA. GRAPHITE BLOCKS, EACH HEAT EXCHANGER 46.9 FT.^{2} (HEAT TRANSFER AREA)
TYPE: POLYBLOC (GRAPHITE)
MFG.: CARBONE CORP., BOONTON, NEW JERSEY

KARBATE HEAT EXCHANGER, C.T. 53,345
SIZE: 87 FT.^{2} HEAT EXCHANGER AREA
SPEC.: SERIES 190 - 1/4 SER. H. E. - 948
TEST PRESS. = 112
MAX. WKG. PRESS HYDRO 75 P.S.I.G.
MAX. WKG. PRESS STEAM 50 P.S.I.G.
MFG. NATIONAL CARBON CO., CLEVELAND, OHIO

END LIQUOR ROTOMETER
USED TO DETERMINE FLOW OF END LIQUOR TO EVAPORATOR
RANGE: 2 1/2 - 32 1/2 G.P.M. SP. GR. 1.280
MAKE: FISHER PORTER 570 4A 2128 A1
MFG. FISHER PORTER

WATER SPRAY ROTOMETER
USED TO DETERMINE AMOUNT OF WATER SPRAY ON FILTER
STD.: R-10M-25-3
RANGE: 1.0 - 10.8 G.P.M. SP. GR. 1.0
MFG. BROOKS ROTOMETER CO., LANSDALE, PA.

BAROMETRIC CONDENSER, C.T. 53,288
USED TO CREATE VACUUM IN EVAPORATOR.
EQUIPPED WITH PRIMARY STAGE, INTERCOOLER, SECONDARY STAGE STEAM VACUUM BOOSTER SYSTEM.

CONDENSER SEAL TANK, C.T. 53,337
USED TO SEAL VACUUM WHILE DISCHARGING WATER OF CONDENSER.
CAPACITY: 6.0 GAL./IN. VOL. - 540 GALS.
SIZE: 7' 6" HEIGHT; 3' 6" I.D.
MATERIAL: 1/4" PLATE STEEL
LINING: 3/16" NEOPRENE
OUTLETS: ONE (1) 8" DIA. OVERFLOW NOZZLE
ONE *1) 4" DIA. DRAIN NOZZLE

RECIRCULATION PUMP NO. 2, C.T. 53230
USED TO RECIRCULATE SLURRY THROUGH HEAT EXCHANGERS AND EVAPORATOR.
TYPE: DUREMET 20, 4 x 3", IMPELLER 12 1/4" DIA.
DRIVE: DIRECT COUPLED
MOTOR: C.T. 51,341
MAKE: GENERAL ELECTRIC "K", 15 H.P., 1165 R.P.M., 3 H.P.
SER. 6097912

END LIQUOR CONCENTRATION
ACID RECOVERY

MARCH 1969

TYPICAL CHEMICAL ANALYSIS

END LIQUOR:	H_2SO_4	=	21.8%
	$FeSO_4$	=	11.6%
CONCENTRATED SLURRY:	H_2SO_4	=	44.0
	$FeSO_4$	=	23.0
FILTERED SLURRY:	H_2SO_4	=	57.0
	$FeSO_4$	=	1.0
FILTER CAKE:	H_2SO_4	=	18%
	$FeSO_4$	=	54%

TYPICAL PHYSICAL PROPERTIES

END LIQUOR:	SP. GRAVITY	=	1.300
	VISCOSITY	=	5.5 CP
	TEMP.	=	45°C.
	SOLIDS	=	1%
CONC. SLURRY:	SP. GRAVITY	=	1.700
	VISCOSITY	=	16.8 CP
	TEMP.	=	70°C.
	SOLIDS	=	20%
FILTERED ACID:	SP. GRAVITY	=	1.475
	VISCOSITY	=	7.5 CP
	TEMP.	=	35°C.
	SOLIDS	=	1 - 3%

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