ORIENTATION PROGRAM

PROCESS ENGINEERING DEPARTMENT

VOLUME II

1982

Process Engineering Department
Ponca City, Oklahoma

ORIENTATION PROGRAM

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VOLUME II

1982

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Conoco Chemicals

ORIENTATION PROGRAM
PROCESS ENGINEERING DEPARTMENT
CHEMICALS DIVISION
VOLUME II

SEPTEMBER 1982

NOTICE

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THE MANUAL IS TO BE RETURNED TO THE PROCESS ENGINEERING DEPARTMENT, PONCA CITY, OKLAHOMA, WHEN IT IS NO LONGER NEEDED BY THE RECIPIENT IN PERFORMANCE OF HIS/HER DUTIES.

PED ORIENTATION PROGRAM

CONOCO CHEMICALS COMPANY

Conoco Chemicals Company's business efforts originated in the manufacture of detergent feedstocks. This continues to be a major part of the business with plants producing biodegradable "NALKYLENE" alkylate at Baltimore, Maryland; surfactant-range "ALFOL" alcohols and ethoxylates at Lake Charles, Louisiana; and sulfonates and sulfates at Hammond, Indiana. Major additions to business included PVC resins and compounds, ethylene, and vinyl chloride monomer.

In October 1980, the Chocolate Bayou Joint Venture with Monsanto was successfully started up. This added approximately four billion pounds per year of olefins and aromatics petrochemicals capacity to the Conoco Chemicals system. In August 1981, as a result of the merger between Conoco and Du Pont, Conoco and Monsanto were required, by the Justice Department, to dissolve the joint venture. Conoco purchased Monsanto's half interest of the venture and now controls the entire production capacity (approximately eight billion pounds per year total petrochemicals at Chocolate Bayou).

A further major business area is the production of carbon black for tire manufacturers. This is operated as a separate company whose president reports to the Chief Operating Officer of Conoco Chemicals. This subsidiary operates four plants in the United States and eight other plants internationally.

Headquarters operations for Conoco Chemicals are located in Houston, Texas. These include specialized support groups for supply and transportation, marketing, product management, project development, business research, and environmental activities. Corporate service divisions also provide specialists in purchasing, financial accounting, recruiting, etc, to cope with problems unique to the chemicals industry.

Conoco Chemicals have branch sales offices across the United States, Europe, South America, and Asia. Europe and Communist block countries are serviced primarily by the office in Brussels, Belgium. Japan, China, and Southeast Asia are serviced by the sales office in Tokyo, Japan. South American sales are handled by an office in Sao Paulo, Brazil.

Conoco Chemicals have shipping terminals in Brazil and Belgium. In addition, they have a portable terminal in the form of an ocean-going tanker which can be anchored at customer receiving areas. This tanker has served as a temporary or supplementary terminal in England, Norway, and Belgium. In addition to these facilities, Conoco Chemicals also use those of affiliate companies in Japan, Spain, and Germany to solve temporary logistics problems created by periodic supply/demand imbalances throughout the world.

A summary of Conoco Chemicals facilities and products is shown in Table I attached.

PED ORIENTATION PROGRAM

TABLE I

CONOCO CHEMICALS FACILITIES

Location	Products	Capacity, MM Lbs/Yr	Ownership Status
United States			
Lake Charles, Louisiana	Ethylene	640	Wholly Owned
	VLM	670	Wholly-Owned
	LAB	150	Wholly-Owned (1982 SU)
	Linear Alcohols	235	Wholly-Owned
	Alcohol Ethoxylates	70	Wholly-Owned
	n-Paraffins	330	Wholly-Owned
	Methyl Chloride	100	Wholly-Owned
	Alumina	40	Wholly-Owned
	Alum	320	Wholly-Owned
	EPA Solvent		Wholly-Owned
	CDR IM	60	Wholly-Owned
Matagorda, Texas	High Density Polyethylene	425	Wholly-Owned (1983 SU)
Baltimore, Maryland	Linear Alkylbenzene	220	Wholly-Owned
	Hydrotropes	13	Wholly-Owned
	Muriatic Acid	135	Wholly-Owned
	Aluminum Chloride Liquor	12	Wholly-Owned
Newark, New Jersey	Cresylics Acids	50	Wholly-Owned
Hammond, Indiana	Sulfonic Acid)		Wholly-Owned
	Sulfonates)	70	Wholly-Owned
	Ether Sulfates)		Wholly-Owned
Aberdeen, Mississippi	PVC Resins	465	Wholly-Owned
	PVC Compounds	50	Wholly-Owned
	Plasticizers	25	Wholly-Owned
Oklahoma City, Oklahoma	PVC Resins	275	Wholly Owned
Continental Carbon	Carbon Black		Wholly-Owned
Sunray, Texas)		430	
Ponca City, Oklahoma)			
Baker sfield, California)			
Phoenix City, Alabama)			
Lake Charles, Texas	Ethylene	1,500	Conoco, 50%; Monsanto, 50%
	Total Petrochemicals	8,000	Wholly-Owned after 8/21/81
Europe			
Condea			
Brunsbuttel, West Germany	Linear Alcohols	165	50% Each Conoco and Texaco
	Alumina	25	
	Natural Alcohols	66	
Petresa			
Algeciras, Spain	n-Paraffins	265	50% Each Conoco and Cepsa
	Linear Alkyl Benzene	330	
Continental Carbon	Carbon Black		
Holland		75	Conoco, International Synthetic Rubber
Rotterdam, Holland		110	Conoco, Cities Service
Algeciras, Spain		40	Conoco, Ashland, Cepsa
Bordeaux, France		135	Conoco, Phillips
Trecate, Italy		90	Conoco, Cities Service
Nissan Conoco Oita, Japan	Linear Alkylbenzene	85	50% Each Conoco and Nissan Chemical Company
Nippon Aluminum Alkyls Sakai, Japan	Aluminum Alkyls Aluminum Alkyl Chlorides	5	75% Conoco, 75% Mitsui Toatsu
Continental Carbon Yokaichiba, Japan	Carbon Black	100	Conoco, Mitsui
Sydney, Australia	Carbon Black	45	Wholly-Owned
Inchon, Korea	Carbon Black	30	Conoco-Bando Trading Company
South America			
Petroquimica Argentina, S.A. Rosario, Argentina	Synthetic Rubber	82	25% Each Conoco, Witco, Uniroyal, Cities Service
	Aromatics and Naphtha	400	

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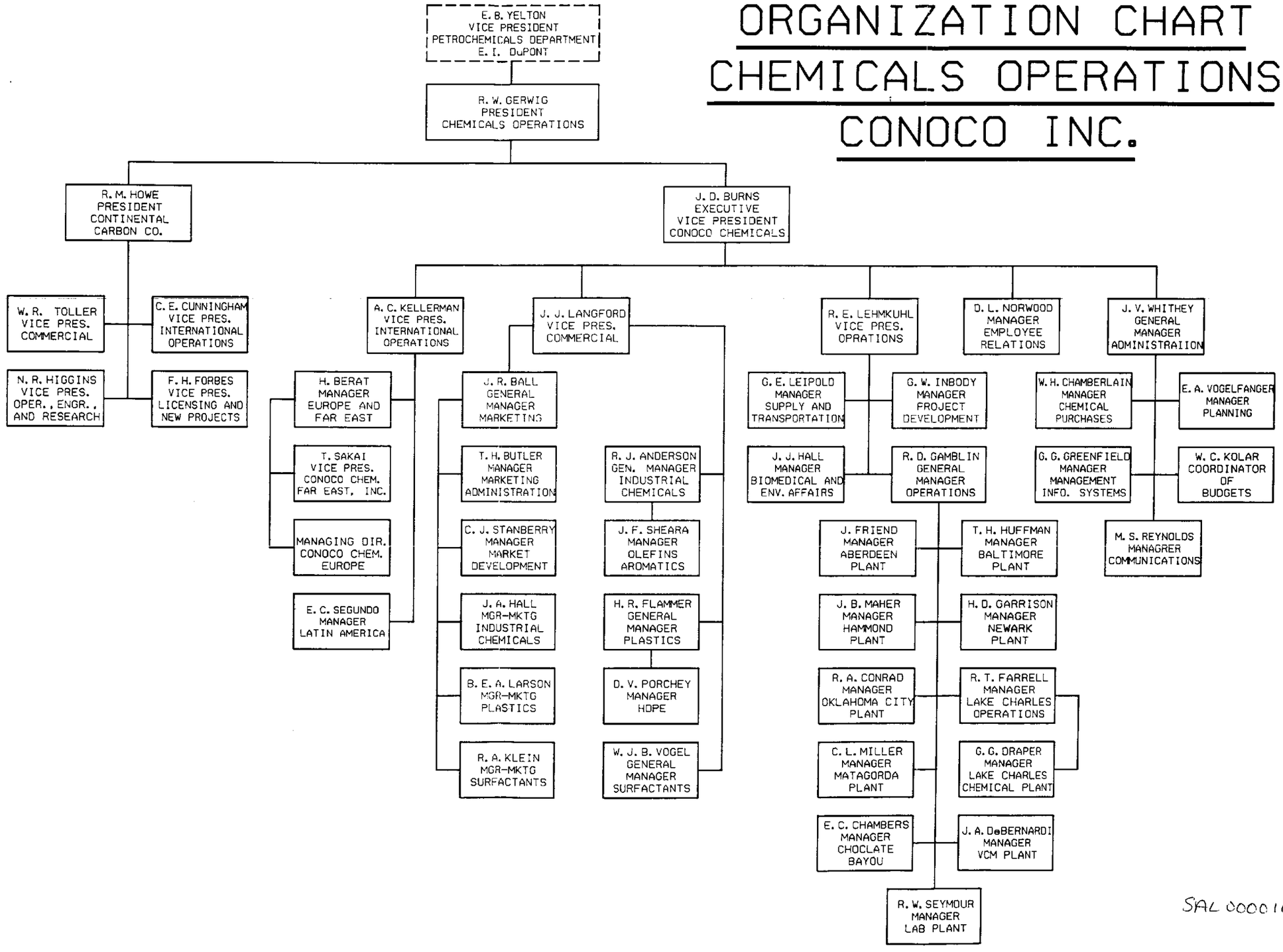
ORGANIZATION CHART
CHEMICALS OPERATIONS
CONOCO INC.



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CT. 56C

LCCP or Lake Charles
Chemical Plant

LAKE CHARLES CHEMICAL PLANT

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ORGANIZATION CHART — LAKE CHARLES CHEMICAL PLANT

MANAGER
LAKE CHARLES OPERATIONS
CONOCO CHEMICALS
R.T. FERRELL

- PLANT MANAGER — G.G. DRAPER
  - SR. OPERATIONS ENGINEER — G.W. DUCHMANN
  - TRAINING SUPERVISOR — P.C. HART
  - OPERATIONS SUPERINTENDENT "ALFOL" ALCOHOL, ALUMINA — L.R. BAUER
    - OPERATIONS ENGINEER "ALFOL" ALCOHOL, ALUMINA
    - OPERATIONS SUPERVISOR "ALFOL" 100/425 — W.O. FULTS — OPERATORS 32 — TRAINEES 6
    - OPERATIONS SUPERVISOR "ALFOL" 550/600, P/L — H.R. CORLEY — OPERATORS 16 — TRAINEES 4
    - PUMPING & LOADING SUPERVISOR — OPEN — PUMPER/LOADERS 7.6 — TRAINEES 1.4
    - SHIFT SUPERVISOR — C.J. BOUTTE, J.A. HARDEY, D.J. BARROW, E.C. GUILLORY, OPEN
  - OPERATIONS SUPERINTENDENT NPE/SOLV, MeCL/ETO, CDR — D.W. FRIESNER
    - OPERATIONS SUPERVISOR NPE/SOLV., MeCL/ETO, CDR — L. LaCOMPTE — OPERATORS 27 — TRAINEES 6
    - OPERATIONS ENGINEER NPE/SOLV., MeCL/ETO, CDR — R.J. TUTTLE
  - OPERATIONS SUPERINTENDENT ETHYLENE UNIT — P.D. CARRICO
    - OPERATIONS ENGINEER ETHYLENE AND ETHYLENE OFFSITES — W.P. SWAN
    - OPERATIONS SUPERVISOR ETHYLENE UNIT — R.L. PATRICK — OPERATORS 24 — TRAINEES 7
    - OPERATIONS SUPERVISOR ETHYLENE OFFSITES — J.P. STOKES — OPERATORS 12 — TRAINEES 3
    - SHIFT SUPERVISOR — D.G. LEDOUX, L.M. LIVELY, M.P. MARTIN, D.C. WRIGHT, J.C. LEWING
  - CHIEF MECHANICAL ENGINEER — H.C. TANNER
    - SENIOR INSTR/ELEC. ENGINEER — F. LEGER
    - INSTR/ELEC. ENGINEER — J.H. VIATOR, R.E. POLK
    - SR. MECHANICAL ENGINEER — OPEN
    - MECHANICAL ENGINEERS — W.P. BROWN, G.D. OSTERHOF, J.M. TILLMAN, D.R. BEATTIE, R.J. KING, B.E. HOLLINGSHEAD, D.N. WAYGOOD, W.E. WHITSON
    - CONTRACTS SUPERVISOR — B.R. FRANKS
    - DRAFTSMAN ENGINEER — M.J. BROUSSARD
    - DRAFTSMAN — F. FAULKNER
    - CED CONSTRUCTION ENGINEER — G. HEGENER
    - CED MECHANICAL ENGINEER — M. INGLE
  - MECHANICAL SUPERINTENDENT — S.J. VINCENT
    - MAINTENANCE SUPT. — H.J. MONTGOMERY
      - AREA MAINT. SUPERVISOR AREA "A" — W. YOUNG
      - AREA MAINT. SUPERVISOR AREA "B" — R. WALKER
      - AREA MAINT. SUPERVISOR AREA "M" — W. HALL
      - AREA MAINT. SUPERVISOR AREA "U" — V.A. TRAMONTE
      - CRAFT SUPERVISOR — A.J. BOUILLION
        - OUTSIDE MACHINIST 13 — APPRENTICES 2 — TOOL ROOM MECH. 1
      - CRAFT SUPERVISOR-PF/W — W.E. McMANEMIN
        - PIPEFITTER 14 — APPRENTICES 3 — WELDERS 7 — APPRENTICES 3
      - AREA MAINT. SUPERVISOR — C.M. BARROW
        - CARPENTERS 3 — APPRENTICES 1 — PAINTER/INS. 4 — APPRENTICES 2 — UTILITY HELPERS 8
      - AREA MAINT. SUPERVISOR — J.R. RYDER
        - EQUIPMENT OPR. 5 — TRAINEES 2
    - MAINTENANCE SUPT. INSTRUMENT/ELECTRICAL — W.W. CRAFT
      - SYSTEMS MAINT. SUPERVISOR ELECTRICIANS — OPEN
        - ELECTRICIANS 5 — APPRENTICES 2
      - SYSTEMS MAINT. SUPERVISOR INSTRUMENT TECHNICIANS — C.G. TAYLOR
        - INST. TECHNICIANS 21 — APPRENTICES 3
      - SYSTEMS MAINT. SUPERVISOR GENERAL — D.R. BURK
      - SR. INSPECTION ENGINEER — K.J. BROWN
      - SR. MECHANICAL INSPECTOR — D.L. CHAMBERLAIN
      - SR. ROTATING EQUIP. SPECIALIST — N. SYMKOWICZ
      - SR. PLANNING SUPERVISOR — M.J. ANDREWS
      - MAINTENANCE PLANNERS — O.R. ESCOUBAS, A.G. SCHWARZER
  - TRAINEES 25.2
- DIRECTOR OF EMPLOYEE RELATIONS — J.D. MITCHELL
  - EMPLOYEE RELATIONS REPRESENTATIVE — J.E. BEALL
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    - PLANT ACCOUNTANT — A.B. CONDITT
      - ACCOUNTANT B — G. MARNIEN, R.L. VERZWYVELT
      - ACCOUNTANT C — K.H. GIMBEL, G. WILKS
      - TRAFFIC SUPERVISOR — T.S. RANDOLPH
      - ACCOUNTANT B — L.E. SIMPSON
      - ACCOUNTANT C — P.A. DIBBLEY, P.F. RICHARDSON
      - SUPERVISOR-WAREHOUSE & PURCHASING — B.J. PHILLIPS
      - ACCOUNTANT B — L.E. ROYER
      - ACCOUNTANT C — T. BOGGAN, B. BOZEMAN, OPEN
      - OFFICE SERVICES COORDINATOR — J.J. BROOKS
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  - CHIEF PLANT CHEMIST — D.D. ZIMMER
    - GROUP LEADER — OPEN
      - PROJECT CHEMIST — K.B. YOUNGBLOOD, A.L. WALLACE, W.W. McGARRY
      - GROUP LEADER — OPEN
      - CONTROL CHEMIST — D.D. PALMER
      - LABORATORY SHIFT SUPERVISOR — A.C. DEATHERAGE, H.S. ROBINSON, C.R. SHREWSBERRY, B.J. WHITE
      - ANALYST 18 — TRAINEES 3
  - CHIEF PROCESS ENG. — G.L. FOSHEE
    - SR. PROCESS ENGINEERS — G. HOENES, M.W. McCAULLEY, S.A. REY
    - PROCESS ENGINEERS — J.E. BIGGART, K.N. CHAMBERLAIN, A.B. FINLEY, M.M. GOODREAU, J.M. KERLEGON, J.C. PIERSON, R.J. SCHETTEK, J.C. SHAMBURGER, B.H. SWAN, P.M. WARD, J.D. ZUILHOF, V.C. ARRINDELL, R.A. DIFULGENTIZ, B.E. DAVIS, R.E. MOSS, L.S. MOSSNER, D.L. TARANTINO

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13	6-1-82	REDRAWN	WW
12	2-1-82	REDRAWN	WW
11	7-15-81	REDRAWN	WW
10	2-1-81	REDRAWN	CVG
9	7-15-80	GEN REVISIONS	WW
8	2-1-80	REDRAWN	WW
7	6-14-79	GEN REVISIONS	RC
6	12-27-78	GEN REVISIONS	CTM
5	7-28-78	GEN REVISIONS	CTM
4	3-3-78	REDRAWN	CTM
3	8-17-77	REDRAWN	CTM
2	6-20-72	GEN REVISION	MJB
1	6-15-69	ORIGINAL	MJB

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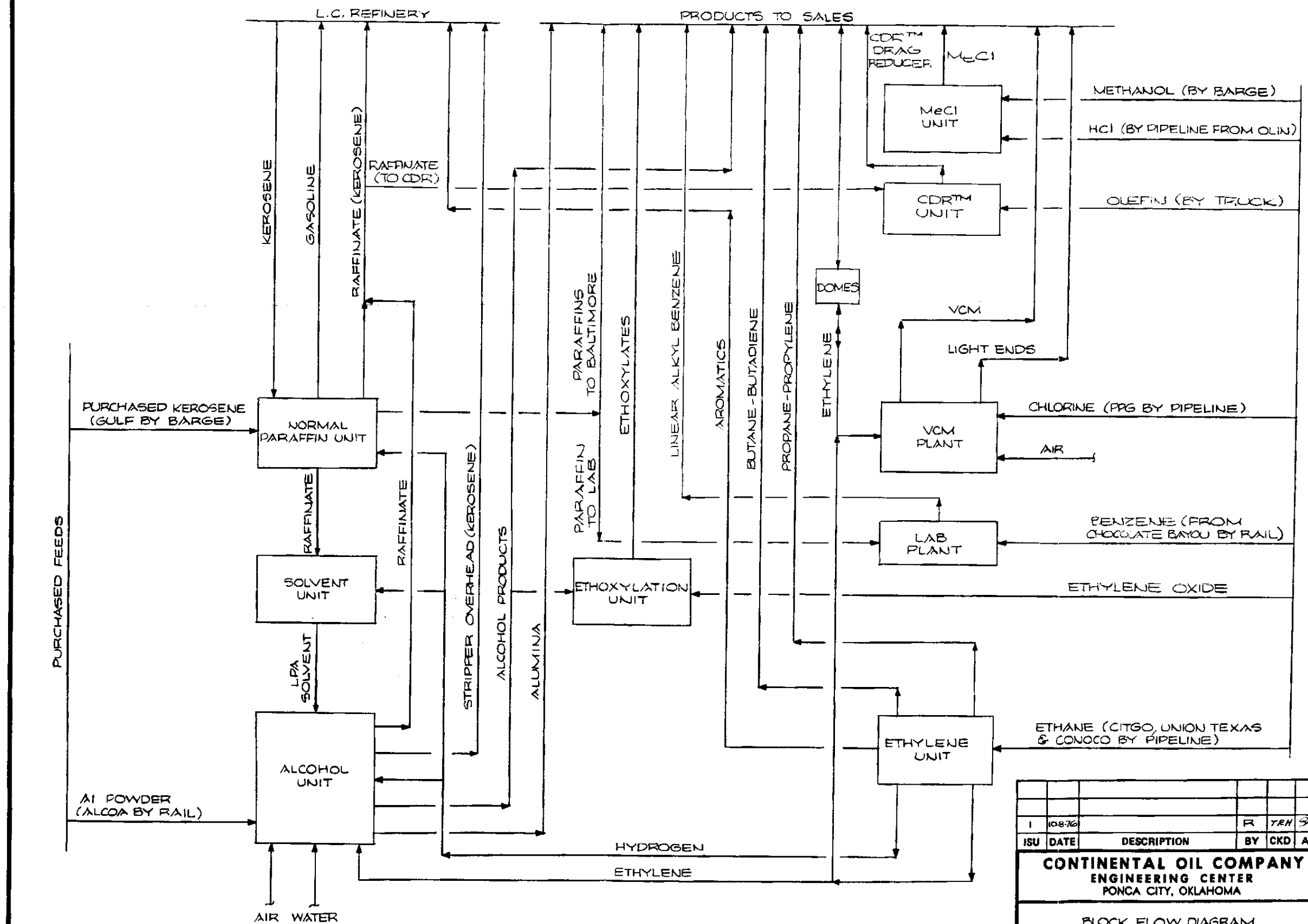

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1	10-8-76		R	TRH	SKB

CONTINENTAL OIL COMPANY
ENGINEERING CENTER
PONCA CITY, OKLAHOMA

BLOCK FLOW DIAGRAM
CHEMICAL COMPLEX
LAKE CHARLES, LA.

USE BORDER SCALE:

APPD: BAR
DATE: 10/11/76

No. CT-12-C

SCALES FOR MICROFILMED DRAWINGS

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"ALFOL" Alcohol Unit

SAL 00001005

PED ORIENTATION PROGRAM

"ALFOL" ALCOHOL UNIT

LAKE CHARLES CHEMICAL PLANT

Conoco's "ALFOL" Alcohol Process produces linear straight-chain alcohols with an even number of carbon atoms. The basis of the process is aluminum alkyl chemistry developed by Professor Karl Ziegler in Germany in the 1930's. The "chemistry" was licensed by Conoco in 1955. The process development was carried out by Conoco's research personnel.

Conoco's "ALFOL" Alcohol Unit is located in Westlake, Louisiana. This plant was the first in the world to start commercial production of normal primary alcohols. Construction of the unit began in 1959, and startup was in 1961. The unit was originally designed for 100 MM pounds per year C_{6}-$_{18}$ alcohols at 4.0 "M" value. In 1971, the plant was expanded to 150 MM pounds per year. The plant is currently operating at an official capacity of about 250 MM pounds per year of C_{6}-$_{18}$ alcohols at 4.2 "M" value.

The short-chained aluminum alkyls are pyrophoric. Above 35 or 40 percent concentration in solvent, they will ignite almost immediately in the presence of oxygen. They react violently when contacted with water or oxygen-containing compounds. Weaker solutions and growth product (aluminum tri-alkyls with longer side chains) may not flame immediately in air. However, a rapid oxidation reaction with substantial heat evolution may raise the fluid temperature rapidly to the flash point. These characteristics require use of special design and operating conditions.

RAW MATERIALS

1. Aluminum powder
2. Ethylene from Conoco's ethylene unit
3. Hydrogen from Conoco's ethylene unit with hydrogen from Conoco's Lake Charles Refinery as backup.
4. Air
5. Sulfuric Acid or water.

GENERAL DESCRIPTION (Refer to attached flow diagrams)

The plant is divided into seven processing units. All are operated continuously except the oxidation process in Section 400.

Section 100 - Aluminum Preparation

The primary purpose of Section 100 is to introduce powdered aluminum into the process for reaction in Section 200. The feeds are aluminum powder and kerosene solvent contaminated with ATE from Section 200. The product of Section 100 is an aluminum powder-solvent slurry to Section 200.

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GENERAL DESCRIPTION (CONTINUED)

Section 200 - ATE Preparation

The function of Section 200 is to manufacture aluminum triethyl (ATE) in solution with kerosene solvent. The aluminum-solvent slurry from Section 100 is combined with recycle ATE-solvent and reacted with hydrogen gas to form aluminum diethyl hydride (ADEH). The ADEH is then reacted with off-gas ethylene from Section 300 to form ATE. The process Reactions are:

$$2Al(C_2H_5)_3 + Al + 1\ 1/2\ H_2 \rightarrow 3Al(C_2H_5)_2H \text{ (Hydrogenation)}$$

$$3Al(C_2H_5)_2H + 3C_2H_4 \rightarrow 3Al(C_2H_5)_3 \text{ (Ethylation)}$$

For every three mols of ATE product from the ethylation reaction, two are recycled to hydrogenation and one is withdrawn as product. The product ATE-solvent-Al slurry stream (containing 4-8 weight percent Al fines) is centrifuged to remove as much of the Al fines as possible.

The products of Section 200 are crude ATE (containing solvent and aluminum) to Section 225 and offgas hydrogen and ethylene to fuel.

Section 225 - ATE Distillation

The function of this section is to distill crude ATE from Section 200 to produce a clear, solids-free ATE solvent feed to Section 300. The feeds consist of ATE-solvent-Al slurry from Section 200 and fresh solvent. The effluents from Section 225, in addition to purified ATE-solvent feed to Section 300, are bottoms ATE and solids to waste drowning (quench reactor) and decomposition offgas to fuel or flare.

Section 300 - Alkyl Growth

The purpose of Section 300 is to form long chain aluminum alkyls from ATE by addition of ethylene molecules. The feeds are ATE-solvent from Section 225 and ethylene from battery limits. The process reaction takes place at 1,600 psig and 250°F and is described as follows:

$$Al(C_2H_5)_3 + 3nC_2H_4 \rightarrow Al[(C_2H_4)nC_2H_5)_3]$$

The alkyl chains formed vary in length approximately according to a Poisson distribution. The effluents are growth product (aluminum alkyls and solvent) to Section 400 and offgas ethylene to Section 200.

Thermal olefins are also formed in the growth reaction by thermal cracking of the aluminum alkyls. The lighter thermal olefins are flashed off with the offgas ethylene, while the heavier ones remain in the growth product.

GENERAL DESCRIPTION (CONTINUED)

Section 400 - Oxidation

The purpose of this section is to oxidize aluminum alkyls to form aluminum trialkoxide. The feeds are growth product from Section 300 and air. The process reaction is:

$$AlR_3 + 1\ 1/2\ O_2 \longrightarrow Al(OR)_3$$

The first part of the reaction step proceeds in a continuous flow-through stirred reactor to yield mono-alkoxy dialkyl aluminum. The remaining two alkyl groups are oxygenated batchwise in stirred reactors. Nitrogen-rich offgas is vented to the atmosphere. The oxidized growth product is charged to Section 425.

Impurities formed in the oxidation reaction constitute the major yield loss in the plant. Some are vented to the atmosphere, but most remain in the oxidized growth product leaving Section 400.

Section 425 - Solvent Stripping

The function of Section 425 is to remove solvent and hydrocarbon impurities from aluminum alkoxide by three-stage flashing and stripping under vacuum conditions. Natural gas is used as a stripping medium in the third stage. The effluents from Section 425 are stripped alkoxide to Section 500, offgas to fuel, and recovered solvent returned to the refinery for reprocessing.

Section 550 - Hydrolysis

Conoco used to hydrolyze stripped alkoxide with sulfuric acid in Section 500 to yield crude linear alcohols and alum (aluminum sulfate hydrate). This operation yielded alum via the following chemistry:

$$Al(OR)_3 + 1\ 1/2\ H_2SO_4 + 18\ H_2O \longrightarrow 3ROH + 1/2\ Al_2(SO_4)_3 \cdot 18\ H_2O$$

The crude reaction products were then phase split and alum was sent to product storage. The crude alcohols were distilled successively to remove water, ethanol, and butanol. The remaining alcohols were then sent to the main alcohol fractionation train in Section 600.

Section 600 - Alcohol Fractionation

In Section 600 the crude alcohols are fractionated into individual one or two alcohol range products. The alcohols are hydrogentated to remove unsaturates. Hydrogen gas from battery limits is reacted under pressure with unsaturated alcohols in the presence of a nickel catalyst as follows:

$$C\text{-}C\text{-}C{=}C\text{-}C\text{-}C\text{-}OH + H_2 \xrightarrow{\text{Ni Catalyst}} C\text{-}C\text{-}C\text{-}C\text{-}C\text{-}C\text{-}OH$$

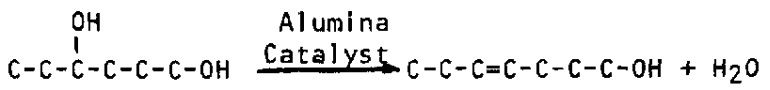
Conoco now operates exclusively a water hydrolysis process to yield crude

GENERAL DESCRIPTION (CONTINUED)

Section 600 - Alcohol Fractionation (Continued)

linear alcohols and alumina. Due to the complexity of this unit and the importance of the alumina product, this process is covered in a subsequent section.

Another function performed in Section 600 is reduction of the diol content of the 12+ alcohols via reaction using alumina catalyst.



The effluents from Section 600 are C_6, C_{810}, C_{1214}, C_{1618}, C_{1620}, or C_{20}^+ alcohols from normal fractionation. A utility fractionation section is used to produce heart-cuts of the alcohols such as C_6, C_8, C_{10}, etc.

PLANT UTILITY SUPPLIES

Solvent is supplied by the LPA solvent portion of the n-Paraffins Unit. Steam is obtained from the ethylene unit boilers. The plant has its own furnaces for hot oil supply and its own cooling towers for cooling water. Nitrogen gas, fuel gas, and electricity are purchased.

PED ORIENTATION PROGRAM

TABLE I

"ALFOL" ALCOHOL PROPERTIES AND USES

CONOCO "ALFOL" ALCOHOL PROCESS

Alcohol Product Range	Intermediates	Typical Uses
Plasticizer Range (C_4 to C_{10})	"ALFOL" Alcohol Phthalates "ALFOL" Alcohol Esters	Imitation Coatings, Cable Industry, Leathercloths, PVC Sheetings, PVC Plates, Films, Bag Makers, Foaming Materials, Extreme Thermo-stable Sheetings
Detergent Range (C_{10}-C_{20}+)	"ALFOL" Alcohol Sulfates "ALFOL" Alcohol Ether-Sulfates "ALFOL" Alcohol Nonionics	Detergents, Emulsifiers, Demulsifiers, Defoamers, Ore Flotation Agents, Textile Processing Agents, Cosmetics, Shampoos, Ointments

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Spec. No. 06-4000

ALFOL 4 Alcohol

Special Identification: 1-butanol

Test	Specifications	Typical Property	Test Method
Total Alcohol, Wt. %	99.0 min.	99.5	1.055
Distribution (100% alcohol basis):			1.055
C_2 OH, Wt. %	1.0 max.	TR	
C_4 OH, Wt. %	98.0 min.	99.8	
C_6 OH, and higher, Wt. %	1.0 max.	0.2	
Alcohol Color, APHA	10 max.	0	1.025
Water, Wt. %	0.1 max.	0.05	1.016
Acid, as Acetic, Wt. %	0.01 max.	0.001	1.024
Iodine Number	0.2 max.	0.05	1.031
Boiling Point Range, °C	4 max.	2.5	ASTM-D-1078

8-15-73
(Replaces 10-1-72)

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Spec. No. 06-4010

ALFOL 6 Alcohol

Special Identification: 1-hexanol

Test	Specifications	Typical Property	Test Method
Total Alcohol, Wt. %	98.5 min.	99.4	1.055
Distribution (100% alcohol basis):			1.055
C_6 OH, Wt. %	98.0 min.	98.6	
Alcohol Color, APHA	10 max.	0	1.025
Water, Wt. %	0.15 max.	0.06	1.016
Acid, as Acetic, Wt. %	0.005 max.	0.001	1.024
Iodine Number	0.2 max.	0.06	1.031

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8-15-73
(Replaces 10-1-72)

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Spec. No. 06-4020

ALFOL 8 Alcohol

Special Identification: 1-octanol

Test	Specifications	Typical Property	Test Method
Total Alcohol, Wt. %	99.0 min.	99.5	1.055
Distribution (100% alcohol basis):			1.055
C_6 OH and lower, Wt. %	0.5 max.	TR	
C_8 OH (normal primary) Wt. %	98.8 min.	99.7	
C_{10} OH and higher, Wt. %	0.5 max.	0.3	
Alcohol Color, APHA	10 max.	0	1.025
Water, Wt. %	0.10 max.	0.05	1.016
Acid, as Acetic, Wt. %	0.005 max.	0.001	1.024
Iodine Number	0.2 max.	0.05	1.031
Carbonyl, as C=O, ppm	80 max.	50	1.026

8-15-73
(Replaces 10-1-72)

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Spec. No. 06-4030

ALFOL 10 Alcohol

Special Identification: 1-decanol

Test	Specifications	Typical Property	Test Method
Total Alcohol, Wt. %	98.5 min.	99.2	1.055
Distribution (100% alcohol basis):			1.055
C_8 OH and lower, Wt. %	-	0.1	
C_{10} OH, Wt. %	98.0 min.	99.3	
C_{12} OH and higher, Wt. %	-	0.6	
Alcohol Color, APHA	10 max.	0	1.025
Water, Wt. %	0.15 max.	0.04	1.016
Acid, as Acetic, Wt. %	0.005 max.	0.001	1.024
Iodine Number	0.2 max.	0.06	1.031
Carbonyl, as C=O, ppm	100 max.	25	1.026

1-26-76
(Replaces 8-15-73)

SAC 000010104.

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Spec. No. 06-4040

ALFOL 12 Alcohol

Special Identification: 1-dodecanol

Test	Specifications	Typical Property	Test Method
Total Alcohol, Wt. %	98.5 min.	99.4	1.055
Distribution (100% alcohol basis):			1.055
C_{12} OH Normal Primary, Wt. %	98.5 min.	99.2	
Alcohol Color, APHA	15 max.	0	1.025
Hydroxyl Number	295-302	300	1.029
Water, Wt. %	0.1 max.	0.04	1.016
Iodine Number	0.2 max.	0.03	1.031
Carbonyl, as C=O, ppm	100 max.	64	1.026

8-15-73
(Replaces 10-1-72)

SAL00001010S

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Spec. No. 06-4050

ALFOL 14 Alcohol

Special Identification: 1-Tetradecanol

Test	Specification	Typical Property	Test Method
Total Alcohol, Wt. %	98.5 min.	99.0	1.055
Distribution (100% Alcohol Basis):			1.055
$C_{12}OH$ and Lower, Wt. %	2.0 max.	0.9	
$C_{14}OH$, Wt. %	96.0 min.	98.4	
$C_{16}OH$ and Higher, Wt. %	2.0 max.	0.7	
Alcohol Color, APHA	20 max.	0	1.025
Hydroxyl Number	255-264	258	1.029
Water, Wt. %	0.1 max.	0.04	1.016
Iodine Number	0.4 max.	0.05	1.031
Carbonyl, as C=O, ppm	200 max.	100	1.026

5-23-79
(Replaces 8-15-73)

SAL00001010G

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Spec. No. 06-4060

ALFOL 16 Alcohol

Special Identification: 1-hexadecanol

Test	Specifications	Typical Property	Test Method
Total Alcohol, Wt. %	97.5 min.	98.9	1.055
Distribution (As-is sample basis):			1.055
C_{14} OH and lower, Wt. %	3.0 max.	0.7	
C_{16} OH, Wt. %	95.0 min.	97.4	
C_{18} OH and higher, Wt. %	3.0 max.	0.8	
Alcohol Color, APHA	40 max.	25	1.025
Saponification Number	1.0 max.	0.8	1.030
Hydroxyl Number	218-238	227	1.029
Water, Wt. %	0.1 max.	0.03	1.016
Iodine Number	0.6 max.	0.15	1.031
Carbonyl, as C=O, ppm	350 max.	140	1.026

8-15-73
(Replaces 10-1-72)

SAL 0C0C1C1C7

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Spec. No. 06-4070

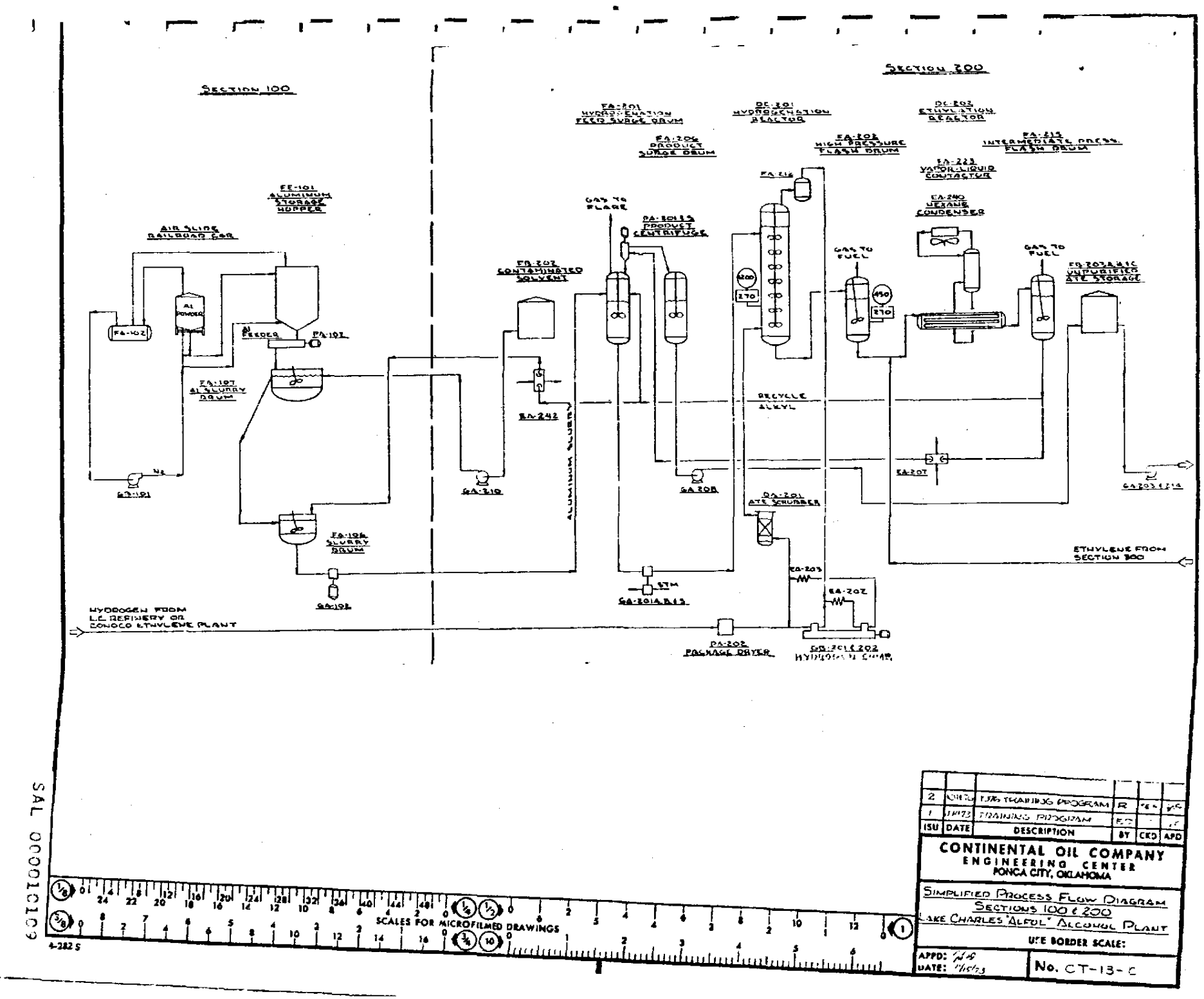
ALFOL 18 Alcohol

Special Identification: 1-Octadecanol

Test	Specification	Typical Property	Test Method
Total Alcohol, Wt. %	98.0	98.2	1.055
Distribution (As-Is Sample Basis):			1.055
$C_{14}OH$ and Lower, Wt. %	0.5 max.	0.2	
$C_{16}OH$ and Lower, Wt. %	3.0 max.	0.7	
$C_{18}OH$, Wt. %	95.0 min.	96.6	
$C_{20}OH$ and Higher, Wt. %	3.0 max.	0.9	
Alcohol Color, APHA	40 max.	20	1.025
Saponification Number	1.5 max.	0.4	1.030
Hydroxyl Number	200-220	200	1.029
Water, Wt. %	0.1 max.	0.05	1.016
Iodine Number	1.0 max.	0.58	1.031
Carbonyl, C=O, ppm	700 max.	600	1.026

5-23-79
(Replaces 8-15-73)

SAL 0000101C8



ISU	DATE	DESCRIPTION	BY	CKD	APD
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1	3/1/73	TRAINING PROGRAM	R.S.	[illegible]	[illegible]

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ENGINEERING CENTER
PONCA CITY, OKLAHOMA

SIMPLIFIED PROCESS FLOW DIAGRAM
SECTIONS 100 & 200
LAKE CHARLES "ALFOL" ALCOHOL PLANT

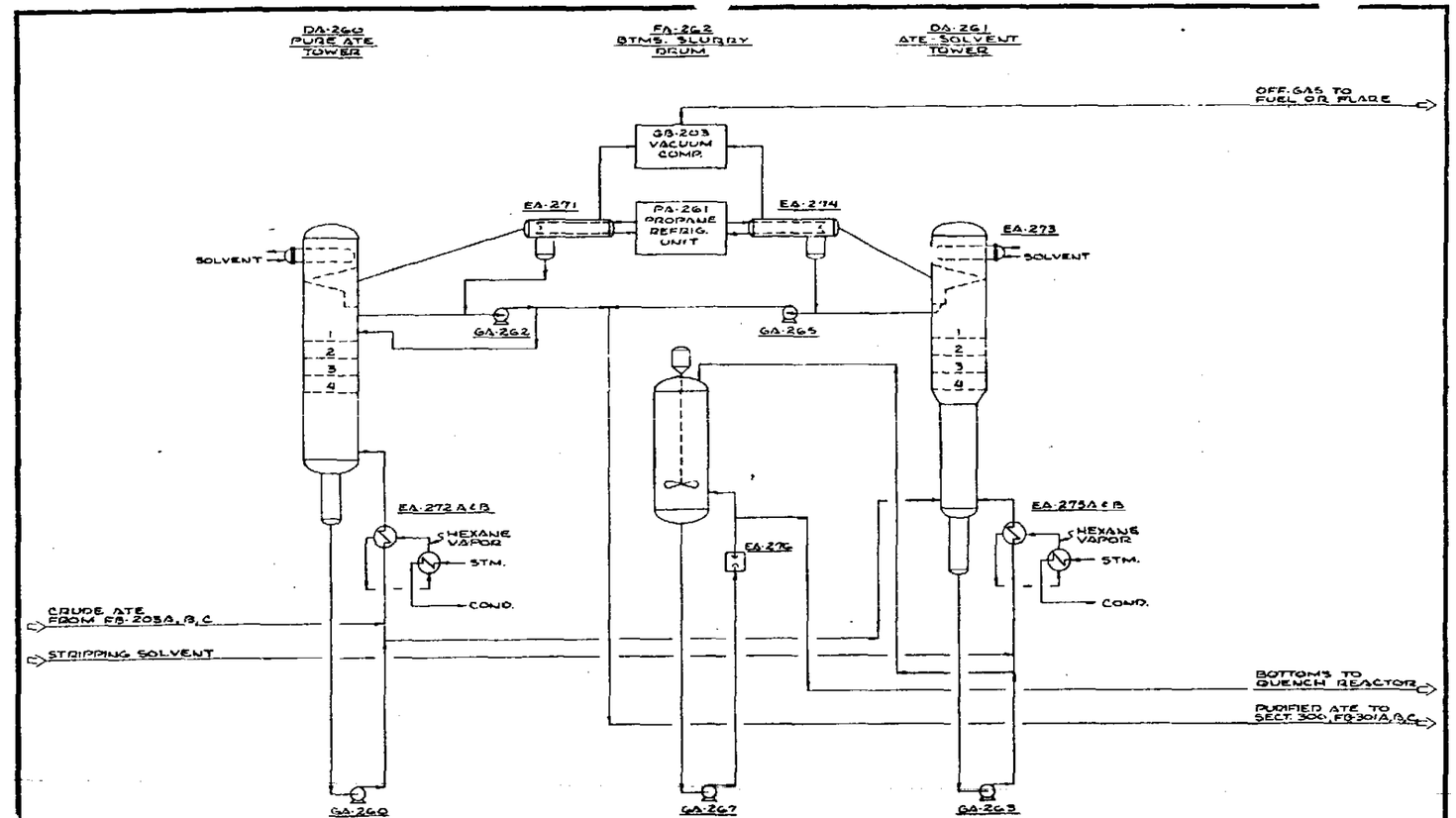
UTE BORDER SCALE:

APPD: [illegible]
DATE: 7/16/73

No. CT-13-C

SAL 00010109

4-282 S



ISU	DATE	DESCRIPTION	BY	CKD	APD
1	11-4-73	TRAINING PROGRAM	ED	JD	JC

CONTINENTAL OIL COMPANY
ENGINEERING CENTER
PONCA CITY, OKLAHOMA

SIMPLIFIED PROCESS FLOW DIAGRAM
SECTION 225
LAKE CHARLES "ALFOL" ALCOHOL PLANT

USE BORDER SCALE:

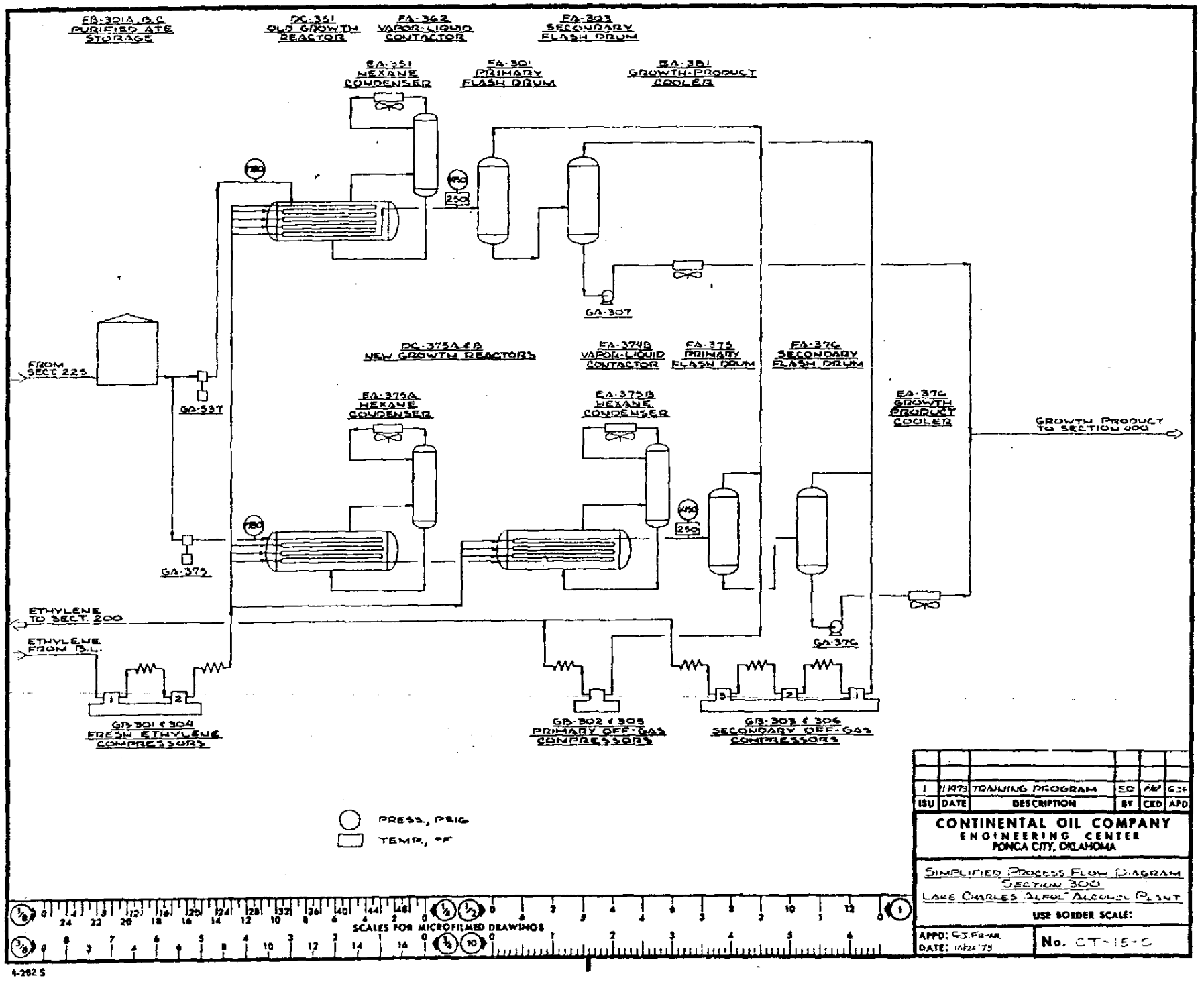
APPD: JC
DATE: 11-4-73

No. CT-14-C

SCALES FOR MICROFILMED DRAWINGS

4-282 S

SAL 00001110



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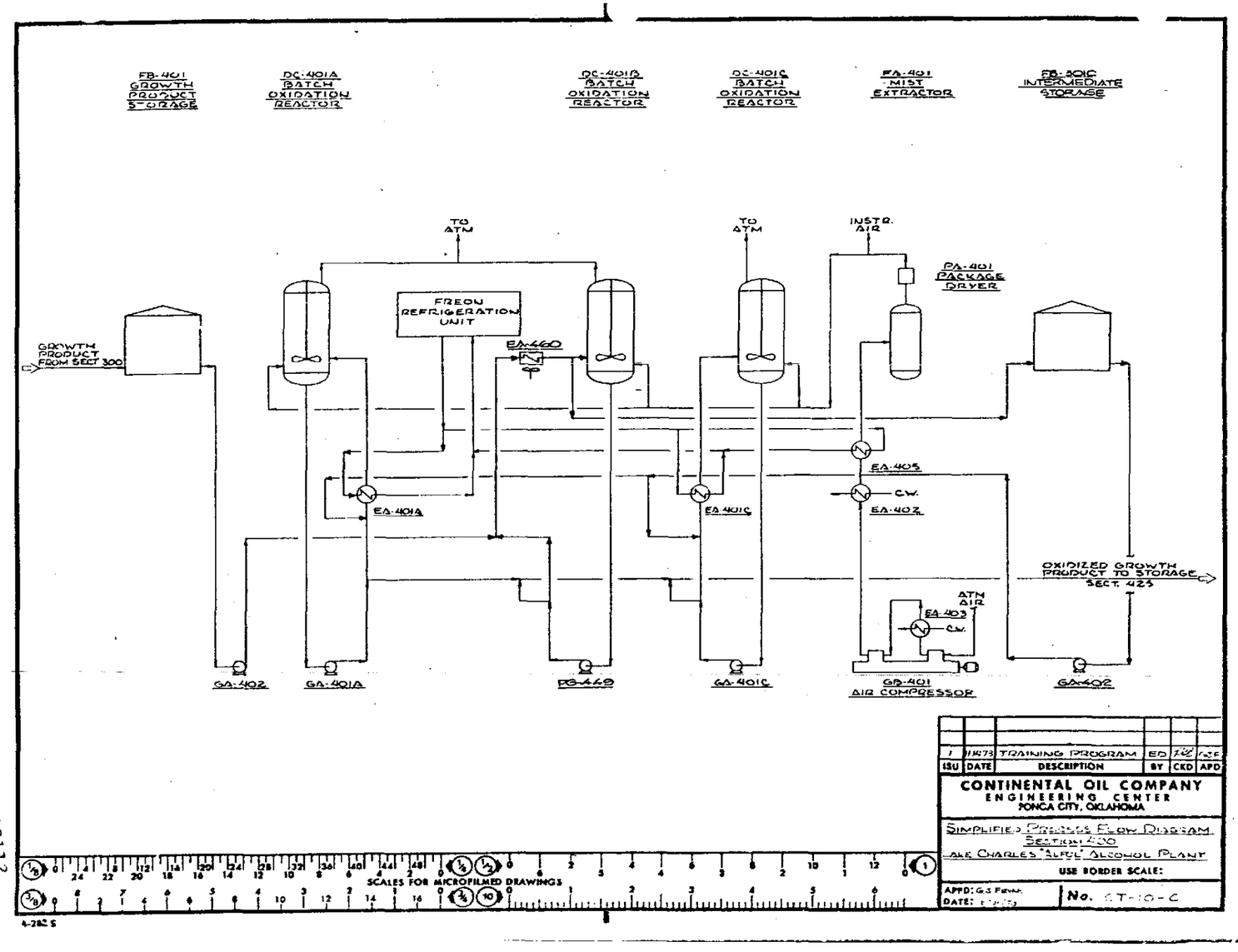

ISU	DATE	DESCRIPTION	BY	CKD	APD
1	11473	TRAINING PROGRAM	ED	TJC	PCF

CONTINENTAL OIL COMPANY
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PONCA CITY, OKLAHOMA

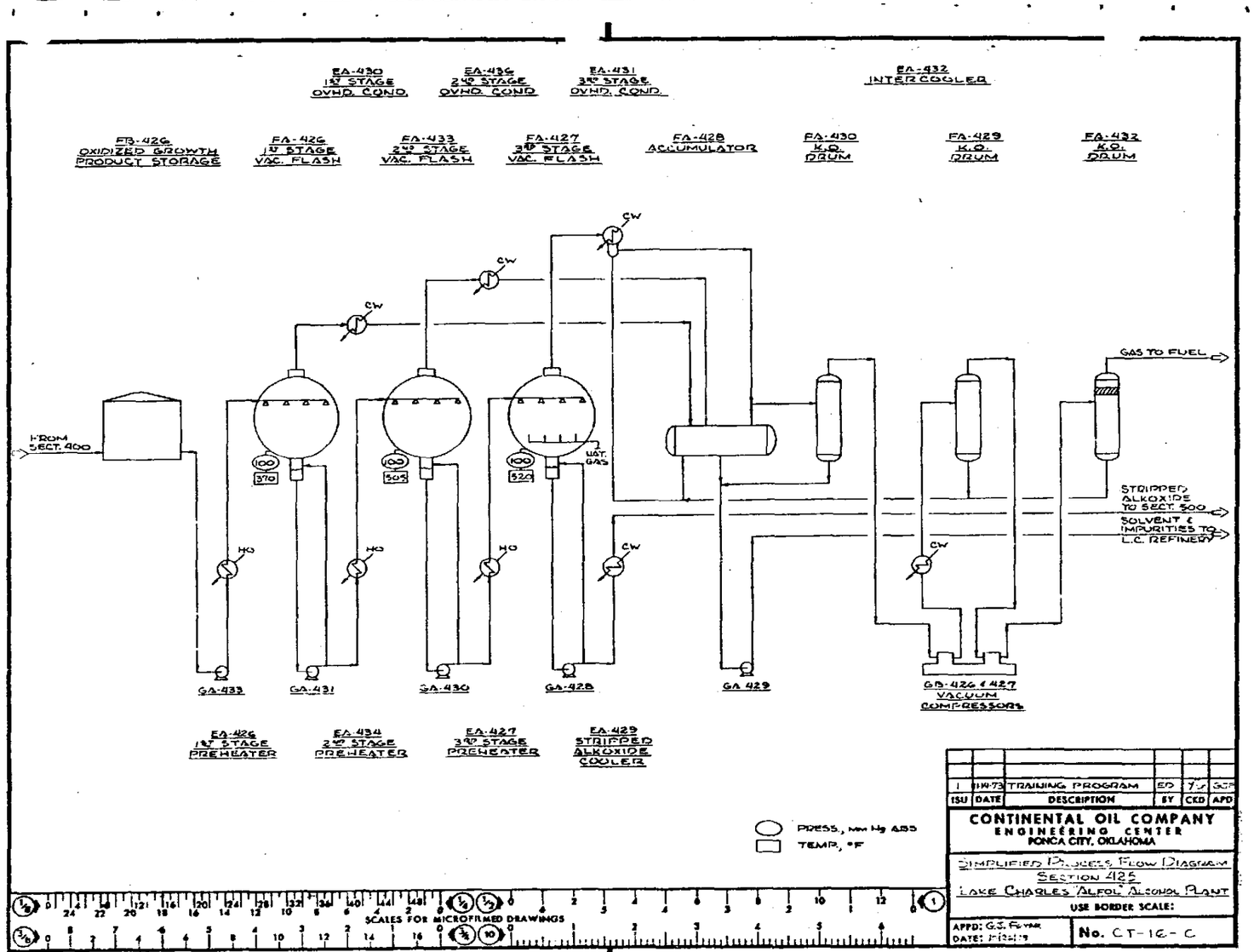
SIMPLIFIED PROCESS FLOW DIAGRAM
SECTION 400
LAKE CHARLES "SUPER" ALCOHOL PLANT

USE BORDER SCALE:

APPD: G.S. FRANK
DATE: 5-20-73

No. 67-10-C

SAL 00010112



SAL 0C0010113

Alumina Unit

SAL 00010114

PED ORIENTATION PROGRAM

SECTION 550 - ALUMINA UNIT
("ALFOL" Alcohol Unit)

LAKE CHARLES, LOUISIANA

The chemistry of the "ALFOL" Alcohol Process requires that some type of aluminum-containing compound issue as a by-product of the aluminum trialkoxide hydrolysis reaction. The Lake Charles plant started up with acid hydrolysis yielding alum by-product. (The acid hydrolysis unit was described within the framework of the "ALFOL" Alcohol Unit). The Condea plant startup three years later produced alumina. Ultimately, the alumina became an important sales factor. Conoco then updated the prior technology and installed a 40 MM pound per year Alumina Unit at Lake Charles. Lake Charles currently operates only the Alumina Unit. The Alumina Unit is presented as a separate entity reflecting the complexity of the process and the importance of the product. Raw materials for the process are water and aluminum trialkoxide from Section 425 of the "ALFOL" Alcohol Unit.

GENERAL DESCRIPTION

Section 550 of the Lake Charles "ALFOL" Alcohol Unit provides for water hydrolysis of solvent-free aluminum alkoxide (produced in Section 425) with the formation of alumina and straight-chain alcohols containing even carbon numbers. High purity alumina, denatured ethanol, and solvent-grade butanol are recovered in this section while C_6+ alcohols are sent to Section 600 for further processing. Three processing systems are contained in Section 550:

A. Hydrolysis-Extraction System
B. Fractionation System
C. Alumina Recovery System

In the hydrolysis-extraction system, stripped aluminum alkoxide reacts with water according to the following equations:

$$\underset{\text{(Monoaluminum Alkoxide)}}{2Al(OR)_3} + 3H_2O \longrightarrow \underset{\text{(Alumina)}}{Al_2O_3} + 6ROH \qquad (1)$$

$$\underset{\text{(Dialuminum Alkoxide)}}{Al_2(OR)_4O} + 2H_2O \longrightarrow Al_2O_3 + 4ROH \qquad (2)$$

$$\underset{\text{(Titanium Alkoxide)}}{Ti(OR)_4} + 2H_2O \longrightarrow TiO_2 + 4ROH \qquad (3)$$

The above reactions are exothermic and carried out at 200°F in the presence of excess water. The two products from the reaction are an alumina-water slurry and crude alcohols. These are mutually insoluble and separate in the hydrolysis reactor. Any C_6+ alcohols contained in the alumina-water slurry from the hydrolysis reactor are removed in a two-stage countercurrent extraction with butanol.

GENERAL DESCRIPTION (CONTINUED)

In the fractionation system, the wet crude alcohol from the hydrolysis reactor is sent to a dehydrator tower where the water is removed. The condensed overheads from the dehydrator tower and the butanol stripper (in the alumina recovery system) are combined and sent to the dehydrator tower overhead accumulator. This combined stream, which consists of water, ethanol, and butanol, separates into two phases in this accumulator. The water phase is combined with makeup condensate and barometric condenser blowdown water and recycled to the hydrolysis reactor. The alcohol phase is fed to an ethanol tower where a 93 percent ethanol product is recovered. The bottoms from the ethanol tower consists of water and butanol which is used for extraction in the hydrolysis-extraction system.

The water-free crude alcohol from the dehydrator tower is sent to a butanol tower where a high purity butanol product is taken overhead. Most of the butanol product is recycled to the process while the net make butanol is sent to the hydrogenation section. The bottoms from the butanol tower is transferred to Section 600 for further fractionation.

In the alumina recovery system, the alumina-water slurry from the hydrolysis-extraction system is sent to a butanol stripper where butanol is stripped from the slurry with steam. The alcohol-free slurry is then sent to a spray dryer for recovery of a high purity, high surface area powdered alumina. The spray dryer utilizes direct heating with air and flue gases which enter at 1,400°F and exit at 300-350°F. The alumina product (75 percent alumina/25 percent water) from the spray dryer is transferred to product storage where it is loaded into railcars and trucks for bulk shipment.

DETAILED PROCESS DESCRIPTION

A. Hydrolysis-Extraction System

Stripped aluminum alkoxide is received from Section 425 at 160°F. The alkoxide is pumped from intermediate storage tanks FB-506A or FB-508, heated to 200°F with steam in EA-551, and injected into the hydrolysis reactor, DV-551, through a small diameter nozzle which promotes turbulence. The alkoxide enters the reactor in the slurry phase between the two impellers of the reactor mixer. It reacts rapidly to form crude "ALFOL" alcohols and hydrated alumina.

The hydrolysis water to the reactor consists of makeup condensate, barometric condenser blowdown water, recycle water from the distillation system, and ammonium hydroxide solution from the ammonia absorber. Condensate is used as process water to prevent contamination of product alumina with foreign minerals. The hydrolysis water is pumped from the hydrolysis water tank, FB-551, to the hydrolysis reactor, DV-441, via the steam ring heater, PA-551. In this heater, the water temperature is raised to about 170°F by direct steam injection.

A. Hydrolysis-Extraction System (Continued)

An ammonia concentration of about 0.15 weight percent is maintained in the hydrolysis water. The ammonia prevents formation of emulsions in downstream equipment. This concentration is maintained by injection of ammonium hydroxide into this stream intermittently.

A wet butanol stream from the extraction system also enters the slurry phase of the reactor to enhance removal of heavier alcohols.

A crude alcohol phase and an alumina-water slurry phase are produced in the hydrolysis reaction. A nitrogen pressure of 80 psig is maintained in the hydrolysis reactor to permit transfer of slurry by pressurization rather than pumping. In normal operation, most of the crude alcohol is pressured directly from the reactor to the dehydrator tower to conserve energy. The remaining alcohol is cooled in EA-553 to 175°F and sent to the alcohol surge tank, FB-553. Five recycle alcohol streams also flow into this tank:

1. Remelted barometric condenser cooling tower sump scrapings.
2. Remelted railcar scrapings.
3. Tower "G" overheads from Section 600.
4. Off-spec alcohols from Section 600.
5. Skimmed alcohols from hydrolysis water tank, FB-551.

These alcohols are pumped from FB-553 to the dehydrator tower continuously during normal operation.

The alumina-water slurry phase from the hydrolysis reactor contains dissolved and entrained alcohols. The alcohols must be removed and recovered to avoid an unacceptable carbon content in the alumina product and avert an alcohol product loss.

A two-stage countercurrent extraction with butanol is used to remove the C_6+ alcohols from the slurry. The alumina slurry is pressured from the hydrolysis reactor, DC-551, to the first stage mixing drum, FA-553. Here contact is made with the butanol phase from the second stage phase separator drum, FA-554. The effluent from the first stage mixer is phase split in the first stage phase separator drum, FA-552. The alcohol phase from FA-552 is pumped to the hydrolysis reactor.

The alumina slurry from the first stage phase separator is pressured to the second stage mixing drum, FA-551. Here it contacts fresh recycle butanol from the recycle butanol tank, FB-552. In order to maintain the alumina slurry at 185°F, the recycle butanol is preheated to 169°F in EA-552. The alcohol/slurry mixture from the second stage mixer is transferred to the second stage phase separator, FA-554. The alcohol phase from this separator is pumped to the first stage mixing drum. The slurry is pressured to the butanol stripper, DA-554.

A. Hydrolysis-Extraction System (Continued)

Parameters affecting the operation of the reaction-extraction system are reaction and extraction temperature, hydrolysis water and recycle butanol flow rates, and ammonia and ethanol concentration. The most economical operation is at the following conditions:

1. Minimum Hydrolysis Water Flow (or Maximum Slurry Concentration)--Since the water in the slurry is evaporated during spray drying, less water results in less condensate makeup and fuel requirements.

2. Minimum Butanol Recycle Flow--Since less butanol decreases the load on the fractionation system.

Hydrolysis water flow rate should be set to give a butanol stripper slurry feed concentration of 9-12 weight percent solids; higher solids concentrations can cause slurry transfer problems. Recycle butanol flow rate should be set at the minimum value which consistently gives acceptable extraction. The design hydrolysis reaction temperature is 200°F. Higher temperatures will decrease alumina product surface area and increase butanol solubility in the slurry. The reaction temperature is regulated by adjusting hydrolysis water and alkoxide feed temperatures. Temperature of the second stage extraction is set based on butanol solubility in the slurry and slurry flow characteristics. Emulsions can occur in the reaction-extraction system due to ammonia deficiencies or excess ethanol concentrations. The ethanol tower removes the net make ethanol and is designed to give minimum ethanol recycle.

B. Fractionation System

Crude alcohol containing approximately 11 weight percent water is fed to the dehydrator tower, DA-551, directly from the reactor, DC-551, and from the alcohol surge tank, FB-553. Before entering the tower, the feed is filtered in FD-551 to remove any entrained alumina particles and then heated to its bubble point of 208°F in EA-555. The dehydrator removes the water as a butanol/water azeotrope along with the ethanol and ammonia. High purity butanol from the butanol tower is "refluxed" to the top tray of the tower to minimize C_6+ alcohol carryover.

The water-free crude alcohol bottoms from the dehydrator tower is fed to the butanol tower, DA-553. A 99+ percent butanol product is distilled overhead in the butanol tower. The butanol tower overhead product provides reflux to the dehydrator tower and supplements the recycle butanol requirements (to the hydrolysis-extraction system). The net make butanol is sent to the hydrogenation section. The butanol-free tower bottoms is transferred to intermediate storage located in Section 600.

The dehydrator tower overhead is condensed, subcooled to 160°F, and combined with the butanol stripper overhead (also at 160°F). The combined stream enters the dehydrator tower overhead accumulator, FA-555, and separates into an alcohol phase and an aqueous phase. The aqueous phase is pumped to the steam ring heater, PA-551, for recycle to the hydrolysis reactor.

B. Fractionation System (Continued)

The alcohol phase from the dehydrator tower overhead accumulator is fed to the ethanol tower, DA-552. The ethanol tower removes the net make ethanol and minimizes ethanol recycle to the hydrolysis-extraction system. Low ethanol recycle rates reduce emulsion formation in the hydrolysis reactor. The ethanol product is removed as an ethanol/water azeotrope (about 93 weight percent ethanol) from a sidedraw on Tray 11 of the ethanol column. The ethanol sidedraw product gravity flows to product storage FB-506C. The top ten trays of the ethanol tower remove ammonia from the ethanol product. The overhead vapor consisting of most of the ethanol and water and essentially all of the ammonia is partially condensed in EA-560 and enters the overhead accumulator, FA-558. The liquid in FA-558 is returned as reflux to the ethanol tower. The uncondensed vapor from FA-558 is partially condensed in the ethanol tower vent condenser, EA-562, and returned by gravity flow to FA-558. The purpose of EA-562 is to minimize ethanol recycled to the hydrolysis-extraction system. The uncondensed vapor from EA-562 which is mostly ammonia with some ethanol and water, is absorbed with service water in the ammonia absorber, DA-55. The ammonium hydroxide solution gravity flows from DA-555 to the hydrolysis water tan, FB-551. This enables almost all the ammonium hydroxide introduced into the process to be recycled, minimizing fresh ammonium hydroxide makeup and pollution problems.

The ethanol tower bottoms is cooled to 50°F in EA-558 and transferred to the recycle butanol tank, FB-552.

C. Alumina Recovery System

The alumina slurry from the second stage separator drum, FA-554, is pressured directly to the butanol stripper, DA-554. The slurry is fed to the top tray. Live steam, desuperheated in PA-552 to process conditions, is used to strip all butanol (as a butanol/water azeotrope) and lighter components from the slurry. A cyclone, FA-560, is provided in the butanol stripper overhead vapor stream to remove any entrained alumina particles. The overhead is condensed and subcooled to 160°F in EA-565 and combined with the dehydrator tower overhead stream (see above).

The alcohol-free slurry is pumped from the butanol stripper using a positive displacement pump with a variable-speed drive. This slurry first enters EA-566 where it is cooled to 195°F and then it flows to the alumina slurry surge tank, FB-554.

The alumina slurry is pumped from FB-554 through the spray dryer feed filter, FD-552, to the spray dryer, EC-55, using another positive displacement pump with a variable-speed drive. The slurry enters the spray dryer through a centrifugal wheel atomizer. The resulting slurry droplets contact the hot air and flue gases from the spray dryer air heater, BA-551. Combined stripper overhead from Section 425 (liquid fuel) is pumped in from FB-555 to fire the heater. The inlet hot air

DETAILED PROCESS DESCRIPTION (CONTINUED)

C. Alumina Recovery System (Continued)

temperature is maintained at 1,400°F by using excess air supplied by GB-551, the combustion air blower. The exhaust gases and alumina leave the spray dryer at 300-500°F and enter a baghouse, FD-444, which recovers the alumina product. The alumina-free exhaust gas is vented to the atmosphere from the spray dryer exhaust fan discharge, GB-552.

The alumina product from the baghouse is transferred to one of the alumina check bins, FB-556A, B, and C (one for each shift). After product testing, the alumina is transferred to one of the product silos, FB-557A, B, C, and D. The product is loaded into trucks and railcars. Inventories are measured by load cells on the silos. An off-specification product silo, FB-558, is also available when needed.

PRODUCT

"CATAPAL-SB" is the tradename for the high purity, high surface area, powdered alumina product. In contrast to alumina produced from mineral deposits, "CATAPAL-SB" and the alumina products can be tailored to have a variety of physical properties. The major use of alumina is catalyst support material for petroleum refining processes. See the following page for typical product specifications.

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Spec. No. 06-5400

CONOCO CATAPAL SB ALUMINA

Test	Specifications	Test Method
Al_2O_3, Wt. %	70 min.-78 max.	1.400
Carbon, Wt. %	0.5 max.	1.401
SiO_2, Wt. %	0.01 max.	1.402
Fe_2O_3, Wt. %	0.01 max.	1.403
Na_2O, Wt. %	0.01 max.	1.404
Physical Properties		
Particle Size Distribution		1.405
Smaller than 45 microns	55% max.	
Greater than 90 microns	15% max.	
Bulk Density, Loose, gms/liter	660-740	1.406
Pore Volume (0-800A)*, ml/gm	0.4 min.	1.407
Spec. Surface Area (BET)*, m^2/gm	230 min.	1.408
Crystallite Structure	Boehmite Type	1.409

* After calcination for 3 hours at 900°F.

7-15-77
(Replaces 11-15-74)

SAL 0000010121

SAL 0000161224

CDR™ Unk

PED ORIENTATION PROGRAM

CDR™ DRAG REDUCER PLANT

LAKE CHARLES CHEMICAL PLANT

CDR™ drag reducer is a very high molecular weight poly alpha-olefin. It was developed by Chemicals Research in cooperation with Production Research for use in crude oil pipelines and as a friction reducer in hydraulic fracture treatments of oil wells.

Conoco was awarded a patent in 1972 for CDR™ drag reducer use in pipelines. The material and its applications were developed during the late 1960's. It was tested in a number of pipelines with its performance being a technical success. However, in each case, economic considerations indicated expansion of pipeline capacity through equipment changes and additions rather than using CDR™ drag reducer. This situation prevailed until 1977 when interest in CDR™ drag reducer was dramatically revived as a result of the fire at pump station No. 8 shortly after startup of the Trans Alaska Pipeline System (TAPS). The problem reduced crude oil flow from 1.2 MM BPD to 730,000 BPD. At that time, Alyeska Pipeline Service Company approached Conoco with an emergency interest in CDR™ drag reducer. However, the pump station outage was to last "only" six months, not enough time to allow for commercialization of CDR™ drag reducer.

Although the immediate need could not be met, enough information was developed to point to a potentially attractive future use of CDR™ drag reducer in TAPS to give additional incremental throughput.

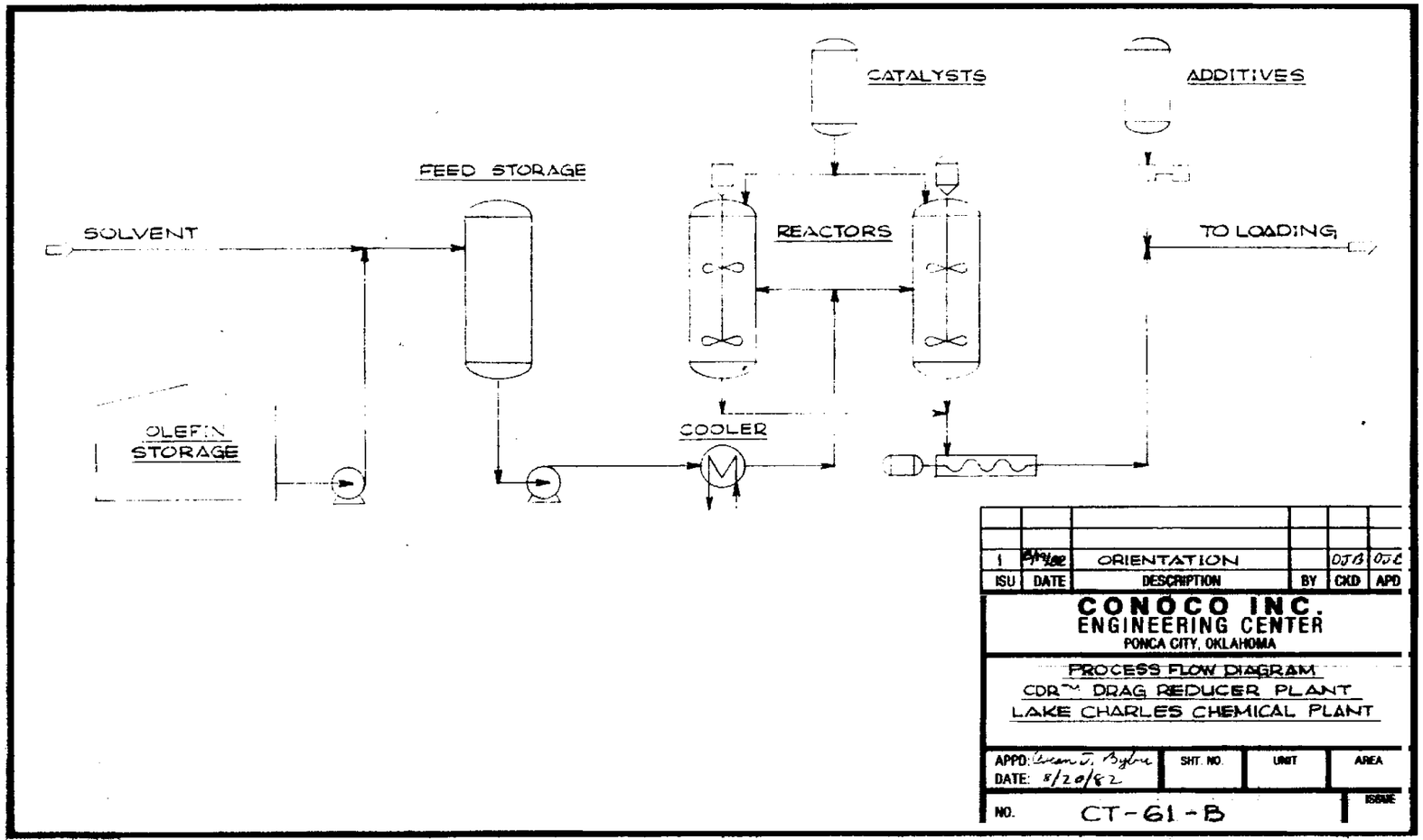
A pilot plant, including a 3,500-gallon reactor, was operated in Ponca City during 1978 to produce an optimum product. Very successful test runs were made in a Conoco eight-inch pipeline and in an Arco fourteen-inch pipeline transporting North Slop crude oil. Finally, a full scale test run was made in TAPS on April 1, 1979. The test was successful and led to a contract with the Alyeska Pipeline Service Company, with supply to begin in July 1979.

The CDR™ drag reducer plant was designed by PED in Ponca City. The plant was then built in Lake Charles and started up on July 13, 1979. The original plant included an olefin storage tank, a chiller, a reactor, catalyst addition, and two loading spots with a capacity of 3 MM gallons per year of CDR™ drag reducer. Since that time, a feed storage tank, a second reactor, an improved catalyst addition system, three more loading spots, and railcar loading facilities have been added with the capacity now being about 9 MM gallons per year.

The improved catalyst addition system enabled production of a more effective drag reducer which, in turn, led to a new long range contract with Alyeska beginning in 1982.

General Process Description

CDR™ drag reducer is made by charging chilled olefin and plant solvent to a stirred reactor. Catalysts are then charged and a polymerization reaction takes place. When the polymerization reaction is complete, the CDR™ drag reducer is pumped to loading with additives being injected to deactivate the catalysts. The CDR™ drag reducer is loaded into either railcars or sea containers for shipping.



ISU	DATE	DESCRIPTION	BY	CKD	APD
1	8/20/82	ORIENTATION		DJB	DJC

CONOCO INC.
ENGINEERING CENTER
PONCA CITY, OKLAHOMA

PROCESS FLOW DIAGRAM
CDR™ DRAG REDUCER PLANT
LAKE CHARLES CHEMICAL PLANT

APPD: [illegible] DATE: 8/20/82	SHT. NO.	UNIT	AREA
NO. CT-61-B			ISSUE

4-261-S(2-82)

SAL JC0010125

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Spec. No. 11-5590

CONOCO CDR

Test	Specification	Typical Property	Test Method
Polymer Content, Wt %	10 min	10.3	1.085-79
Viscosity, Inherent (300 sec^{-1})	8.5 min	9	1.084-79
Chlorides, Total, Wt %	0.12 max	0.07	1.083-79
Metals Analysis, WT %			1.086-79
Sodium	0.09 max	0.05	
Titanium	0.03 max	0.02	
Aluminum	0.05 max	0.03	
Lead	0.002 max	<0.001	
Nickel	0.005 max	<0.001	
Copper	0.002 max	<0.001	
Ash, Total, Wt %	0.30 max	0.19	1.087-79

8-1-80
(Replaces 7-16-79)

SAL 000010126

Ethoxylation Unit

SAL 0C0C1C127

PED ORIENTATION PROGRAM

ETHOXYLATION UNIT

LAKE CHARLES CHEMICAL PLANT

The ethoxylation technology was developed by Conoco and practiced commercially beginning in 1962. The plant is located adjacent to the "ALFOL" Alcohol Unit in Westlake, Louisiana. The unit has production capability for up to 77 MM pounds per year of "ALFONIC" ethoxylates. These products are excellent feedstocks for detergent manufacturers and are completely biodegradable. Raw materials required include ethylene oxide and linear "ALFOL" alcohols.

GENERAL DESCRIPTION

The process chemistry is represented typically as follows:

$$\underset{\text{"ALFOL" 14 Alcohol}}{C_{14}H_{29}OH} + 6\ \underset{\text{Ethylene Oxide}}{C_2H_4O} \xrightarrow{\text{Catalyst}} \underset{\substack{\text{"ALFONIC" 14 Ethoxylate} \\ \text{(6 Mol Adduct)}}}{C_{14}H_{29}O(C_2H_4O)_6}$$

The plant is operated in batch style. The following description is that of a typical batch cycle (see attached process flow diagram).

A batch of "ALFOL" alcohol is pumped from storage to the stirred pot ethoxylation reactor vessel. The reactor is mounted on a weight indicator (load cells) so that the exact amounts of reactants can be determined. The alcohol is circulated through a heater-cooler back to the reactor and heated with steam to 300°F. Caustic catalyst (about 0.14 weight percent based on alcohols charge) is then injected into the reactor. The reactor is then pressured with nitrogen, and the alcohol is further heated to 360°F. Ethylene oxide is then fed to the reactor from storage. Heat of reaction is removed by circulating the reaction mass through the water-cooled heater-cooler. Addition of ethylene oxide is continued until the desired amount has reacted.

At the end of the batch, a sample from the reactor is titrated to check basicity; and the required amount of glacial acetic acid for neutralization of caustic is added to the reactor from a small acid hopper. The neutralization salts are soluble in the ethoxylate product, and the product is water clear. The product is then cooled and transferred to a rundown tank and to storage. Total batch time is about 2 hours 45 minutes to four hours, depending on product.

Ethylene oxide is volatile, flammable, extremely reactive, and toxic. The vapor is subject to explosive decomposition at high temperature and pressure. During addition of ethylene oxide to the reactor, the unit operator must watch reactor operation very closely.

PRODUCT

Major products volumewise are:

"ALFONIC" 10-12-60	Ethoxylated "ALFOL" 10-12 Alcohol Containing 60 Weight Percent EO
"ALFONIC" 16-18-65	Ethoxylated "ALFOL" 16-18 Alcohol Containing 65 Weight Percent EO
"ALFONIC" 10-14-40	Ethoxylated "ALFOL" 10-14 Alcohol Containing 40 Weight Percent EO
"ALFONIC" 14-12-40	Ethoxylated "ALFOL" 14-12 Alcohol Containing 40 Weight Percent EO
"ALFONIC" 14-12-60	Ethoxylated "ALFOL" 14-12 Alcohol Containing 60 Weight Percent EO

"ALFONICS" are sold to detergent manufacturers for use in liquid and powder detergent formulations. Some "ALFONICS" are also sold as defoaming agents.

"ALFONIC" 10-12-60 is used "as is" as nonionic detergent in light duty liquid detergent formulations.

"ALFONIC" 16-18-65 is used "as is" as nonionic detergent in heavy duty powder detergent formulations.

"ALFONIC' 10-14-60 and 14-12-40 are sulfonated with SO_3 and neutralized with NaOH or NH_3 to form cationic or anionic detergents for use in light duty liquid detergent formulations.

Spec. No. 06-4300

ALFONIC 610-50 Alcohol Ethoxylate

Special Identification: Typical 50% Ethoxylate of ALFOL 610 Alcohol (Spec. No. 06-4110)

Test	Specifications	Typical Property	Test Method
Hydroxyl Number	196 ± 10	196	1.029
pH, 1% in Water	6-7	6.8	1.047
Water, Wt. %	0.1 max.	0.03	1.016
Suspended Matter	Substantially Free	-	Visual

11-30-78
(Replaces 10-1-72)

This sheet is CONFIDENTIAL and should not be distributed outside Continental Oil Company

Spec. No. 06-4321

ALFONIC 1012-40 Alcohol Ethoxylate

Special Identification: Typical 40 ± 1 Wt. % Ethoxylate of ALFOL 1012-GB Alcohol (Spec. No. 06-4152)

Test	Specification	Typical Property	Test Method
Hydroxyl Number	190-205	201	1.029
Color, APHA	50 max.	5	1.025
pH, 1% in Water	6.0-8.0	6.8	1.047
Water, Wt. %	0.1 max.	0.03	1.016
Cloud Point, ml of Water	33 ± 4	33	1.056

7-15-77
(Replaces 5-01-77)

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Spec. No. 06-4320

ALFONIC 1012-60 Alcohol Ethoxylate

Special Identification: Typical 57 ± 2 weight percent ethoxylate of ALFOL 1012-HA (Spec. No. 06-4150)

Test	Specifications	Typical Property	Test Method
Hydroxyl Number	140-155	143	1.029
Color, APHA	50 max.	10	1.025
pH, 1% in Water	6.0-8.0	7.0	1.047
Water, Wt. %	0.1 max.	0.03	1.016
Cloud Point, 1% in Water, °C	32-38	34.0	1.049
Glycol, Wt. %	1.0 max.	0.8	1.034
Specific Gravity (25°/25°C)	0.972 ± 0.003	Passes	-

7-15-77
(Replaces 5-01-77)

FB-1 & 2 STG. TANKS

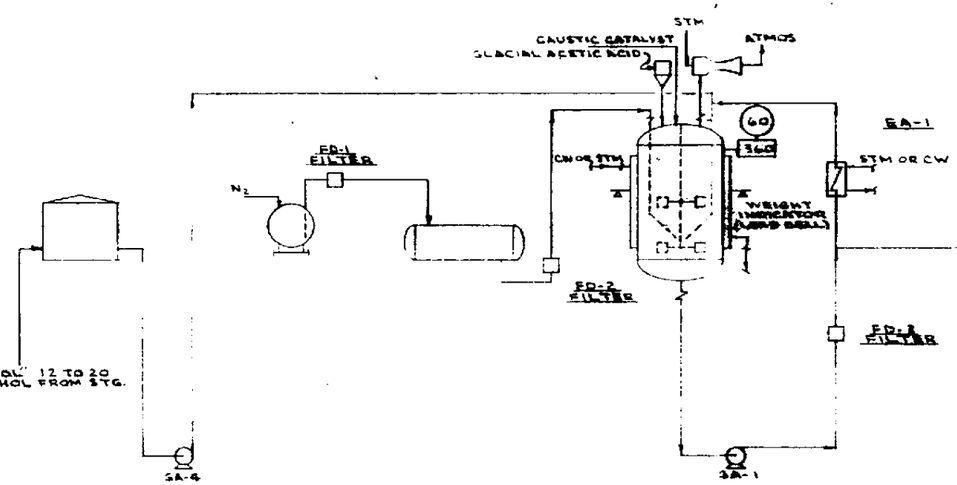
ETHYLENE OXIDE RAILROAD TANK CAR

EA-1 ETHYLENE OXIDE STG. DRUM

DC - ETHOXYLATION REA. TOR

FB-3 PRODUCT SUMDOWN TANK

FB-4 & 5 PRODUCT STG. TANKS



LEGEND

TEMP °F

PRESS. PSIG

CW COOLING WATER FROM PLANT COOLING TOWER

2	REV	1976 TRAINING PROGRAM	10-11-76	R	CDK
1		FOR TRAINING	1-3-69	DHC	

CONTINENTAL OIL COMPANY
ENGINEERING CENTER
PONCA CITY, OKLAHOMA

SIMPLIFIED PROCESS FLOW DIAGRAM
ETHOXYLATION UNIT
LAKE CHARLES "ALFOL" ALCOHOL PLANT
SCALE:

APPD: [illegible]
DATE: 10/14/26

No. CT-22-B

-281 S

SAL 00001133

SAL 0F0010C134

n-Paraffins Unit

PED ORIENTATION PROGRAM

n-PARAFFIN UNIT

LAKE CHARLES CHEMICAL PLANT

The n-Paraffin Unit was started up late in 1964 to produce straight-chained paraffin feedstock for the Baltimore plant's "NALKYLENE" Alkylate Unit and the Lake Charles LAB Plant (on stream, 1982). The n-Paraffin Unit utilizes Universal Oil Products' (UOP) proprietary "Unibon" and "Molex" technologies to recover normal paraffins from refinery kerosene at the rate of 330 MM pounds per year (beginning the first quarter of 1982). A portion of the raffinate remaining from the extraction step is hydrogenated in an auxiliary unit (originally a cyclohexane plant) to yield a low polynuclear aromatic solvent. The latter is used as the solvent in the "ALFOL" Alcohol Process and is also marketed. The remaining raffinate, significantly enhanced in armoatics content, is returned to Conoco's Lake Charles Refinery for use in making gasoline. The feedstocks include a 310°F-490°F virgin kerosene from the refinery and hydrogen from the ethylene unit.

PROCESS DESCRIPTION (See attached Overall Process Flow Diagram)

Light virgin kerosene from Conoco's Lake charles Refinery is supplemented with outside purchases of highly paraffinic kerosene. The mixture, containing 18-21 percent n-paraffins, is preheated and stripped of light hydrocarbons. After gravity removal of any contained water, the kerosene is elevated to 600°F in a fired heater. The hot kerosene is mixed with hydrogen and enters the Unibon reactor at 585-600°F and 775 psig. Olefinic materials are hydrogenated and sulfur-bearing compounds are reduced to hydrogen sulfide (among other things). The hot reactor effluent exchanges heat with the heater feed and is flashed to remove light gaseous hydrocarbons to fuel. The degassed stream is processed through a stabilizer tower to remove hydrocarbons boiling lower than decane. The stabilized kerosene is feedstock for the "Molex" process.

The "Molex" process uses 5A molecular sieves to selectively adsorb straight chain paraffins from a mixture of hydrocarbons. The sieves are loaded in a fixed bed adsorbent chamber which has a number of beds. Above each bed is an access line which is connected to a rotary distributing valve. The net streams to the adsorbent chamber (feed and desorbent) pass through the rotary valve and are directed to the appropriate access line. Simultaneously, the two product streams (extract and raffinate) are flowing from the chamber via their appropriate access lines through the rotary valve. After a predetermined interval, the valve automatically switches the four net flows to the next adjacent access line. This periodic change of net inlet and outlet points approximates the motion of the adsorbent past fixed inlet and outlet lines. All flows are continuous through the distributor, and the only motion is the periodic partial rotation of this valve.

The adsorbent chamber is divided into four zones:

PROCESS DESCRIPTION (CONTINUED)

Zone 1 - Normal paraffins are selectively adsorbed onto the sieves. The adsorbed paraffins displace desorbent and non-normal hydrocarbons from the sieves into the circulating liquid stream.

Zone 2 - This is the primary rectification zone in which the desorbtion of "non-normals" into the circulating liquid stream is completed.

Zone 3 - The adsorbed normal paraffins are displaced from the sieves by desorbent into the circulating liquid stream. The desorbent enters the chamber at the base of Zone 3, while the extract stream containing only normal paraffins and desorbent is removed from the chamber at the top of Zone 3.

Zone 4 - This is the secondary rectification zone in which the desorbent is displaced from the sieves with "non-normals" in the circulating liquid from the top of Zone 1. The circulating liquid leaving Zone 4 and entering Zone 3 contains only desorbent. The raffinate stream removed from the bottom of Zone 4 contains only "non-normal" hydrocarbons and desorbent.

The liquid in the adsorbent chamber is circulated from the top bed back to the bottom bed to complete the countercurrent contacting sequence. Since the required liquid flow is different for each of the four zones, the circulating pump is programmed to change the flow rate each time a new zone is switched from the top bed to the bottom bed.

The raffinate and extract streams leaving the sieve chamber each contain desorbent. These two streams are routed through the rotary valve to their respective fractionation columns. Normal paraffin product and raffinate are produced as bottoms from their respective columns. The overhead desorbent streams are recovered and returned to the adsorbtion chamber. The n-paraffin recovery is approximately 95 percent, with a purity of 99.1 percent. It is fractionated in a tower at Conoco's Lake Charles Refinery into a C_{10}-C_{12} cut and a C_{12}-C_{16} cut. Both are shipped via tanker to Baltimore. The C_{10}-C_{12} cut is used to make N-500 "NALKYLENE" alkylate. The C_{13}+ cut is fractionated into C_{13}-C_{14} and C_{14}-C_{16} cuts. The former is used to make N-600 "NALKYLENE" alkylate while the latter is sold.

A portion of the raffinate is charged to the "Solvent Unit," which is actually the reaction portion of a cyclohexane unit built in 1963-1964. The raffinate is subjected to a severe hydrotreating over catalyst to saturate polynuclear aromatice (PNA). This qualifies the solvent for use in the "ALFOL" Alcohol Process and other external solvent uses subject to the FDA's 50 ppb limit on PNA.

PRODUCTS AND BY-PRODUCTS

C_{10}-C_{12} n-Paraffins	- To Baltimore Chemical Plant and LAB Plant
C_{13}-C_{16} n-Paraffins	- To Baltimore Chemical Plant, LAB Plant, and Sales
LPA Solvent	- To "ALFOL" Alcohol Unit and Sales
Fuel Gas	- Used in Lake Charles Chemical Plant
Aromatic-Rich Raffinate	- Returned to Lake Charles Refinery

This sheet is CONFIDENTIAL and should not be distributed outside Continental Oil Company.

Spec. No. 06-3000

CONOCO C_{10}-C_{12} Normal Paraffins

Special Identification: C_{11} Average n-Paraffins

Test	Specification	Typical Property	Test Method
Total n-Paraffins, Wt. %	97.5 min.	97.7	1.072
Homolog Distribution, Wt. %	-	-	1.053
C_9 and Lower	2.0 max.	0.4	-
C_{10}	25.0 max.	18.3	-
C_{11}	-	38.9	-
$C_{10} + C_{11}$	50.0 min.	-	-
C_{12}	25.0 min.	33.9	-
$C_{10} + C_{11} + C_{12}$	85.0 min.	-	-
C_{13}	15.0 max.	8.0	-
C_{14} and Higher	2.0 max.	0.5	-
Aromatics, %	1.0 max.	0.75	1.036
Bromine Number	0.2 max.	0.01	ASTM D-1491-60
Color, Saybolt	25+ min.	30+	1.011
Molecular Weight	-	160	1.053
Pounds/Gallon, 60°F	-	6.22	

3-1-79
(Replaces 4-30-76)

SAL 000010137

Spec. No. 06-3005

CONOCO C11-C13 Normal Paraffins

Test	Specification	Test Method
Total N-Paraffins, Wt. %	97.0 Min	1.072
Homolog Distribution, Wt. %		
C10 and Lower	1.0 Max	1.053
C11		
C12	Balance	
C13		
C14 and heavier	1.0 Max	
Aromatics, Wt. %	0.8 Max	1.036
ASTM Distillation (Typical)		ASTM D-86
1 BP °F	390	
5%	400	
50%	410	
95%	445	
EP	450	
API at 60°F (Typical)	56	ASTM D-287
Sulfur, Wt. ppm	1 Max	ASTM D-1266
Nitrogen, Wt. ppm	1 Max	ASTM D-148
Carbonyl No., as c=o, ppm	100.0 Max	1.026
Bromine Number	0.02 Max	1.012

10-25-76
(Replaces 10-17-75)

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Spec. No. 06-3012

CONOCO C_{12}-C_{16} Paraffins

Special Identification: C_{13} Average n-Paraffins

Test	Specification	Typical Property	Test Method
Total n-Paraffins, Wt. %	97.0 min.	97.3	1.072
Homolog Distribution, Wt. %			1.055
C_{11} and Lower	1.0 max.	0.5	
C_{12}	8-25	11.0	
C_{13}	-	53.0	
C_{14}	-	27.0	
C_{15}	-	7.0	
C_{16}^{+}	-	1.5	
Aromatics, Wt. %	1.5 max.	0.9	1.036
Bromine Number	0.2 max.	0.02	1.012
Color, Saybolt	-	15+	1.011

1-21-77
(Replaces 4-30-76)

This sheet is CONFIDENTIAL and should not be distributed outside Continental Oil Company

Spec. No. 06-4600

CONOCO LPA SOLVENT

Special Identification: An aliphatic hydrocarbon with a low polynuclear aromatic hydrocarbon content

Test	Specifications	Typical Property	Test Method
Gravity API		44	ASTM-D-287
Distillation, °F			ASTM-D-86
IBP	350 min.	370	
10% evaporated		395	
20% evaporated		405	
50% evaporated		430	
90% evaporated		475	
95% evaporated		480	
End Point	530 max.	518	
Sulfur, ppm by Wt.		<1	ASTM-D-1266
Aniline Point, °F		160	ASTM-D-611
Aromatics, Wt. %	1.0 max,	0.10	1.036
Flash Point, P.M., °F	140 min.	144	ASTM-D-93
Particulate Matter, mg/gal.		2	ASTM-D-2276
Color, Saybolt	+25 min.	+30	ASTM-D-156
Nitrogen, ppm		<1	ASTM-E-148
Bromine Index		<200	ASTM-D-2710
Carbonyl, as C=O, ppm		10	1.026
Kauri Butanol Value		31.5	ASTM-D-1133
Water, ppm by Wt.	100 max.	20	1.016
UV Absorbence	Passes Test	Passes Test	(1)

(1) Test method to satisfy FDA requirements--CFR 121.1182 and CRF 121.2594

12-15-73
(Replaces 10-1-72)

SAL 000010140

U O P "UNIBON" PROCESS

RETURN TO SOLVENT UNIT

KEROSENE FEED FROM BATTERY UNITS

NATURAL GAS

SOLVENT REACTOR EFFLUENT

HYDROGEN

X-115 · X-1C · X-7 STM.

W-102 COLD STRIPPER

D-20 WATER KO DRUM

H-101 REACTOR HEATER · R-101 HYDROTREATER REACTOR · D-101 HI-PRES. SEPARATOR · D-105 WATER ACCUMULATOR · D-102 LO-PRES. SEPARATOR

R-102 SECONDARY REACTOR

NW-101 STABILIZER · H-102 STABILIZER REBOILER · D-103 ACCUMULATOR

X-101 · X-102 · X-103 · X-104 · X-105 · X-106 · X-116 · X-126 · X-110

C-101 · P-101 · P-102 · P-103 · P-105

OFFGAS TO FUEL · OFFGAS TO FUEL · UNSTABILIZED GASOLINE · OFFGAS TO FUEL

U O P MOLEX PROCESS

Mo-V1 HOT OIL DRUM · Mo-V2 MOLEX ADSORBENT CHAMBER · ROTARY VALVE · Mo-H1 DESORBER & HOT OIL HEATER · Mo-V3 RAFFINATE COLUMN · Mo-H2 REBOILER · Mo-V4 RECOVER · Mo-V5 EXTRACT COLUMN · Mo-H3 REBOILER · Mo-V6 RECEIVER · Mo-V20 DESORBENT SPLITTER · Mo-H1 DESORBER & HOT OIL HEATER · Mo-V12 RECEIVER

Mo-E8 · Mo-E11 · X-10 · X-14 · X-3 · X-15 · E-2 · E-3 · E-4 · E-5 · E-6 · E-8 & E-12

FEED · RAFFINATE · EXTRACT · DESORBENT · FLUSH

P-1 · P-2 · P-7 · P-6 · P-4 & 5 · P-8 · P-51 · P-3 · P-50

RAFFINATE TO REFINERY · FEED TO SOLVENT UNIT · EXTRACT TO STORAGE

ISU	DATE	DESCRIPTION	BY	CKD	APD
1	11-27-5	TRAINING FLOW DIAGRAM			

CONTINENTAL OIL COMPANY
ENGINEERING CENTER
PONCA CITY, OKLAHOMA

OVERALL PROCESS FLOW DIAGRAM OF PARAFFIN LAKE CHARLES CHEMICAL PLANT

APPD:
DATE:

NO. CT-21-D

Methyl Chloride Unit

SAL 0C0C10142

PED ORIENTATION PROGRAM

METHYL CHLORIDE UNIT

LAKE CHARLES CHEMICAL PLANT

The methyl chloride plant was built at Westlake in 1961 as a joint 50/50 venture of Conoco and Ancon Chemical. Conoco operated the plant and Ansul marketed the product. Design capacity of the Reactor was 20 MM pounds per year. Design capacity of downstream equipment was 40 MM pounds per year. Conoco and Ancon did the process design based on pilot plant data from Ancon. Blow-Knox built the plant. Shortly after startup, the plant operated at 28 MM pounds per year. Several changes and additions were completed prior to 1966 based on a design by PED which increased the capacity to 55 MM pounds per year. Near the end of 1966, a design was issued by PED to expand the plant capacity to 78 MM pounds per year. The plant was expanded to 98 MM pounds per year capacity in 1970.

In 1972, Conoco acquired full ownership of the methyl chloride plant in return for some concessions to Ancon. Another expansion was completed late in 1973 to raise production capability to 112 MM pounds per year.

GENERAL DESCRIPTION

Methanol and HCl are reacted at approximately 320°F and 22 psig in the presence of 60-70 percent ZnCl solution catalyst to yield methyl chloride and water as shown in the following reaction:

$$CH_3OH + HCl \xrightarrow[320°F]{ZnCl} CH_3Cl + H_2O$$

Theoretical conversion for CH_3OH and HCl is approximately 97 to 98 percent at these conditions. Actual reaction conversions typically run above 94 percent for HCl and approximately 89 percent for methanol. Overall plant yields for both HCl and methanol are 94 percent. The methanol recovery unit recycle raises the overall methanol yield above reaction conversion.

An undesirable side reaction also occurs which produces dimethyl ether. This tends to reduce methanol conversion to methyl chloride.

$$2CH_3OH \longrightarrow CH_3OCH_3 + H_2O$$

Operating experience has shown that this reaction can be minimized by limiting the reaction temperature to under 350°F and feeding a slight excess of HCl. Typical mol feed ratios are 1.05-1.10 HCl to methanol.

PROCESS DESCRIPTION

Methanol is partially vaporized in the preheaters and mixed with anhydrous HCl vapor as the reactor feed. The HCl-methanol mixture is sparged through zinc chloride catalyst solution and reacts to form methyl chloride and water at approximately 320°F and 22 psig. Some dimethyl ether is also produced in a side reaction.

The reactor effluent vapor is condensed at 85°F. The vapor-liquid mixture is separated in the vapor-liquid separator. The liquid HCl-methanol solution is fed to the methanol recovery column, and the impure methyl chloride vapor enters the brine scrubbers.

Methanol in the feed to the methanol recovery column (approximately 9 percent) is separated by steam distillation from the HCl solution and taken overhead at a concentration of approximately 80 percent. The recovered methanol is recycled to the reactors. The HCl solution bottoms is used to preheat the column feed and is then neutralized in an oyster shell pit or with waste caustic before discharging to settling ponds.

The vapor mixture to the brine-caustic scrubbers is mostly methyl chloride with about 2 percent dimethyl ether and small amounts of water and HCl vapor. Inerts such as nitrogen and CO_2 are also present. The brine-caustic scrubbers neutralize all of the HCl. Small amounts of methyl chloride product liquid are flashed into the scrubbers as a direct contact refrigerant to counteract the heat of reaction between HCl and NaOH. Brine-caustic is circulated from the brine-caustic receivers to the scrubbers which are packed with polypropylene intalox saddles. Fresh brine-caustic is metered into the second brine-caustic receiver, and spent brine-caustic is discarded from the first brine-caustic receiver.

The vapor flows through a knockout pot before entering the sulfuric acid scrubbers. This prevents entraining caustic solution into the acid scrubbers during upset operation.

The sulfuric acid scrubbers remove dimethyl ether and water from the methyl chloride stream. Methyl chloride refrigerant is flashed into each scrubber to compensate for heats of solution and lower the temperature to 50°F out the last scrubber. Acid is circulated from the acid receivers to the scrubbers which are also packed with polypropylene intalox saddles. Fresh 93 percent H_2SO_4 is metered into the last acid receiver and spent acid removed from the first acid receiver goes to storage.

The methyl chloride flows through a knockout pot to prevent any sulfuric acid from entering the carbon bed or compressor suction. The carbon bed removes impurities which would foul the compressor cylinders. The methyl chloride is compressed from 10 to 165 psig in the two-stage reciprocating lubricated compressors and is cooled to 120°F in the aftercoolers. Oil from the compressor cylinders, along with a small amount of condensed methyl chloride, is separated from the main process vapor stream in the oil knockout pot. This material goes to slop storage and is recycled to the process.

PROCESS DESCRIPTION (CONTINUED)

The methyl chloride vapor is condensed at 100°F and 160 psig. Most of the CO_2 also condenses in the methyl chloride stream. The condenser vent gas containing methyl chloride, nitrogen, and some CO_2 goes to a vent gas chiller which uses methyl chloride as a refrigerant to condense most of the remaining methyl chloride before venting the gas to the atmosphere at 20°F. The refrigerant methyl chloride is recycled to the compressors.

The methyl chloride product containing CO_2 goes to intermediate storage where it is pumped to the CO_2 stripper. Heat for stripping is furnished by a steam reboiler. The CO_2-rich vapor is taken overhead and goes to an overhead condenser similar to the vent gas chiller. Most of the methyl chloride in the gas stream is condensed and recovered before venting the CO_2 stream at 40°F. As in the vent chiller, methyl chloride is used as a refrigerant and is recycled to the compressors.

The purified methyl chloride product (stripper bottoms) is cooled to 100°F in the product cooler and flows to product storage where it is loaded into tank cars.

PRODUCT

Approximately 500 MM pounds of methyl chloride are produced per year in the U.S. However, 300 MM pounds are for captive requirements, and the other 200 MM pounds are marketed. Conoco market all of their methyl chloride making it the supplier of 50 percent of all methyl chloride available for sale in the U.S. Conoco are the only true marketer since the other 100 MM pounds sold are excess production from companies with captive uses.

The main uses of methyl chloride are as follows:

1. Silicones
2. Tetramethyl lead
3. Additive in synthetic rubber
4. Propellant gas in aerosol spray bombs
5. Weed killers

Silicones and tetramethyl lead combined account for approximately 75 percent of the methyl chloride end use. At present, usage for each product is about the same, but usage for silicones will increase while tetramethyl lead will decrease due to environmental regulations concerning lead content in gasoline.

The third important use for methyl chloride is in synthetic rubber production. All other uses of methyl chloride, such as weed killers and propellant in aerosol spary bombs, are minor.

METHYL CHLORIDE

Test	Specification	Typical Property	Test Method
Methyl Chloride, Wt %	99.95 Min	99.97	1.500
Dimethyl Ether, ppm Wt	20.0 Max	<5	1.500
Lower Boiling Compounds, ppm Wt	250.0 Max	50	1.500
Higher Boiling Compounds, ppm Wt	100.0 Max	<25	1.500
Water, ppm Wt	80.0 Max	35	1.500
Acidity, as HCl, ppm	10.0 Max	<4	1.501
Residue, ppm Wt	100.0 Max	<50	1.502

D-26 K.O. POT | R-1 REACTOR | R-2 REACTOR | R-3 REACTOR | R-4 REACTOR | D-9 MeCl VAPORIZER | W-1A SEPARATOR | W-2 BRINE SCRUBBER | W-3 BRINE SCRUBBER | W-1B BRINE SEPARATOR | W-4 ACID SCRUBBER | W-5 ACID SCRUBBER | W-6 ACID SCRUBBER | W-7 ACID SCRUBBER | D-11 CARBON BED

D-1 | D-2 | D-3 | D-4 | D-5 | D-6

VCM PLANT HCl
PPG HCl
OLIN HCl
METHANOL
METHYL CHLORIDE
STM
X-1
X-2
X-3
X-11
CWS
COND.
FRESH ACID
P-4
P-5
P-18
P-19
P-24
P-25
ATM
X-113
X-114
X-7
X-112
X-10
X-102
X-4
X-101
X-GG8
P-22
C-1 & C-2

W-112 CO_2 STRIPPER | D-120 K.O. DRUM | D-25 REFLUX DRUM | W-22 METHANOL STRIPPER | W-21 METHANOL STRIPPER | D-8 OIL K.O. POT | D-7 ACID K.O. POT

ISU	DATE	DESCRIPTION	BY	CKD	APD
1	11 3 76				

CONTINENTAL OIL COMPANY
ENGINEERING CENTER
PONCA CITY, OKLAHOMA

OVERALL PROCESS FLOW DIAGRAM
METHYL CHLORIDE UNIT
LAKE CHARLES CHEMICAL PLANT

APPD: [illegible]
DATE: [illegible]

NO. CT-25-D

SAL 000010147

Ethylene Unit

SAL 00001C148

PED ORIENTATION PROGRAM

ETHYLENE UNIT

LAKE CHARLES CHEMICAL PLANT

The Lummus-designed ethylene unit was brought on stream in February 1968 after a record-low startup period of nine days. The nameplate capacity was 500 MM pounds per year ethylene using ethane feedstock. Subsequent debottlenecking, including addition of a new cracking heater, has elevated capacity to 650 MM pounds per year using both ethane and propane feedstock. The technology employs Lummus' "SRT" high severity cracking heaters to maximize ethylene yield. The primary feedstock is ethane, although supplemental quantities of propane are charged during periods when ethane is in short supply.

PROCESS DESCRIPTION

The following is a brief description of the processing sequence as shown on the overall process flow diagram. Reaction chemistry is represented typically as follows:

$$C_2H_6 \xrightarrow{\text{Heat}} C_2H_4 + H_2$$

$$C_3H_8 \xrightarrow{\text{Heat}} C_2H_4 + CH_4$$

A. Cracking and Quench

Ethane and propylene-propane recycle are cracked in tubular heaters in the presence of dilution steam to an outlet temperature of 1,560°F. The heater effluents are cooled to 600°F in transfer line exchangers which generate high pressure steam at 650 psig.

The effluents from the transfer line exchangers are combined and directed to the quench tower. By direct contact water cooling, the greater part of the dilution steam and some heavier hydrocarbons are condensed. Net overhead vapor at 110°F flows to the compressor system. Quench water plus condensed steam is separated from the condensed hydrocarbons in a quench water surge drum which is operated at 180°F. The circulating hot water is used to preheat the ethane furnace feed and further cooled in the air-cooled quench water coolers and against cooling water. Condensed dilution steam is sent to the process water stripper, where it is stripped of dissolved gases and light hydrocarbons, vaporized against 200 psig steam, and reused as heater dilution steam.

B. Charge Compression and Acid Gas Removal

The quench tower overhead vapors are compressed in four centrifugal compressor stages to a pressure of 535 psig, with interstage cooling to 110°F. Between the third and fourth stages, the gas is treated for acid gas removal in the caustic and water wash tower. The fourth

B. Charge Compression and Acid Gas Removal (Continued)

stage discharge is cooled with water and with propylene refrigerant to 60°F. Liquid condensate is separated and the vapor is sent to the desiccant dryers.

Interstage hydrocarbon and water condensates from the first three stages are sent back to the quench water surge drum. Fourth stage condensate is recycled to the third stage discharge drum.

1. Drying and Feed Chilling

The final compressor discharge gas at 60°F and 525 psig is dried in packed bed dryers using activated alumina before passing to the low temperature recovery section. Three dryers are provided. Two are on stream in series, while the third is regenerated. Both regeneration and cooling of the dryers are accomplished using hydrogen-rich offgas on a once-through basis. A regeneration heater and a cooler are provided for the regeneration operation.

The dried gas at 60°F is progressively chilled and partially condensed, with condensate removal at -30°F, -95°F, -145°F, and -185°F. The remaining vapor is the hydrogen-rich offgas. The condensates are fed to the demethanizer. The chilling is achieved with propylene and ethylene refrigeration, vaporizing feed and recycle ethane and reheating hydrogen and methane offgas streams. An expander is used on the hydrogen-rich offgas to provide the lowest level refrigeration. The hydrogen-rich offgas and the methane-rich offgas are sent to fuel after reheating in the chilling train and against liquid propylene refrigerant.

2. Demethanization and Deethanization

The demethanizer, which operates at 445 psig, has a bottoms temperature of 31°F and an overhead temperature of -113°F. The column is reboiled with propylene refrigerant, and reflux is condensed with ethylene refrigerant. The demethanizer overhead is the methane offgas, which is sent to fuel after reheating in the chilling train and against liquid propylene refrigerant. The demethanizer bottoms flow to the deethanizer where C_2's are separated from the C_3 and heavier fraction. The deethanizer operates at 395 psig with 16°F and 182°F overhead and bottoms temperature, respectively. The reboiler utilizes low pressure steam and reflux is condensed with propylene refrigeration. The bottoms product is sent to the depropanizer and the overhead flows to the acetylene removal system.

PROCESS DESCRIPTION (CONTINUED)

B. Charge Compression and Acid Gas Removal (Continued)

3. Acetylene Removal and Ethylene Fractionation

The deethanizer overhead vapor, after feed-effluent exchange and preheat, is sent to the acetylene converter. Acetylene is hydrogenated over a palladium catalyst in the packed bed reactor. Hydrogen offgas after enrichment, methanation, and drying over molecular sieves is injected into the converter feed to provide the hydrogen requirements. Two vessels are provided, one is on stream while the other is on standby. A fired heater is provided for regeneration. The converter effluent is used to preheat the feed and sent to the ethylene fractionator.

The feed to the ethylene fractionator consists of ethylene, ethane, and unreacted hydrogen and methane from the acetylene removal system. The tower operates at 285 psig with overhead and bottoms temperatures of -20°F and 21°F, respectively. Condensing and reboiling are done by propylene refrigeration. The ethylene product is withdrawn as a side stream from the tower, and unreacted hydrogen and methane from the acetylene removal system are recycled from the reflux drum to the charge compressor. The liquid ethylene product is vaporized and delivered to the battery limits at 600 psig and 225 psig.

The fresh ethane feed is dried over molecular sieves, combined with the bottoms ethane product from the ethylene fractionator, and vaporized against demethanizer feed. The total ethane vapor stream is superheated against propylene refrigerant and circulating quench water and then charged to the cracking heaters.

4. Fractionation of Propane and Heavier Components

The depropanizer, operating at 160 psig with 77°F and 228°F overhead and bottoms temperatures, respectively, receives feed from the deethanizer bottoms. Steam and propylene are the reboiling and condensing mediums, respectively. The overhead vapor product is recycled to the cracking furnace.* The bottoms product containing C_4 and heavier is sent to the debutanizer.

The debutanizer operates at 65 psig with 119°F top and 249°F bottom temperatures. Cooling water is the reflux condensing medium, while steam is used for reboiling. The overhead liquid is the mixed C_4 product which is sent to battery limits, and the bottoms is the light aromatic distillate product which is cooled to 120°F and sent to battery limits.

*The C_3 overhead is now being sold as product and is not recycled to the furnaces.

PROCESS DESCRIPTION (CONTINUED)

B. Charge Compression and Acid Gas Removal (Continued)

5. Propylene Refrigeration

The propylene refrigeration system is a closed, multistage system using a centrifugal compressor. It provides refrigeration at four levels: -35°F, -5°F, 37°F, and 60°F. The compressor effluent is cooled and condensed against cooling water and subcooled against various produce and process streams. Interstage condensing at the various levels is obtained from reboiling the demethanizer and the ethylene fractionator and also from the vaporization of the liquid ethane feed and ethylene product.

6. Ethylene Refrigeration

The ethylene refrigeration system is a closed, multistage system using a centrifugal compressor. The ethylene system has three levels of refrigeration: -150°F, -100°F, and -65°F. The compressor effluent is desuperheated against propylene refrigerant and then condensed against the lowest level propylene refrigerant.

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Spec. No. 07-5100

ETHYLENE

Test	Specification	Test Method
Ethylene, Wt. %	99.8 min.	By difference
Other Hydrocarbons, Wt. %	0.2 max.	1.251
Propylene, ppm	65 max.	1.251
Acetylene, ppm	20 max.	1.252
Carbon Dioxide, ppm	350 max.	1.253
Carbon Monoxide, ppm	10 max.	1.254
Hydrogen, ppm	10 max.	1.255
Oxygen, ppm	5 max.	1.254
Sulfur, ppm	2 max.	1.256
Water, ppm	5 max.	-

12-15-73

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Spec. No. 07-5122

Propane-Propylene

Test	Specification	Typical Property	Test Method
Distribution			GC
C_4's and lower, wt.%	2.5 max.	1.2	
Propylene, wt.%	65 min.	79.5	
Propane, wt.%	35 max.	18.5	
C_4's and higher, wt.%	3 max.	0.8	
Sulfur, ppm wt.	10 max.	< 1	1.256

5-01-77

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Spec. No. 07-5142

BUTADIENE

Test	Specification	Typical Property	Test Method
Distribution			GC
C_3's and lower, wt.%	2.0 max.	1.2	
Butanes, wt.%		12.0	
Butenes, wt.%		14.0	
1,3 Butadiene, wt.%	65.0 min.	71.3	
1,2 Butadiene, wt.%		0.2	
C_4 Acetylenes, wt.%	2.0 max.	1.2	
C_5 and higher, wt.%	1.0 max.	0.1	
Peroxides, ppm as H_2O_2	10 max.	< 1	ASTM D-1022 MOD
Sulfur, ppm	10 max.	< 1	1.256
Antioxidant (TBC) ppm	=	100	LC-EU-8

5-01-77

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Spec. No. 07-5180

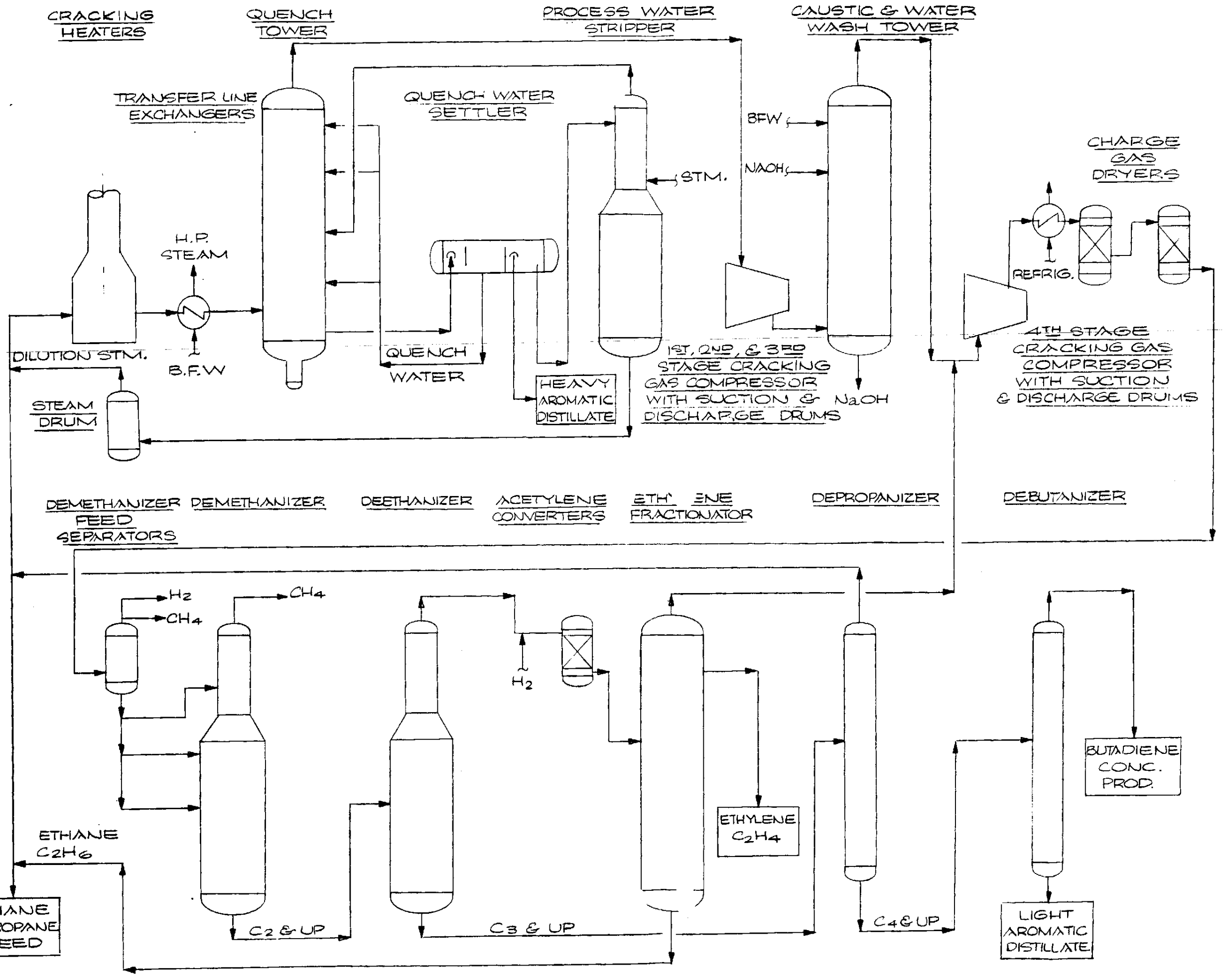
Light Aromatic Distillate

Test	Specification	Typical Property	Test Method
Benzene, Wt. %	40.0 min.	48.6	GC
C_4's and Lower, Wt. %	3.0 max.	1.5	GC
90% Evaporation Point, °F	400 max.	340	ASTM D-86

10-15-79

SAL 000010156

ETHYLENE UNIT AT LAKE CHARLES PLANT



SAL 000010157

DWG. NO. E5877-2A

BA-101-105 CRACKING HEATERS | DA-101 QUENCH TOWER | FA-108 QUENCH WATER SETTLER | DA-102 PROCESS WATER STRIPPER | FA-201 1ST STAGE SUCTION DRUM | FA-202 2ND STAGE SUCTION DRUM | FA-203 3RD STAGE SUCTION DRUM | FA-204 3RD STAGE DISCHARGE DRUM | DA-203 CAUSTIC & WATER WASH TOWER | EA-219 SURFACE CONDENSATE COOLER | F8-206 CAUSTIC DAY TANK | FA-207 4TH STAGE SUCTION DRUM | FA-208 4TH STAGE DISCHARGE DRUM | FA-209A B C CHARGE GAS DRYERS

EA-101A-105A | EA-101B-105B | EA-109 | EC-101 | EA-108 | EA-504 | FA-107 DILUTION STEAM DRUM | EA-111 | EA-112 | BLOWDOWN | GA-101 | EA-110 | GA-102 | GA-103 | PA-101 OIL FILTER | GA-104 | EA-107 | EA-201 | EA-202 | EA-203 | GB-201 1ST STAGE | GB-201 2ND STAGE | GB-201 3RD STAGE | GA-203 | CHARGE COMPRESSOR

HA-203 DEGASSING POT | EE 201 LINE MIXER | EA-204 | TO FLARE | FRESH CAUSTIC FROM B.L. | SURFACE COND. | SPENT CAUSTIC TO B.L. | GA-204 | GA-205 | EA-214 | EA-210 | BFW FROM DEAERATOR | GB-201 4TH STAGE | EA-213 | EA-212 | EA-211

EA-316 | EA-301 | EA-304 | EA-305 | EA-306 | EA-307 | EA-309 | EA-310 | EA-308 | FA-303 DEMETHANIZER FEED SEPARATOR NO. 3 | FA-304 DEMETHANIZER FEED SEPARATOR NO. 4 | FA-301 DEMETHANIZER FEED SEPARATOR NO. 1 | FA-302 DEMETHANIZER FEED SEPARATOR NO. 2 | FA-305 ETHANE SURGE DRUM | FA-306 ETHANE FEED DRYERS | ETHANE FEED FROM BATTERY LIMITS

DA-401 DEMETHANIZER | EA-401 | FA-401 DEMETHANIZER REFLUX DRUM | GA-401 | DA-402 DEETHANIZER | EA-403 | FA-402 DEETHANIZER REFLUX DRUM | FA-305 HYDROGEN DRYERS | GA-402 | EA-402 | EA-404 | EA-505 | GC-301 EXPANDER | METHANE RICH OFF GAS TO FUEL GAS DRUM | HYDROGEN RICH OFF GAS TO FUEL GAS DRUM

A&B ACETYL. CONVERTERS | DA-403 ETHYLENE FRACTIONATOR | FA-412 H.C. DRUM | EA-408 | EA-406 | EA-407 | EA-409 | EA-410 | FA-404 ETHYLENE FRACTIONATOR REFLUX DRUM | GA-403 | EA-315 | HA-302 WATER K.O. POT | EA-313 | EA-314 (FOR STARTUP ONLY) | FA-306 METHANATOR | EA-312 | EA-506 | HA-301 HYDROGEN SEPARATOR POT | FA-413 ETHYLENE PRODUCT DRUM | GA-404 | EA-411 | EA-412 | EA-424 | EA 503 | EA-502 | 225 PSIG ETHYLENE PROD. TO BATTERY LIMITS | 600 PSIG ETHYLENE PROD. TO BATTERY LIMITS

DA-404 DEPROPANIZER | EA-413 | FA-405 DEPROPANIZER REFLUX DRUM | GA-405 | EA-419 | EA-415 | GA-413 | LIGHT AROMATIC DISTILLATE TO BATTERY LIMITS

DA-407 DEBUTANIZER | EA-422 | FA-408 DEBUTANIZER REFLUX DRUM | GA-409 | EA-423 | BUTADIENE CONC. PROD. TO BATTERY LIMITS

HEAVY AROMATIC DISTILLATE TO BATTERY LIMITS

LIST OF CHANGES REV. AS INDICATED "C"
1. EA-403 AND EA-413;
2. STEAM AT EA-214 WAS OR WAS STM.
3. ADDED EA-213 & EA-303.
4. FA-206 AND FA-207.

LEGEND
CW - COOLING WATER
STM - STEAM
CSR - PROPYLENE REFRIGERANT
CER - ETHYLENE REFRIGERANT

5877	6	CUST 2-6		4	APPROVED FOR CONSTRUCTION								
5877	5	CWF 4-5		3	APPROVED FOR DESIGN								
5877	2	CUST 4-5		2	FOR APPROVAL								
3877	1	11-26		1	PRELIMINARY OCT 15, 1965								

ISSUE 2-6 62

THE LUMMUS COMPANY
NEW YORK, N.Y.
LUMMUS JOB NO. 3877 | CUSTOMER'S ORDER NO.

ETHYLENE PLANT
CONTINENTAL OIL COMPANY
LAKE CHARLES, LOUISIANA

OVERALL PROCESS FLOW DIAGRAM

SCALE | DWG. NO. E5877-2A-5

SAL 00001C15e

SAL 00001159

Lake Charles LAB Plant

PED ORIENTATION PROGRAM

LAB PLANT

LAKE CHARLES, LOUISIANA

Preface

The LAB plant is presently under construction and plant startup is scheduled for late 1982.

PED ORIENTATION PROGRAM

LAB PLANT

LAKE CHARLES, LOUISIANA

INTRODUCTION

The HF Detergent Alkylation Process is a catalytic process to alkylate benzene with linear olefins to form linear alkyl benzene. The linear alkylbenzenes produced from the C_{10}-C_{13} linear olefins are useful detergent intermediates and can be readily sulfonated (treatment with oleum or sulfur trioxide and neutralization with NaOH) to yield linear alkylbenzene sulfonates. These compounds constitute the "active" ingredients of many household detergents. They are surface active compounds (surfactants) which are combined with various builders (often inorganic salts) to make up a detergent formula. (See Table I).

During the 40's and 50's, the detergent market was primarily captured by dodecylbenzene (DDB), product formed by alkylation of benzene with propylene tetramer in a hard detergent alkylation unit. It was found, however, that the branched structure of the alkyl group was responsible for the poor biodegradability of the detergent and the linear alkylbenzenes (LAB) introduced in the early 60's have substantially replaced their branched counterparts. Some of the new alkylation units are designed for the dual purpose of taking care of present markets (hard detergent) and future markets (soft detergents); they can be charged with propylene tetramer or Pacol linear olefin and yield the respective alkylbenzenes.

Sodium linear alkylbenzene sulfonate (LAS) is a high quality, safe and biodegradable detergent which is produced at competitive costs. It is one of the most important sources of detergent and the world-wide annual production of LAB was 820,000 MT in 1973 while the production of DDB was 230,000 MT during the same period.

The first unit in a detergent complex is an n-paraffin extraction unit (like the UOP Molex Unit) which extracts n-paraffins from a kerosene feed. Pure n-paraffins are then dehydrogenated in the UOP Pacol Unit and the mixture of about 12 percent n-olefins and 88 percent n-paraffins is charged to the detergent alkylation unit. The unreacted normal paraffins are continuously recycled to the Pacol feed after going through the alkylation section while the n-olefins are completely alkylated in the alkylation reactors. It was found that adjusting the Pacol reactors to about 12 weight percent olefin conversion is the most economical way to operate the units in terms of product quality and operating costs.

PROCESS DESCRIPTION OF THE PACOL UNIT

The UOP Pacol* Process is a process to dehydrogenate high purity linear paraffins to their corresponding mono-olefins. An olefin-free stream from the process downstream of the Pacol Process is usually recycled to the Pacol Unit and combined with the fresh feed. This recycle stream may be the raffinate product from an Olex Unit, or the unreacted paraffins from an Alkylation Unit.

The Pacol Process Unit consists of a catalytic, fixed-bed reactor wherein a portion of the combined linear paraffin feed is dehydrogenated to the corresponding mono-olefins. The reaction is carried out in the presence of hydrogen at low pressure and moderately high temperatures. The feed to the downstream unit is from the product stripper in which light ends and water are taken overhead.

PACOL UNIT PROCESS VARIABLES

Reactions

As is true with other hydrogen-producing processes, the Pacol reactor outlet temperature will be lower than the inlet. The reactor conditions are adjusted to maintain a total linear olefin concentration in the reactor separator liquid effluent of about 11.0-12.5 weight percent, depending on the design factors for each particular unit. The selectivities are such that about 90 percent of the converted linear paraffins are mono-olefins. The remaining ten percent are mainly di-olefins and aromatics with lesser amounts of light ends, iso-paraffins and iso-olefins. The gas produced by the dehydrogenation reactor is a hydrogen-rich gas ranging from 99 to 76 mol percent H_2 over the length of the run. The double bond of the product mono-olefins is randomly distributed along the chain, with about ten percent in the alpha position.

There is no difficulty in operating at a lower total normal olefin concentration than design. Higher concentrations, however, should be avoided because of the excessive amounts of aromatics that would be formed.

At a given temperature, the lower carbon number hydrocarbons in each class are less reactive than the higher carbon number hydrocarbons of the same class. Naphthenes have a lower reactivity than any of the paraffins. The aromatics are essentially non-reactive, except for the small amount that may be hydrocracked. Thus, on startup and following each change in conversion rate in the reactor, the equilibrium concentrations will shift. When the conversion rate is increased, the recycle paraffin stream will slowly become enriched with the lower carbon number paraffins. As the conversion rate is decreased, the recycle stream will become enriched with the higher carbon number paraffins, with the resulting reduction in the lower carbon number paraffins.

*Registered Trademark

PACOL UNIT PROCESS VARIABLES (CONTINUED)

Hydrogen Recycle Ratio (Continued)

The reason that low a hydrogen:hydrocarbon ratio is bad is that the rate of coke formation increases as the ratio decreases below 8.0. Above 8.0, however, the rate of coke formation is not a function of hydrogen:hydrocarbon ratio. The big disadvantage in operating at a ratio too much above 8.0 is that a higher reactor temperature is required, apparently because of the higher velocities in the catalyst bed. This higher temperature results in shorter runs as well as poorer selectivity.

The recommended operating rage is 6.0 to 6.5.

Water Injection

Water injection facilities are provided to maintain 2,000 weight ppm water in the combined hydrocarbon feed to the catalyst. More water than this has little or no effect. Less water, however, tends to decrease catalyst stability. The most consequential result of insufficient water is that the lost catalyst stability cannot be regained by additional water injection. Therefore, it is very important to assure that the proper water injection rate is always maintained.

PROCESS DESCRIPTION OF THE SOFT DETERGENT ALKYLATION UNIT

The n-olefins produced in the Pacol Unit are alkylated with benzene in the alkylation reactor in the presence of an HF acid catalyst to yield linear alkylbenzene. The Friedel-Crafts reaction proceeds through an intermediary carbonium ion created on the original olefin by migration of the two electrons of the double bond induced by the electrophylic catalyst. The catalyst is liquid hydrofluoric acid and is continuously circulated in the system. The reaction is carried through a two-stage reactor settler system. The reactor effluent proceeds to a series of fractionation columns where the various compounds are separated, the reagents being recycled and the products sent to storage. The first column is the HF stripper, where HF is removed overhead and HF-free components flow off the bottom to the "non-acid" part of the unit. The benzene is then recycled to the alkylation reactor from the overhead of the benzene column and the n-paraffins are recycled to the Pacol Unit from the paraffin column. The final rerun column splits out the alkylate product into two streams, the LAB overhead and the heavy alkylate bottoms. The n-paraffins are alumina treated for removal of combined fluorides before being recycled to the Pacol Unit. The purity of the HF acid is maintained high by continuously sending a slipstream to an acid regenerator column and returning the regenerated acid into the system.

The product composition and quality is a function of various operating parameters which will be discussed in more detail in the following pages.

Refer to Figure 1 for a simplified flow diagram of the process.

PROCESS DESCRIPTION OF THE SOFT DETERGENT ALKYLATION UNIT (CONTINUED)

Effluent Control

Hydrofluoric acid handling and disposal is a delicate operation and the plant is designed with safety of both equipment and personnel as a major objective. All effluents containing acid or trace acid are treated before leaving the unit.

Some Chemistry

The main reaction is the alkylation of benzene with the straight chain olefins to yield a linear alkylbenzene:

1. R-C-C-C=C-C-R+  $\xrightarrow{HF}$ R-C-C-C-C-C-R 

At the existing conditions, some side reactions take place like the formation of dialkylbenzene.

2. 2R-C-C-C=C-R+  $\xrightarrow{HF}$ R-C-C-C-C-C-R  R-C-C-C-C-C-R

or the formation of diphenylalkanes

3. R-C=C-C=C-C-R+2  $\xrightarrow{HF}$ R-C-C-C-C-C-C-R

or the formation of heavier components by combination of the above reactions.

Quality Control of the Products

The most desired product is the linear alkylbenzene described in reaction 1 and the unit is operated such that more than 90 percent of the crude alkylate produced will be LAB. The linear alkylbenzene is fractionated from the heavy alkylates in the rerun column, where one can adjust the split to get the desired product purity. Typically, the rerun column overhead will contain about 97 percent alkylbenzene with 3 percent indans and tetralins and 94 percent of linear alkyl benzene (see Table II). The rerun column bottoms will contain five to twenty percent LAB, 30 to 40 percent dialkylbenzenes, 30 to 40 percent diphenylalkane and other heavier components.

The linear olefins produced in the Pacol Unit are a mixture of the various isomers in similar proportion except for the olefins which are less than ten percent of the total isomers. (In the case of a chain of even carbon number, the olefin with the double bond in the central position will be in lower concentration because there is only one possibility to generate it.)

PROCESS DESCRIPTION OF THE SOFT DETERGENT ALKYLATION UNIT (CONTINUED)

Quality Control of the Products (Continued)

The alkylation reaction will respect the above distribution, except that the 1 phenyl-n alkane will not exist. Because of the lower content of alpha olefins, the 2 phenyl n-alkane will exist in lower proportion but all the other isomers will be about equally distributed.

Because of the higher foaming properties of the 2 phenyl alkane, some customers will require upper limits in its concentration and the UOP detergent process will meet them because of the preceding considerations. It should also be noted that because the Pacol process produces pure normal olefins with little branched chain olefins, the detergent alkylate produced has excellent biodegradability and compares favorably with the competition's detergent quality.

Another important quality control element is the Bromine Index of the LAB product which is an indication of the degree of unsaturation and condensed aromatic rings. Since these components will discolor the sulfonated LAB, a maximum bromine index of ten to twenty is usually specified for the LAB product. The olefins and polyaromatic compounds dissolve to some extent in HF and will be removed as HF regenerator bottoms as long as HF acid is not saturated with aromatics. The Bromine Index of the LAB is usually a function of the efficiency of the regeneration in the HF regenerator. The Bromine Index of the overhead product is also a function of the split performed in the rerun column.

The di-alkylbenzene component of the heavy alkylates produced from the bottom of the rerun column have been used to produce lube oil detergents. Their color is an important specification and usually is directly related to the performance of the HF regenerator. The viscosity must also be kept high to be used as lube oil additives.

ALKYLATION UNIT PROCESS VARIABLES

HF/Hydrocarbon

Most units are designed with an HF to hydrocarbon volume ratio of two. This normally provides sufficient acid to obtain the expected yield structure and product quality. Increasing the ratio should theoretically improve the yield structure but this effect has not been conclusively observed in commercial units. Most commercial units operate at HF/HCBN ratios of 1.5 to 2.0, and the lower ratios have been obtained at higher than design throughputs which probably improved the mixing. Reducing the HF circulation does not significantly affect the utilities consumption; it does, however, tend to increase the bromine index of the final product.

ALKYLATION UNIT PROCESS VARIABLES (CONTINUED)

Benzene/Olefin

Most units are designed with a benzene to olefin mol ratio of ten. This normally provides enough olefin dilution to obtain the expected yield structure.

Above a ratio of ten, however, the improvement is usually insignificant. Decreasing the ratio increases the formation of di-alkylbenzene and heavy alkylate. Most commercial units operate with a benzene/olefin ratio between five and twelve. The lower ratios generally produce a yield structure that would not meet a typical UOP guarantee.

Reducing the benzene circulation saves a large amount of heat because all the circulated benzene is a fractionator overhead product. Some trade-off in lower yield for lower utilities may be economically justified in most plants.

Temperature

The reactors are usually operated around 100°F. Since the soft detergent is more thermally stable than the hard detergent, the temperature of the second reactor can sometimes be increased to 140°F to eliminate the last traces of olefins. However, the reaction is very rapid and is usually complete in the first reactor; so the observed effect of temperature is minimal.

Pressure

The pressure has essentially no effect on the reaction. It should be kept high enough to keep all components in the liquid phase.

HF Regenerator Feed/LAB Product

HF has the property to selectively extract some heavy aromatic byproducts of the alkylation reaction which would otherwise wind up in the finished products. The HF regenerator is the recovery column of this extraction process, where pure solvent is recovered in the column overhead and the extract (in this case, the heavy polyaromatics) found in the column bottom,

This extraction can be viewed as a two-stage, countercurrent extraction, the two stages corresponding to the two reactors. To set up this counter-current system, the regenerated acid from the regenerator overhead is directed to the inlet of the second-stage reactor while a slipstream of acid from the second-stage settler is recycled to the first-stage reactor. To complete the loop, a slipstream of acid from the first-stage system is fed to the HF regenerator. To maintain a constant acid inventory in both first and second stages, the three flows described above are equal.

ALKYLATION UNIT PROCESS VARIABLES (CONTINUED)

HF Regenerator Feed/LAB Product (Continued)

The extent of this circulation determines the purity of the HF. As in any solvent system, higher extract loads in the solvent reduce the effectiveness of the solvent. Thus, the purer the acid, the better the alkylate quality. These polyaromatics (improperly called polymers) found in the HF regenerator bottoms are highly unsaturated compounds which would otherwise wind up in the rerun column both in the overhead product and in the bottom product.

In the overhead they will increase the bromine index of the LAB, and in the bottom they will color the heavy alkylates beyond acceptable specifications. One way to characterize these unsaturated compounds is to assign them a "J" number. The "J" number of a product is a measure of the degree of unsaturation of this product. For example, the "J" number of benzene is 6. These "polymers" have a "J" number as high as "J=18".

If the HF regenerator performs satisfactorily, the amount of feed fed to it will determine the purity of the circulating HF. This number is usually logged in as HF regenerator feed/LAB product, and is usually around 0.5 to 1. It is a very important parameter to control the quality of the final products.

If the acid is saturated with polyaromatics, its color will be dark. The color of the acid is a good indication of its purity - the lighter the color, the purer the acid. The color of the acid can range anywhere between almost white to dark brown and can take all the shades of yellow and brown. Looking at the acid color in the HF regenerator overhead receiver, one can tell immediately if the regenerator is functioning well. The purity of the circulation acid is usually maintained above 95 percent, with about .5 percent residual water and the rest heavy hydrocarbons.

Another feature of the HF regenerator is that it removes all water present in the system. In a distillation column, water and hydrofluoric acid form an azeotrope containing about 40 percent acid and 60 percent water. This azeotrope is normally called CBM (constant boiling mixture) because of its obvious properties. Any water present in the system will come off the regenerator bottoms in the form of CBM. The HF regenerator bottoms will be drained batchwise to a polymer surge drum where CBM will separate from the hydrocarbon phase. CBM is a very corrosive substance and will corrode much more readily than pure HF. It is to be noted that in normal circumstances the production of CBM should be minimal since there is very little water entering the system; the n-paraffins are being stripped in the Pacol stripper and the benzene is dried in the benzene drying column. Any excessive CBM made is the result of a faulty operation, most probably a leak somewhere in the startup. Because of the corrosive properties of CBM, any excessive CBM formation should be checked by closely monitoring the water entering the system. The regenerator will eventually move the water, but it is not specifically designed to separate water from HF by distillation; and, in any case, elimination of a given volume of water will cause a loss of HF of almost the same amount.

ALKYLATION UNIT PROCESS VARIABLES (CONTINUED)

Water Injection

Water is not a true variable because it does not affect yields or rates. The acid settlers are designed to separate the acid and hydrocarbon phases when the water content of the acid is about 0.3 weight percent. Increasing the water above 0.5 weight percent will increase the corrosion rates in the HF section. Reducing the water below 0.1 weight percent will increase the time required to separate the acid from the hydrocarbon and create an emulsion. At extremely low water contents, the HF-hydrocarbon mixture will require about one week to separate.

TABLE I

STANDARD ANIONIC SURFACTANT FORMULATION

PED ORIENTATION PROGRAM

LAB PLANT

LAKE CHARLES, LOUISIANA

Components	Weight, %
Sodium Alkylbenzene Sulfonate	20
Sodium Tripolyphosphate	40
Sodium Sulfate	34
Sodium Silicate	5
Carboxymethylcellulose	1

TABLE II

TYPICAL PROPERTIES OF LINEAR DETERGENT ALKYLATE

PED ORIENTATION PROGRAM

LAB PLANT

LAKE CHARLES, LOUISIANA

Bromine Number	0.01
Saybolt Color	+30
Unsulfonatable Content, Weight, %	1.0
Alkylbenzene Content, Weight, %	97.4
Normal Alkylbenzene, Weight, %	94.0
Biodegradability, %	
ASTM D 2667	98.1
O.E.C.D.	95.4
2-Phenyl Isomer, Weight, %	20.0
Paraffin, Weight, %	0.1
Saybolt Color of the Sodium Alkylbenzene Sulfonate	+26
Water, Weight, %	0.01
Doctor Test	Negative
Average Molecular Weight	240
Specific Gravity at 15.5°C	0.8612
Refractive Index, N_D^{20}	1.4837
Flash Point (ASTM D 93), °C	138
Distillation (ASTM D 86), °C	
IBP	281
10 Vol, %	286
30 Vol, %	288
50 Vol, %	290
70 Vol, %	293
90 Vol, %	298
95 Vol, %	302
EP	309

conoco

NALKYLENE 550L LINEAR DETERGENT ALKYLATE

TENTATIVE SPECIFICATIONS

Properties	Specification	Typical	Test Method
Alkylate Homolog Dist. wt.%			1.090
C_{10}	5-15	8.1	
C_{11}	—	27.9	
C_{10} + C_{11}	30-50	36.0	
C_{12}	—	37.7	
C_{13}	—	25.8	
C_{14}	10 max	0.5	
C_{13} + C_{14}	30 max	26.3	
C_{15} + higher	2 max	0.5	
2-Phenyl isomer, wt.%	12-22	17.0	1.090
Total Material < C_{10} LAB, wt.%	0.5 max	0.2	1.090
Average Molecular Weight	240-248	242	1.090
Bromine Number	0.01 max	<0.01	ASTM D 2710-72
Saybolt Color	29 min	30 +	1.011
Completeness of Sulfonation, %	98 min	98.5	1.059
Doctor Test	Negative	Negative	ASTM D-484
Specific Gravity (60°F/60°F)	0.860-0.870	0.8620	1.023
Water, wt.%	0.1 max	0.004	1.016
Color of Na Sulfonate, Klett (5% AI)(oleum)	60 max	50	1.042
Biodegradability	Must pass	Pass	SDA Test

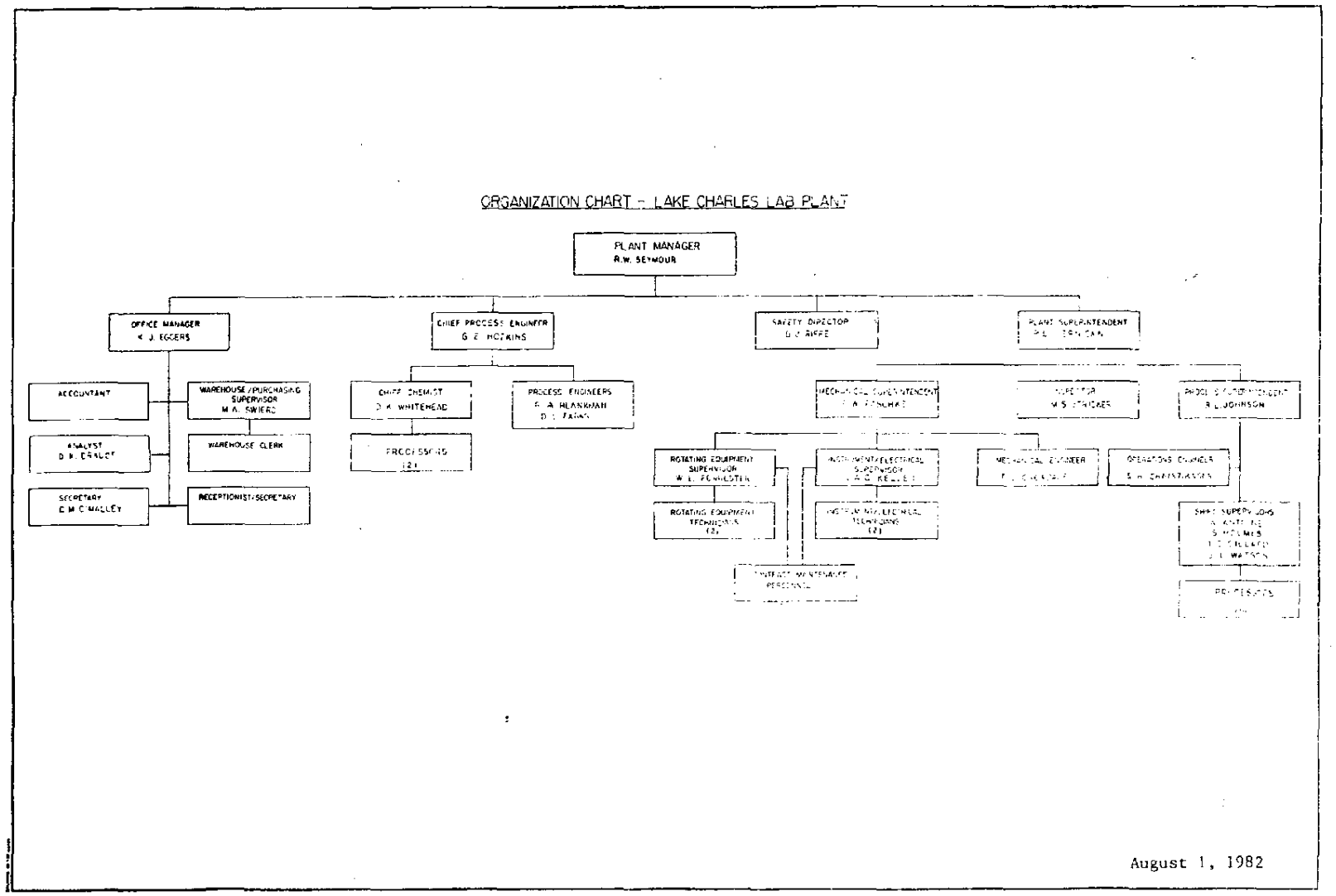
CONOCO

NALKYLENE 600L LINEAR DETERGENT ALKYLATE

TENTATIVE SPECIFICATIONS

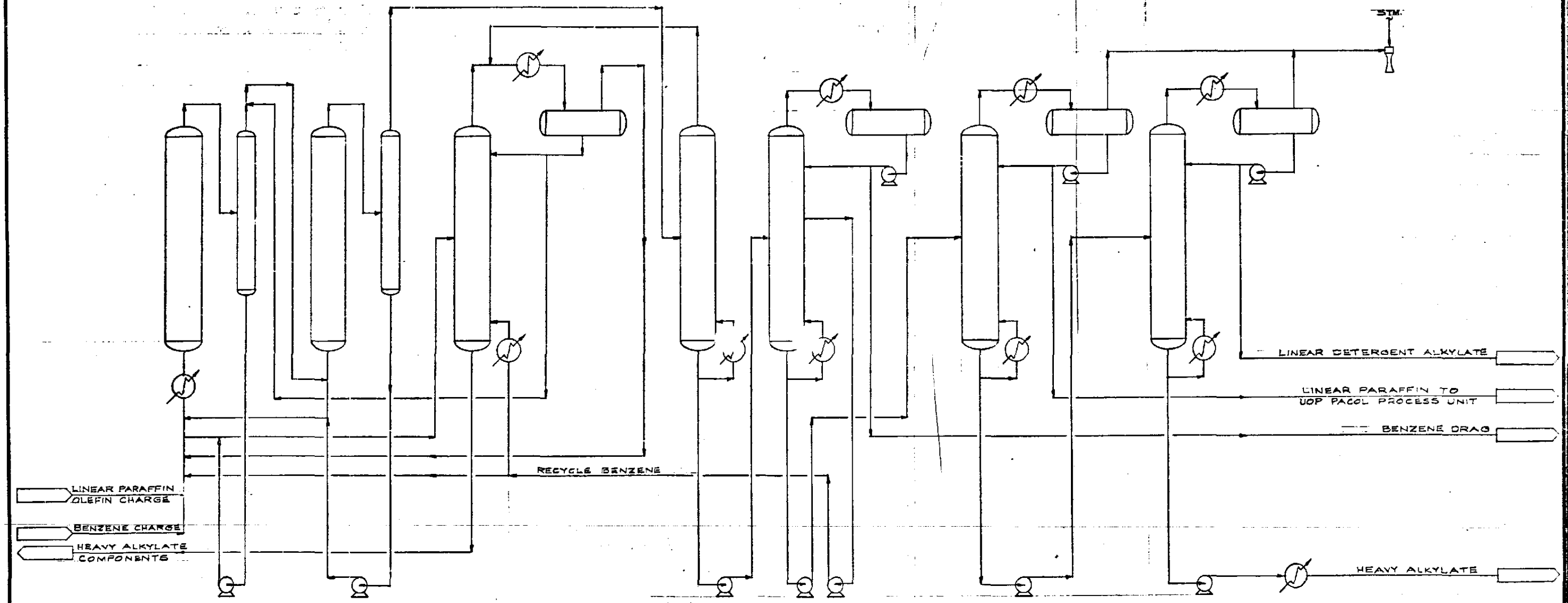
Properties	Specification	Typical	Test Method
Homolog Distribution, Wt.%			1.090
C_{10}	2 max	nil	
C_{11}	—	0.2	
$C_{10} + C_{11}$	5 max	0.2	
C_{12}	—	14.2	
C_{13}	—	51.1	
C_{14}	45 max	31.3	
$C_{13} + C_{14}$	70-90	82.4	
C_{15} + higher	5 max	3.2	
2-Phenyl Isomer, Wt.%	10-20	15.0	1.090
Total Material < C_{10} LAB, Wt.%	0.5 max	0.1	1.090
Average Molecular Weight	258-266	263	1.090
Bromine Number	0.01 max	<0.01	1.091
Saybolt Color	29 min	30 +	1.011
Completeness of Sulfonation, %	97.5 min	98	1.059
Doctor Test	Negative	Negative	ASTM D-484
Specific Gravity (60°F/60°F)	0.858-0.868	0.8600	1.023
Water, Wt.%	0.1 max	0.004	1.016
Color of Na Sulfonate, Klett (5% AI)(oleum)	90 max	75	1.042
Biodegradability	Must pass	Pass	SDA Test

Revision 06-01-82



SAL 00001173

FIRST STAGE REACTOR | SETTLER | SECOND STAGE REACTOR | SETTLER | HF REGENERATOR | SETTLER | HF STRIPPER | BENZENE COLUMN | RECEIVER | PARAFFIN COLUMN | RECEIVER | RERUN COLUMN | RECEIVER



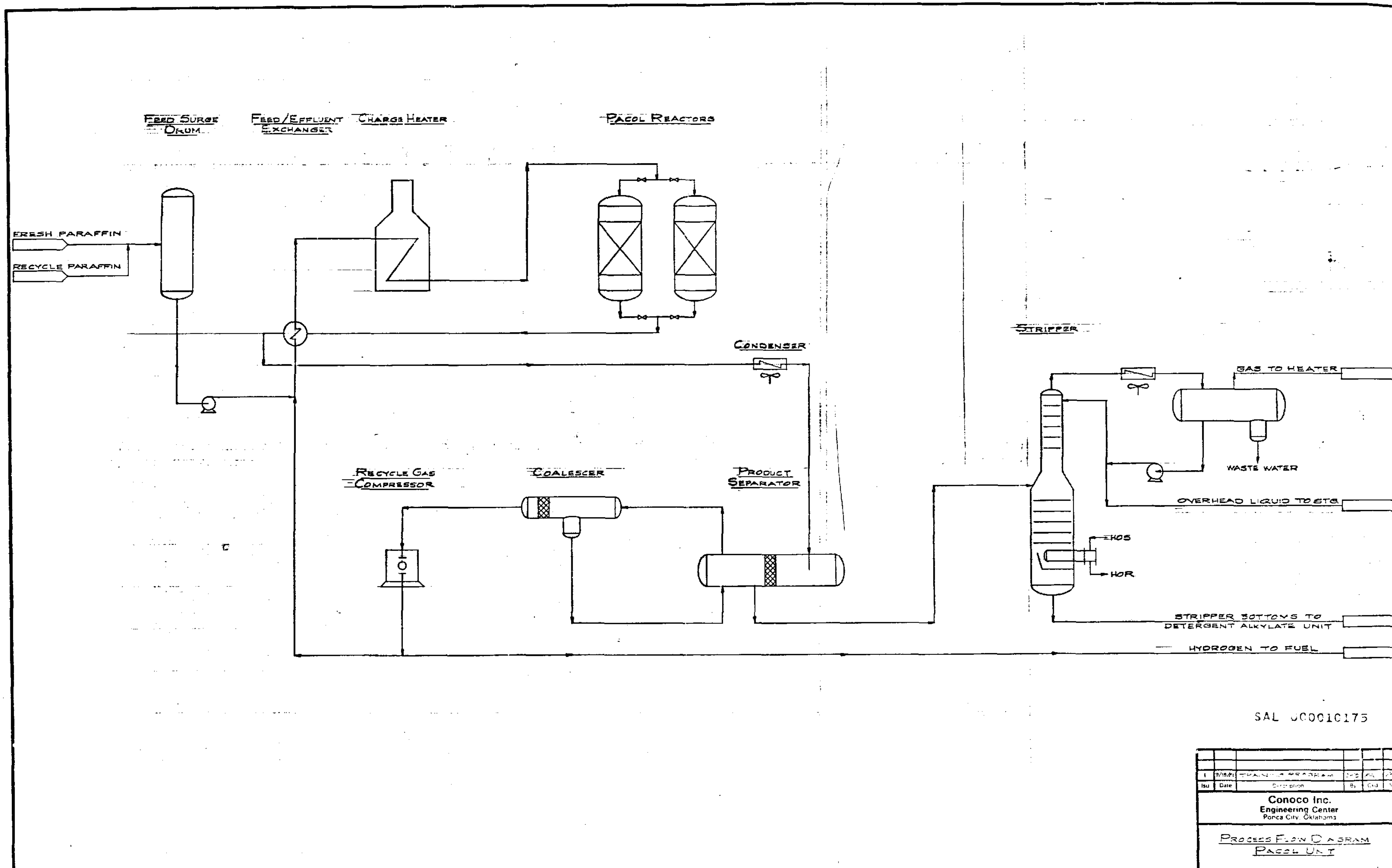
SAL 000010174

No	Date	Description	By	Chk	Appd
1	7/1/81	TRAINING PROGRAM			

Conoco Inc.
Engineering Center
Ponca City, Oklahoma

FIGURE 1
LINEAR DETERGENT ALKYLATE PROCESS

Appd [illegible] | No CT-54-D



L.C. VCM Plant

PED ORIENTATION PROGRAM

VCM PLANT

LAKE CHARLES, LOUISIANA

In 1966 Conoco entered into an agreement with the Stauffer Chemical Company concerning use of the Stauffer-developed VCM technology. Conoco and Stauffer proceeded with a 50/50 joint venture to construct a 600 MM pound per year plant in Lake Charles, Louisiana. The plant was engineered by Stauffer. Plant construction began in late 1966.

A court ruled in 1966 that the Stauffer-Conoco arrangement was in violation of Federal anti-trust laws. It ordered Stauffer to divest itself of its interest in the Lake Charles plant to Conoco. In turn, Conoco was required to self their PVC interests in Massachusetts. We were allowed to continue operating the Aberdeen, Mississippi, PVC facility. Control of the Lake Charles plant officially became Conoco's on January 1, 1968, approximately one month prior to startup. At startup, the Lake Charles plant was the largest VCM plant in the world. A series of minor plant debottlenecking projects and operational improvements have resulted in a current plant capacity of 670 MM pounds per year.

GENERAL DESCRIPTION

A block flow drawing of a typical VCM production facility is shown on drawing CT-48-A. Such a facility would typically include the following sections:

A. Direct Chlorination EDC Production
B. Oxychlorination EDC Production
C. EDC Purification
D. EDC Cracking
E. HCl and VCM Purification

A brief description of each of the processing units follows (refer to drawing CT-20-L, "Overall Process Flow Diagram--VCM Plant"):

A. Direct Chlorination EDC Production

Direct chlorination is accomplished via a liquid phase reaction utilizing ferric chloride as a catalyst. The reaction is:

$$\underset{\text{Chlorine}}{Cl_2} + \underset{\text{Ethylene}}{C_2H_4} \xrightarrow{FeCl_3} \underset{\substack{\text{1,2-Dichloroethane}\\(\text{EDC})}}{C_2H_4Cl_2}$$

The reaction is highly exothermic and proceeds to completion, producing a high quality product. Reactor product contains about 99.7 mol percent EDC with 1,1,2-trichloroethane and ethyl chloride as the main by-products. The necessary catalyst, ferric chloride, is carried into the reactor

GENERAL DESCRIPTION (CONTINUED)

A. Direct Chlorination EDC Production (Continued)

with the chlorine or results from selective corrosion within the reactor itself. $FeCl_3$ concentrations in the reactor are 25 to 50 ppm. The reaction conditions are 45°C and 22 psig. The heat of reaction is removed via an internal cooling water exchanger. All materials of construction are carbon steel.

B. Oxychlorination EDC Production

The Stauffer ethylene oxychlorination process is based on vapor phase reaction of ethylene, anhydrous hydrogen chloride, and oxygen in the presence of copper chloride catalyst to form ethylene dichloride and water. The reaction which occurs is:

$$\underset{\text{Ethylene}}{C_2H_4} + \underset{\substack{\text{Hydrogen}\\\text{Chloride}}}{2HCl} + 1/2\ \underset{\text{Oxygen}}{O_2} \xrightarrow{CuCl_2} \underset{\text{1,2-EDC}}{C_2H_4Cl_2} + \underset{\text{Water}}{H_2O}$$

Oxygen for the reaction is obtained by use of air as a feed to the system.

Three oxychlorination reactors are used in series. The total hydrogen chloride and ethylene, together with about 1/3 of the total air, are mixed and fed to the first reactor. The products and unreacted feed from the first reactor plus an additional 1/3 of the total air are mixed and fed to the second reactor. The final 1/3 of the total air and the effluent from the second reactor are fed to the third reactor.

Reaction temperature in each of the reactors is controlled between 280°C and 320°C. The lowest temperatures consistent with acceptable yields are used, as low temperatures tend to prolong catalyst life. The reactor hot spot temperature and the rate of reaction can be adjusted through varying the stoichiometric feed ratios, the reactor coolant temperature (by adjusting the steam pressure), or the air split ratio to the three reactors.

The system is operated with an 8-10 percent excess ethylene and a 20 percent excess air relative to the HCl feed. Excess ethylene is recovered by converting it to EDC via a vapor phase direct chlorination reaction over copper chloride catalyst.

Remaining chlorine and ethylene pollutants are removed from the oxychlorination vent gas stream in a fifth reactor by catalytically converting these components to chlorinated organic compounds. The principal organic product is 1,2-dichloroethane (EDC). This is recovered in the existing plant EDC recovery system.

GENERAL DESCRIPTION (CONTINUED)

C. EDC Purification

EDC produced in the direct chlorinator and EDC produced in the oxy section flow to an acid wash vessel. Here they are washed with a 4 percent solution of HCl, which is produced in the oxy section. The purpose of this step is to remove the $FeCl_3$ from the EDC. The ferric chloride promotes by-product reactions which form tars. It must be removed prior to distillation, or the additional tars production will lower yields and foul the equipment.

The acid-washed crude EDC is phase-separated and then neutralized by washing with a weak NaOH solution. This neutralized EDC is then fed to a light ends distillation column.

The light ends column removes water and light boiling components from the crude EDC. The overhead stream from this column is phase-separated. A small light ends product stream is removed and sent to storage. The water phase is combined with water from the acid and caustic wash systems and flows to a steam stripper which removes EDC. Recovered EDC flows to the caustic wash system while the water stream flows to the waste treating section.

The bottoms stream from the light ends column flows to a heavy ends column which fractionates out the heavy boiling impurities. The purified EDC is sent to the cracking section. The bottoms stream from this tower flows to a tars column where residual EDC is recovered and returned to the caustic wash section. A tars product is recovered and sent to storage.

D. EDC Cracking

Purified EDC flows to an EDC vaporizer and then to one of two cracking furnaces. The furnaces crack EDC at about 180 psig and 500°C to VCM and HCl.

$$\underset{\text{EDC}}{C_2H_4Cl_2} \longrightarrow \underset{\text{VCM}}{C_2H_3Cl} + HCl$$

About 50-55 percent of the EDC is cracked per pass. The furnace outlet stream flows to a quench column. Here the hot EDC, HCl, and VCM gases are cooled with use of a circulating cooling system. The quenched product streams are recovered from the top of the quench tower and flow to the purification towers.

A small amount of by-products is recovered from the bottom of the quench column. The stream flows to a VCM tar still system. The recovered overhead from this system is sent to the heavy ends tower in EDC purification or returned to the quench column while the tars are sent to tars storage.

GENERAL DESCRIPTION (CONTINUED)

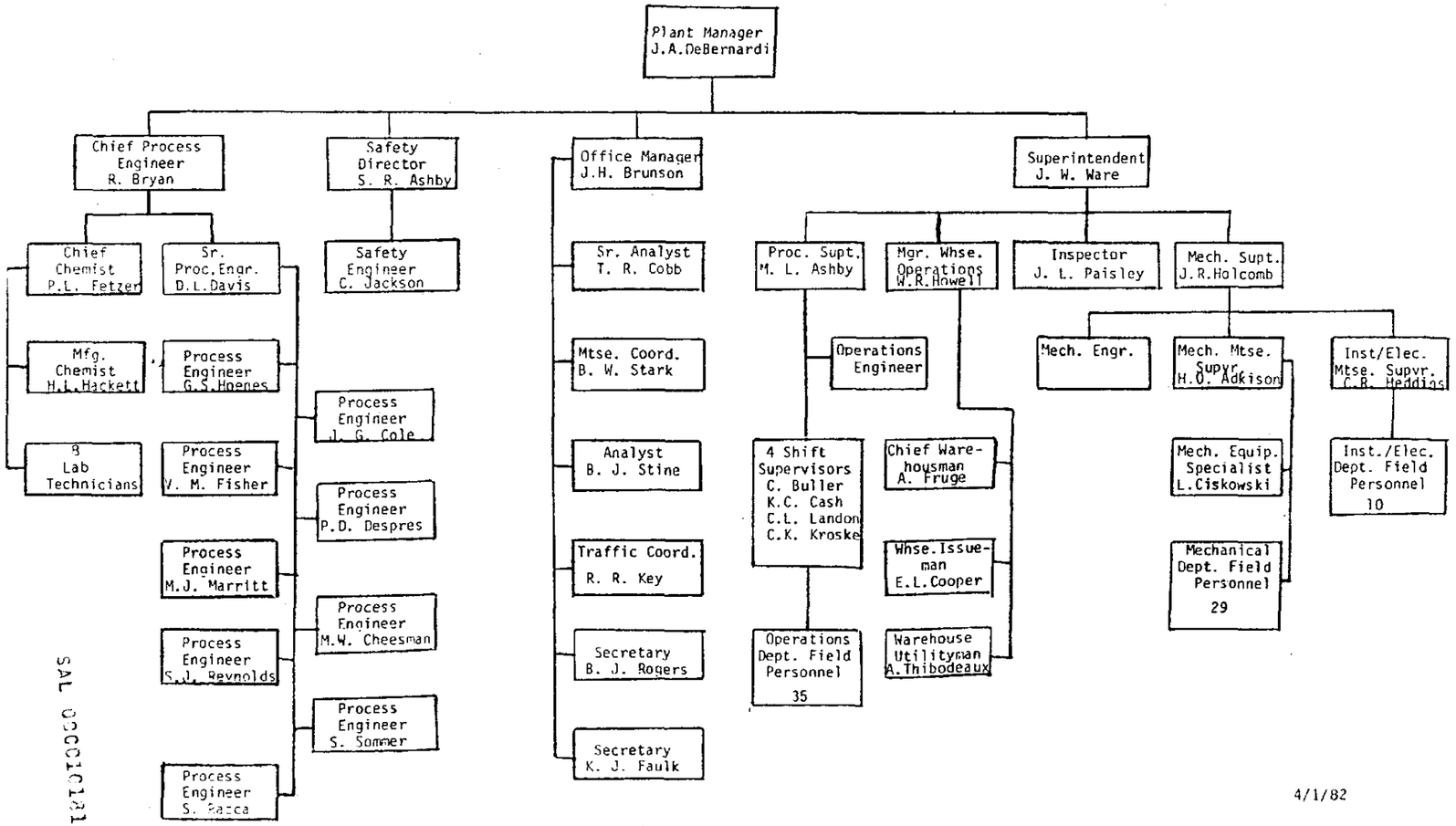
E. HCl, VCM Purification

Products from the quench column flow to the HCl column where HCl is purified and fed to the oxy unit. The bottom stream from this column contains EDC and VCM and is sent to the VCM column.

The VCM column performs the final purification on the product. The overhead stream is product VCM. It flows through flake caustic dryers to remove any residual HCl and then is sent to product storage.

The bottoms stream from the VCM column is EDC. This stream is recycled to the EDC Purification Section for removal of unwanted impurities.

VCM PLANT ORGANIZATIONAL CHART



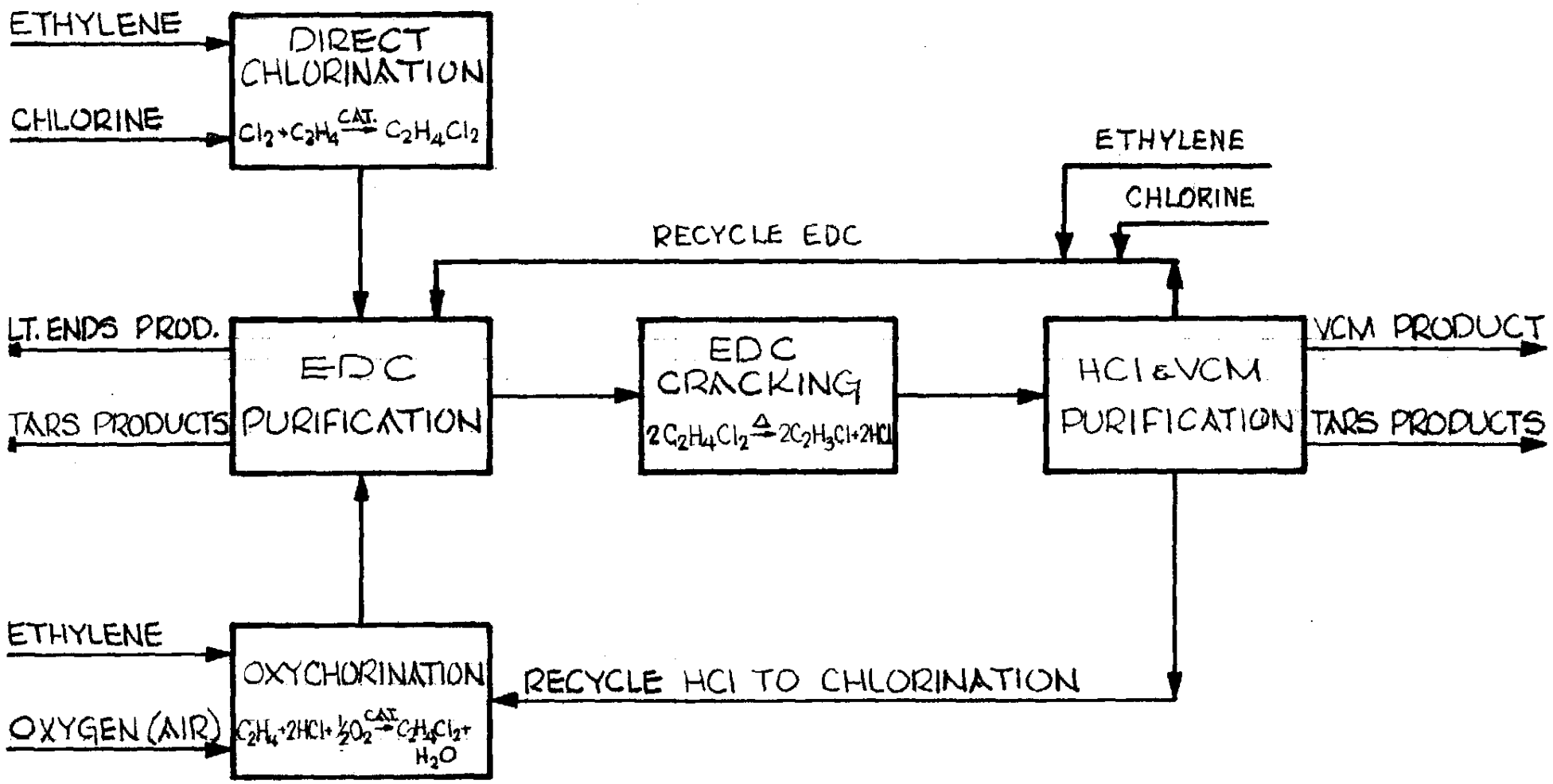
4/1/82

SAL 0000121

FIGURE 1

BLOCK FLOW DIAGRAM

VCM PROCESS



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Spec. No. 08-5000

VINYL CHLORIDE MONOMER

Special Identification: General sales specification

Test	Specifications	Test Method
Acetylene*	2.0	1.100
Methyl Chloride*	100.0	1.100
1,3 Butadiene*	15.0	1.100
Acetaldehyde*	0.7	1.114
Nonvolatiles*	100.0	1.113
Water*	200.0	1.110
Iron*	0.5	1.112
Sulfur*	1.0	1.115
Acidity* (as HCl)	2.0	1.111
Color	Colorless	
Appearance	Clear and free from suspended matter	

*parts per million by weight, maximum

Remark (for plant use only):

Oxygen: 1000 ppm max. before loading
500 ppm max. after loading

10-1-72

SAL 0000C10183

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Spec. No. 08-5010

VCM Light Ends By-Product

Special Identification: General Sales

All specifications and tests must be approved by VCM plant personnel prior to customer contact because stream composition will continue to change as process improvements are made.

Typical compositional component ranges are shown below:

	Wt. %
Vinyl Chloride	1-5
Ethyl Chloride	25-50
1,1-Dichloroethene	1-8
1,1-Dichloroethane	2-6
T-1,2-Dichloroethene	3-7
C-1,2-Dichloroethene	1-5
Trichloromethane (Chloroform)	15-30
Tetrachloromethane (Carbon Tetrachloride)	5-12
1,1,2-Trichloroethylene	Nil - 0.3
1,2-Dichloroethane	3-12
Water	1000-3000 ppm

5-16-78
(Replaces 12-15-73)

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Spec. No. 08-5015

VCM Tars By-Product

Special Identification: General Sales

All specifications and tests must be approved by VCM Plant personnel prior to customer contact because compositions will continue to change as process improvements are made.

Typical compositional component ranges are shown for those components analyzed.

EDC	8 - 25%
1,1,2-Trichloroethane	35 - 65%
Acid (as HCl)	500 - 2000 ppm
Water	Nil - 400 ppm
Non-volatiles	2.0 - 6.0%

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Spec. No. 08-5050

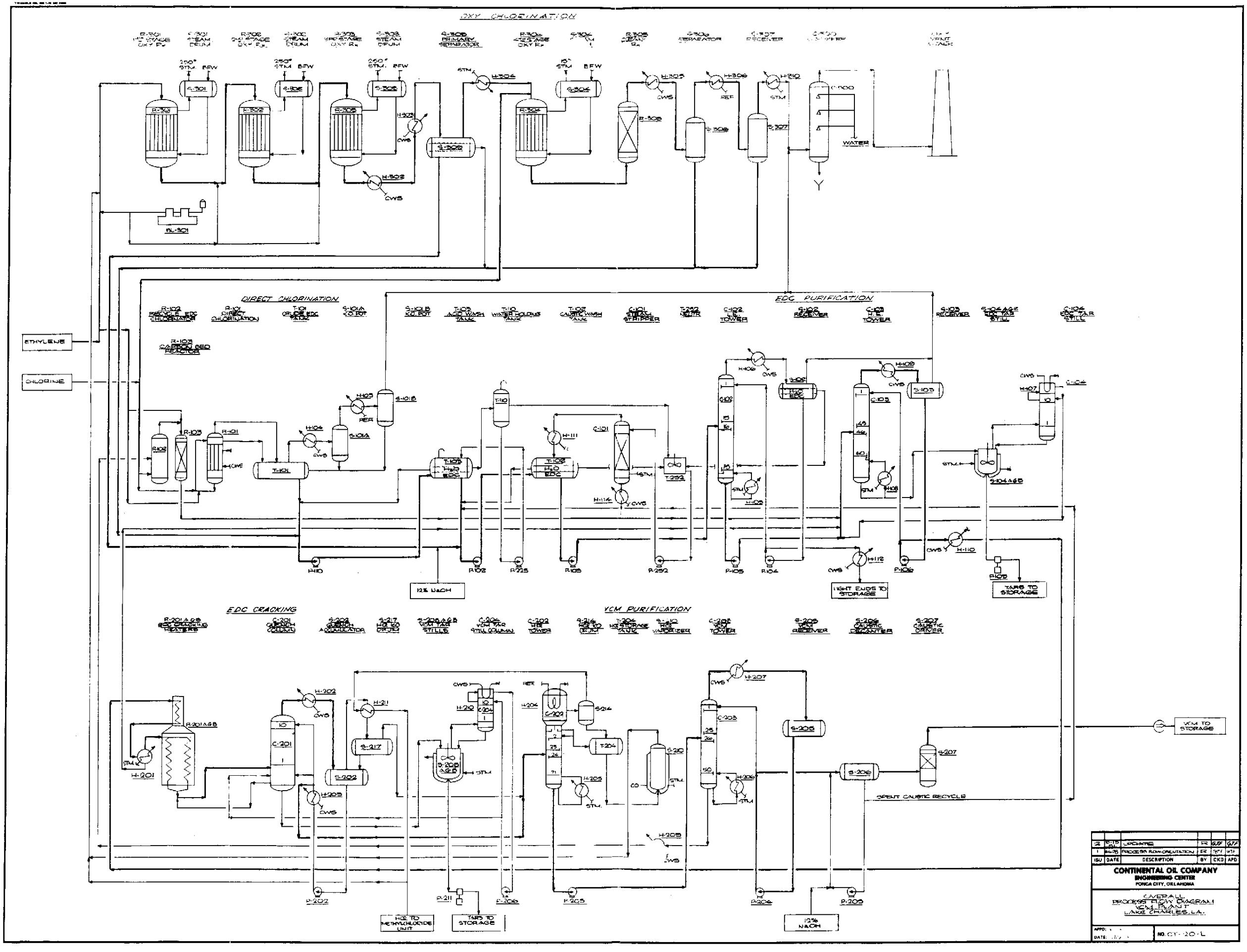
ETHYLENE DICHLORIDE

Special Identification: For Public Sale

Test	Specifications	Test Method
Purity, Wt. %	99.0 min	1.120
Specific Gravity (20°C/20°C)	1.252-1.259	ASTM-D-1298
Distillation Range	100% within 2°C including 83.5°C	ASTM-D-86
Acidity (as HCl), ppm	10 max	1.117
Residue on Evaporation, ppm	20 max	1.113
Water Content, ppm	100 max	1.110
Appearance	clear and free of suspended matter	Visual

Note - These specifications apply only to material FOB Lake Charles, LA

9-30-76
(Replaces 2-7-73)



SAL 000010187

Aberdeen PVC Plant

SAL 0C0C1C183

PED ORIENTATION PROGRAM

ABERDEEN PVC PLANT

ABERDEEN, MISSISSIPPI

The Aberdeen plant, and other facilities on the East Coast, were acquired from Thompson-Apex Company in early 1965. This entry into the PVC business was temporarily set back in late 1967 when the U.S. Government, by consent decree, forced Conoco to divest of all Thompson-Apex plants except that at Aberdeen. This edict occurred partially as a result of the Conoco-Stauffer joint venture VCM facility then under construction at Lake Charles, Louisiana.

The original Aberdeen plant was built in 1962-1963. It included a reactor-dryer building, a PVC compound line, a plasticizer plant, garden hose manufacturing unit, and a large warehouse. Expansion in 1965 essentially doubled plant facilities. Subsequent expansions raise output to 200 MM pounds per year PVC resins from sixty 2,200-gallon polymerizer vessels. An 18,000-gallon prototype reactor installed in 1970 proved highly successful. As a result, the plant constructed seven more large reactors while shutting down all small polymerizers. This latter shutdown was prompted by OSHA's promulgation of the "VCM Ambient Air Standard" in 1974 and EPA's promulgation of the "VCM Emission Standard" in 1976. The plant was further expanded with the addition of two 32,000-gallon reactors which started up in early 1982. Plant capacity is now 455 MM pounds per year of PVC resin.

Plant feedstock is vinyl chloride monomer from Conoco's VCM plant at Lake Charles, Louisiana.

GENERAL DESCRIPTION

PVC is produced by batch polymerization of VCM in a water suspension. VCM is charged to the reactor along with water, a methyl cellulose suspending agent to stabilize the VCM in water suspension, and a peroxide initiator. Major features of the polymerization reaction are:

1. Polymerization temperature determines the PVC molecular weight which in turn influences resin characteristics. Molecular weight is not dependent on conversion. Since a narrow molecular weight distribution is desired, isothermal polymerization conditions are used. Typical number average and weight average molecular weights are 30,000 and 60,000, respectively.

2. Particle size and distribution are influenced by the suspension agent concentration. Typical mean particle size is 165 microns.

3. The polymerization is autocatalytic; that is, the rate increases with conversion. Reaction rate also depends on the type of initiator and the polymerization temperature. Typical temperatures are 120°F to 150°F.

GENERAL DESCRIPTION (CONTINUED)

PVC is never used in its "pure" form but rather is mixed with filler, plasticizer, stabilizer, etc, enroute to the final product. The quality of the final material rests not only on its molecular weight and particle size but also on the PVC particle morphology. Quality is generally defined by how well it processes, ie, rate of plasticizer adsorption or heat stability during extrusion.

PROCESS DESCRIPTION (See Attached Simplified Process Flow Drawings)

The PVC polymerization process is straightforward. A typical reactor batch cycle is described below.

The empty polymerization reactor is evacuated to remove air and then charged with VCM, water, and suspension agent. Following charge, the peroxide initiator (which is stored at -20°F because of its instability) is poured into a small charge pot and pressured into the reactor with water. The polymerization then proceeds under temperature control with cooling water removing the heat of polymerization. The reaction is terminated at 75 to 85 percent conversion of VCM to PVC (determined by reactor pressure). The excess monomer is recovered by depressuring the reactor. During this stage, the polymer slurry is heated by injecting steam to facilitate VCM removal from the polymer.

The PVC water slurry is then drained from the reactor and pumped to intermediate storage. The slurry is displaced with air. The reactor is rinsed with a hot caustic solution which retards polymer buildup in the vessel, rinsed with water, and evacuated for the next batch.

Typical batch cycle time is around eight hours, with a polymerization time of six hours.

Th slurry is pumped from intermediate storage to continuous solid bowl centrifuges for gross dewatering. The "wet cake" gravity flows into a gas-fired rotary drum dryer or a fluid bed dryer. The dried resin is screened and is then pneumatically conveyed to product storage.

Aberdeen has additional facilities to produce dry blend (for rigid PVC applications such as pipe) and compounds (for flexible PVC applications).

The dry blend unit prepares a uniform mixture of PVC resin, filler, color, stabilizer, and lubricant. The most common filler is atomite. Capacity is 77 MM pounds per year.

All the raw materials are fed to use bins of the dry blend area which in turn gravity feed to the weigh tanks. When all the raw materials are in the use bins, the dry blending process is started. All raw materials are automatically weighed, based on the amount of each that have been preset on the weighing control panel in the dry blend control room. When all the ingredients are weighed, they are automatically discharged in a Welex intensive mixer. The Welex mixer vigorously mixed the ingredients so that a homogeneous mixture is achieved quickly. During mixing, mechanical energy is transferred to the dry blend material causing the temperature to rise.

PROCESS DESCRIPTION (CONTINUED)

The dry blend is automatically dropped at a preset temperature, usually about 220°F, to a ribbon blender where the dry blend is cooled to about 120°F before being transferred to product storage.

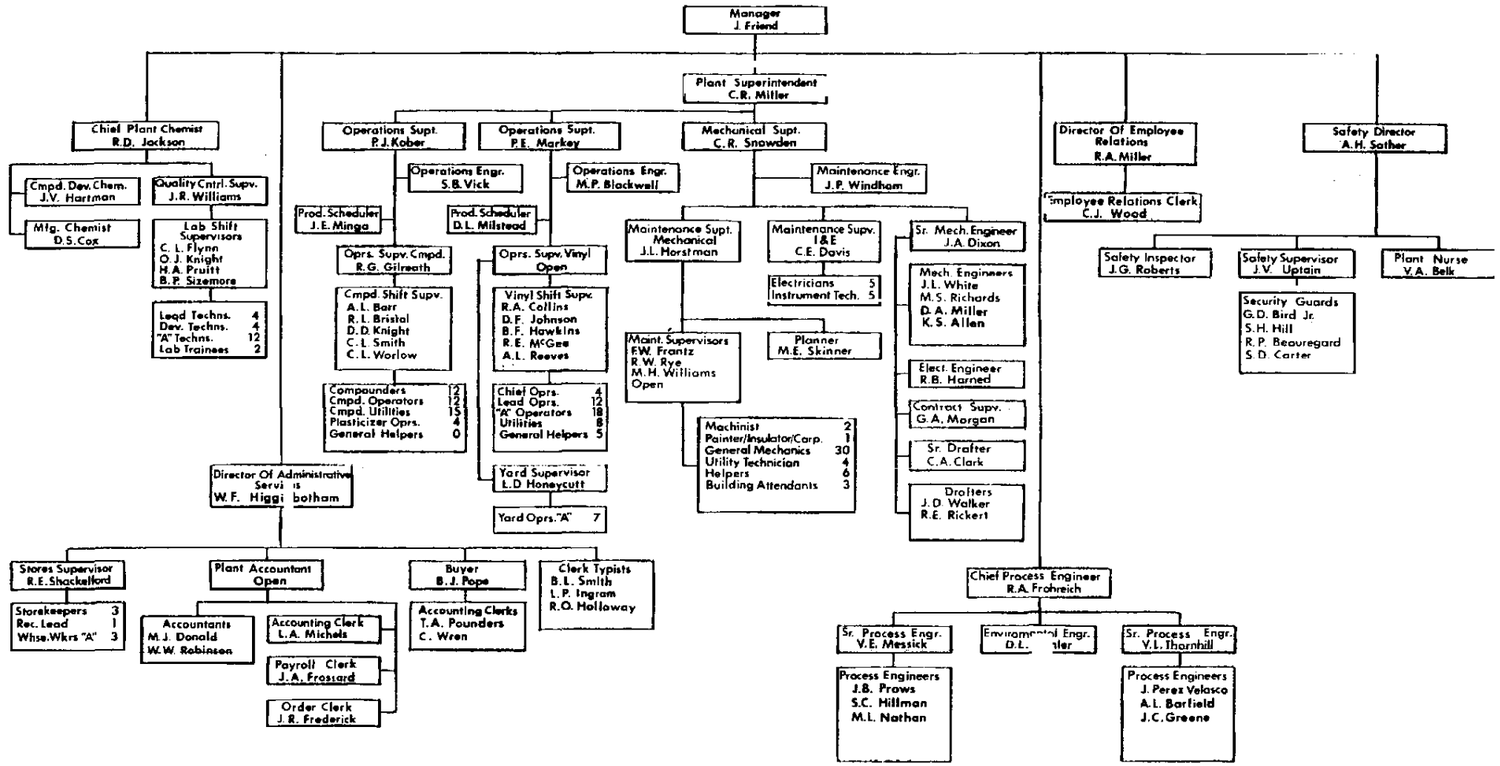
The plant has three compound units. A compound unit produces a fused and homogeneous mixture of PVC resin, plasticizer, stabilizer, color, filler, and other miscellaneous small additives. Total capacity is 80 MM pounds per year.

The process flow for compound line III is as follows: The raw materials are weighed and added to a ribbon blender where the mixture is allowed to blend until uniform throughout. The mixture is then fed to a Farrell continuous mixer. This mixer fuses the material into a "rubberlike" mass. The fused material is then sent to a two-roller mill where a ribbon (8-14 inches wide, 1/8 inch thick) of compound is made which is then cooled in cooling troughs and cooling tanks before being diced into pellets. After dicing, the pellets are screened to remove oversize and fines and conveyed by air pressure to product storage.

Aberdeen also produces plasticizer mostly for internal consumption. Capacity is 25 MM pounds per year. There are a variety of plasticizers produced, depending on compound formulation. The most popular one is made by reacting phthalic anhydride with either 2-ethylhexanol or "ALFOL" 610 alcohol to form the ester. An acid catalyst is used. When the batch reaction is complete, the reactant mass is neutralized and steam stripped to remove excess alcohols. The ester is then filtered and stored.

PRODUCTS

Aberdeen produces five separate grades of PVC resin which are ultimately used for electrical insulation, shoe soles, automobile parts, toys, upholstery, floor mats, and plastic pipe. The largest market is for resin used to make plastic pipe.



ORGANIZATION CHART
ABERDEEN, MISS.
AUG. 1, 1982

SAL 09CC1C192

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Spec. No. 15-1822

CONOCO PVC RESIN NO. 5305-1

Special Identification: Generally for Flexible Applications

Test	Specification	Typical Property	Test Method
Total Volatility, %	0.50 max	0.25	8.004
Moisture, %	0.30 max	0.20	8.020
Bulk Density, gms/cc	0.528-0.624	0.58	8.007
Contamination Count	50 max	20	8.017
Inherent Viscosity	0.72-0.77	0.74	8.009
Particle Size Distribution, %			8.018
On 40 Mesh	0.1 max	Trace	
Through 140 Mesh	25.0 max	15.0	
Through 200 Mesh	5.0 max	2.5	
Color, Gardner b	3.5 max	2.8	8.012
Heat Stability	Equal to Standard	Passes	8.011
Static Flow	Free Flow	Passes	8.016
Residual VCM, ppm	10.0 max	3.0	8.014

2-3-81
(Replaces 8-1-80)

SAL 0C0C10193

Spec. No. 15-1849

CONOCO PVC RESIN NO. 5385-1

Test	Specification	Typical Property	Test Method
Total Volatility, %	0.50 max	0.25	8.004
Moisture, %	0.30 max	0.20	8.020
Bulk Density, gms/cc	0.512-0.576	0.53	8.007
Contamination Count	25 max	15	8.017
Inherent Viscosity	0.90-0.95	0.92	8.009
Gel Rating	3 max	2	8.015A
Brabender Dry Time, Minutes	6.0 max	5.5	8.008
Particle Size Distribution, %			8.018
On 40 Mesh	0.1 max	Trace	
Through 140 Mesh	20.0 max	15.0	
Through 200 Mesh	5.0 max	2.5	
Color, Gardner b	3.0 max	2.5	8.012
Heat Stability	Equal to Standard	Passes	8.011
Static Flow	Free Flow	Passes	8.016
Residual VCM, ppm	10.0 max	1.5	8.014

2-3-81
(Replaces 8-1-80)

SAL 000010194

Spec. No. 15-1302

CONOCO 34431 VINYL COMPOUND

Special Identification: SPT 1 and 2 Cords, T & TW Building Wire, 80°C Appliance Wire, UF & WMC Insulation and Jacket

Test	Specification	Typical Property	Test Method
Specific Gravity	1.37 ± 0.03	1.37	ASTM D792
Hardness, Aged			8.005-76
Instantaneous	D-44 ± 3	D-46	
15-Sec Delay	—	D-35	
Tensile Strength, psi		2540	ASTM D638
100% Modulus, psi		1630	ASTM D638
Elongation, %		315	ASTM D638
Brittlepoint, °C		–23	ASTM D746
VR, ohm-cm			8.006B-77

7-1-81

(Replaces 8-1-80)

Spec. No. 15-1900

CONOCO 610-P

Special Identification: Phthalate Diester of ALFOL ADE 610 Alcohol (Spec. No. 03-4114-01)

Test	Specification	Typical Property	Test Method
Specific Gravity at 25°/25°C	0.974 ± 0.004	0.974	1.023
Color, APHA	25 max	10	1.025
Moisture Content, wt %	0.10 max	0.05	1.016
Acidity (as Acetic Acid), wt %	0.01 max	0.005	1.024
Refractive Index at 25°C		1.483	ASTM D1218
Alcohol Content, wt %	0.30 max	<0.1	
Odor	Characteristic		
Appearance	Free From Haze		

7-1-81
(Replaces 8-1-80)

Spec. No. 09-1410

CONOCO 90281 PVC Dry Blend

Special Identification: White Vinyl Siding Dry Blend

Test	Specification	Typical Property	Test Method
Fusion Time (T_1) Minutes	± 0.35 minutes from control blend	0.7	8.013
Stability Time (T_2-T_1), Minutes	Minimum two minutes less than control blend	17.5	8.013
Equilibrium Torque Meter Grams	± 150 M-gm from control blend	1750	8.013
Specific Gravity	1.47 ± .02		

3-1-78
(Replaces 10-26-77)

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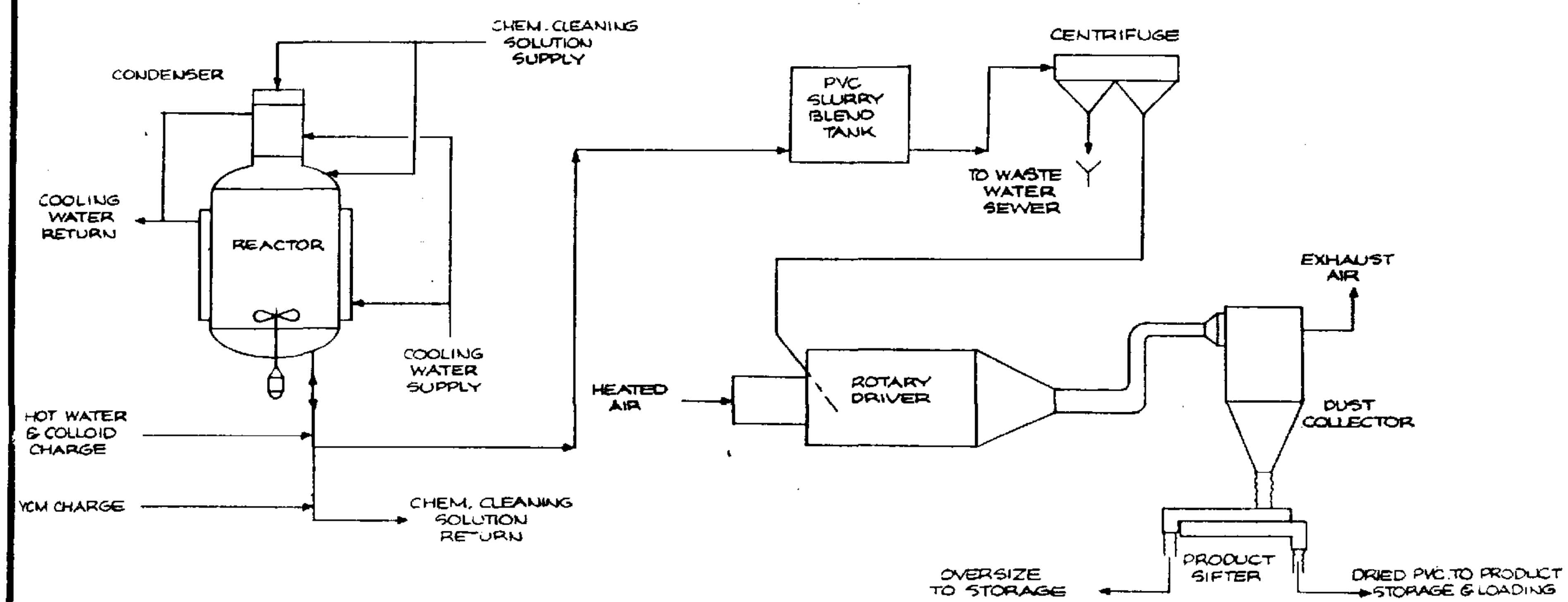
Spec. No. 15-1910

CONOCO DOP

Special Identification: Phthalate Diester of 2-Ethyl Hexanol

Test	Specification	Typical Property	Test Method
Specific Gravity at 25°/25°C	0.983 ± 0.003	0.983	1.023
Color, APHA	25 max	10	1.025
Moisture Content, wt %	0.10 max	0.05	1.016
Acidity (as Acetic Acid), wt %	0.01 max	0.005	1.024
Refractive Index at 25°C	1.4850		ASTM D1218
Ester Content, wt %	99 min	99.6	
Alcohol Content, wt %	0.10 max	0.05	
Odor	Characteristic		
Appearance	Free From Haze		

7-1-81
(Replaces 8-1-80)



1	11-7-73	PED TRAINING PROGRAM			JWB
ISU	DATE		DWN	CKD	APD

CONTINENTAL OIL COMPANY
ENGINEERING CENTER
PONCA CITY, OKLAHOMA

SIMPLIFIED PROCESS FLOW DIAGRAM
LARGE REACTOR PVC PLANT
ABERDEEN, MISS.

SCALE:

APPD: GJP
DATE: 11/16/78

No. CT-49-A

4-281 S

NORMAL USE BIN MATERIALS

USE BIN	MATERIAL
1	RESIN
2	FILLER
3	REWORK
4	MASTER BATCH
5	SPARE
6	STABILIZER
7	STABILIZER

PVC RESIN FROM VINYL ALEX

BULK RESIN SILOS (2)

RESIN BLOWER

EXHAUST

USE BIN #1

USE BIN #2

USE BIN #3

USE BIN #4

USE BIN #5

USE BIN #6

USE BIN #7

"A" SCALE

"B" SCALE

"C" SCALE

"D" SCALE

STABILIZER TOTE TANK

DIVERTER VALVES

#1 WELEX

#2 WELEX

INTENSIVE COOLER

ROTARY DIVERTER VALVE

"C" COOLER

"A" COOLER

"B" COOLER

"D" COOLER

PRODUCT BLOWER

MANUAL CHARGE HOPPER

BAGS DUMPED MANUALLY

BLOWER

MASTER BATCH CHARGE BLENDER

DRY BLEND PRODUCT SILOS

PRODUCT TO GAYLORD, RAILCARS OR TRUCKS

ISU	DATE	DESCRIPTION	BY	CKD	APD
1	8-20-82	PFD ORIENTATION	MAC	RCB	RCB

CONOCO INC.
ENGINEERING CENTER
PONCA CITY, OKLAHOMA

SIMPLIFIED PROCESS FLOW DIAGRAM
DRY BLEND
ABERDEEN, MISSISSIPPI

APPD: RC Bailey
DATE: 8-27-82

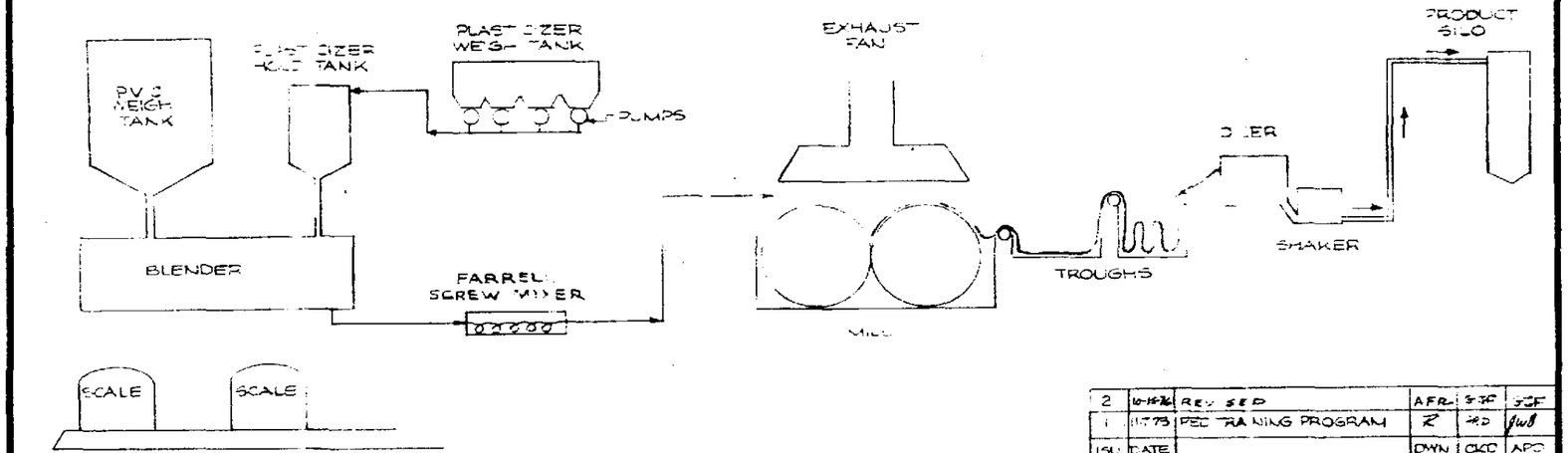
SHT. NO.	UNIT	AREA

NO. CT-28-C

ISSUE

BRUNING 40-21 52296

4-282-S (9-81)



2	6-1-76	REVISED	AFR	[illegible]	[illegible]
1	11-7-75	PEC TRAINING PROGRAM	R	HD	JWB
ISU	DATE		DWN	CKD	APD

CONTINENTAL OIL COMPANY
ENGINEERING CENTER
PONCA CITY, OKLAHOMA

SIMPLIFIED PROCESS FLOW DIAGRAM
COMPOUND AREA
ABERDEEN, MISS.

SCALE:

APPD: [illegible]
DATE: 3-7-72

No. CT-27-B

4-281 S

SAL 20010201

Oklahoma City PVC Plant

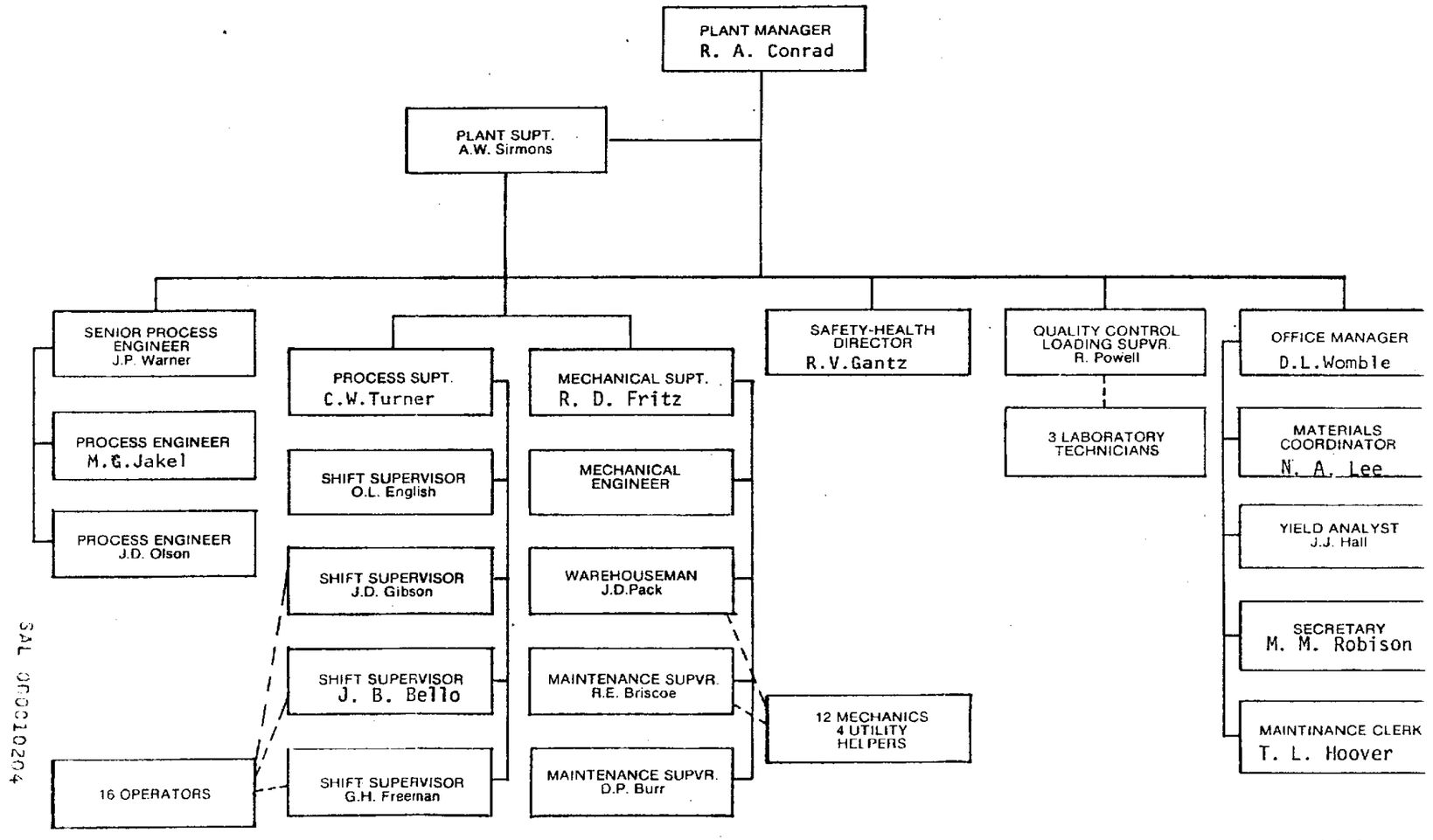
PED ORIENTATION PROGRAM

OKLAHOMA CITY PVC PLANT

OKLAHOMA CITY, OKLAHOMA

The Oklahoma City plant became operational in April of 1971. It was constructed primarily to provide pipe-grade PVC resin to an adjacent Carlon Plastics Company plant. The plant scope included a four-reactor (large volume) module with associated resin drying facilities, utilities, and offsites. While the initial design nameplate capacity was 70 MM pounds per year, the plant's output far exceeded that from the start. Subsequent expansions have added two new large reactors and other facilities which have elevated output to 275 MM pounds per year. Details of the process for PVC resin production are similar to those described for the Aberdeen PVC Plant.

ORGANIZATIONAL CHART
OKLAHOMA CITY PLANT



SAL 05C01204

Rev 8/82

Spec. No. 15-1850

CONOCO PVC RESIN NO. 5385-3

Test	Specification	Typical Property	Test Method
Total Volatility, %	0.50 max	0.25	8.004
Moisture, %	0.30 max	0.20	8.020
Bulk Density, gms/cc	0.512-0.576	0.53	8.007
Contamination Count	100 max	60	8.017
Inherent Viscosity	0.90-0.95	0.92	8.009
Brabender Dry Time, Minutes	7.0 max	6.0	8.008
Particle Size Distribution, %			8.018
On 40 Mesh	0.1 max	Trace	
Through 140 Mesh	20.0 max	15.0	
Through 200 Mesh	5.0 max	2.5	
Color, Gardner b	3.5 max	2.5	8.012
Heat Stability	Equal to Standard	Passes	8.011
Static Flow	Free Flow	Passes	8.016
Residual VCM, ppm	10.0 max	2.0	8.014

+ **Note:** This product also manufactured at Oklahoma City, OK, plant — see Specification 16-1850.

2-3-81
(Replaces 8-1-80)

VCM UNLOADING AND STORAGE

VCM TANK CAR

VCM STORAGE BULLETS

FRESH VCM STORAGE TANK

RECOVERED VCM STORAGE TANKS

SUSPENDING AGENT

PROCESS WATER

PROCESS WATER

COMMON EVACUATION EJECTOR

STEAM

ATM

POLYMERIZATION REACTORS & AUXILLIARIES

(2)

RECOVERED VCM L.G. DRUMS

RECOVERY VACUUM PUMPS

RECOVERY COMPRESSORS

RECOVERED VCM CONDENSERS

SEAL WATER SEPARATORS

RECOVERED VCM RECEIVER

SEAL WATER DRAIN(2)

ATM

VCM INCINERATOR

CONTROL DEVICE

INERT VENT REFRIGERATION SYSTEM

REFRIGERANT

(2)

CONTROL DEVICE

CONTROL DEVICE VACUUM RECOVERY SYSTEM

WASH WATER EFFLUENT

ATM

(2)

REACTOR CLEANING SYSTEM EXHAUST BLOWER

WASTE WATER TREATMENT PONDS

REACTOR CLEANING SYSTEM

OVERSIZED PRODUCT

REACTOR CLEANING RINSE WATER(2)

LIQUID DRAIN(2)

WATER STRIPPERS

STEAM

PROCESS SEWER

ATM

(2)

DUMP SCREENER EXHAUST BLOWER

SLURRY DUMP SCREENER

OVERSIZED PRODUCT

SLURRY BLEND TANKS

CENTRIFUGES

CENTRATE WATER (1)

AIR

ROTARY DRYERS

BAG TYPE PRODUCT & DUST COLLECTORS

ATM

(1)

DRYER INDUCED DRAFT FANS

PRODUCT SIFTERS

OVERSIZED PRODUCT

AIR

PNEUMATIC CONVEYING AIR COMPRESSORS

ATM (1)

BAG TYPE DUST COLLECTORS

STORAGE SILOS

RAIL CAR LOADING SILOS

HOPPER RAIL CARS

AIR

PNEUMATIC CONVEYING AIR COMPRESSORS

CONVEYED TO ADJACENT PROCESSING PLANT

RETIN SETTLING BASIN

NOTES:

(1) CONTINUOUS FLOW
(2) INTERMITTENT FLOW

ISU	DATE	DESCRIPTION	BY	CK'D	APP'D
3	11/19/81	GENERAL REVISIONS	[illegible]		
2	[illegible]	UPDATE	[illegible]		
1	[illegible]	EPA REPORT	[illegible]		

CONTINENTAL OIL COMPANY
ENGINEERING CENTER
PONCA CITY, OKLAHOMA

[illegible] PLANT
[illegible]

USE BORDER SCALE:

APP'D:
DATE:

No.

SCALES FOR MICROFILMED DRAWINGS

4-233 S

PROCESS ENGINEERING DEPARTMENT

ORIENTATION PROGRAM - 1982

OIL AND GAS PROCESSING DIVISION

SEPTEMBER 9, 1982

TABLE OF CONTENTS

1982 ORIENTATION PROGRAM

OIL AND GAS PROCESSING DIVISION

PED ORIENTATION PROGRAM

BALTIMORE CHEMICAL PLANT

BALTIMORE, MARYLAND

The Baltimore Petrochemical Plant was originally the Prudential Oil Refinery. It was converted to the production of dodecylbenzene, better known by the tradename "NEOLENE", in the early 1950's. "NEOLENE" alkylate was produced from dodecene and benzene by aluminum chloride alkylation. "NEOLENE" alkylate and similar alkylates produced by other companies were sulfonated with sulfuric acid or oleum to produce most of the detergents sold in the 1950's and 1960's.

Detergents made from "NEOLENE" alkylate were classified as "hard" because they are not biodegradable. The increased use of synthetic detergents led to foaming in rivers and lakes near major population centers. Conoco Research recognized the problem and developed a new detergent alkylate. "NALKYLENE" alkylate, which produced "soft" or biodegradable detergents. "NALKYLENE" alkylate is also an alkylbenzene, but the alkyl group is linear, whereas "NEOLENE" alkylate had a branched paraffin sidechain. A "NALKYLENE" alkylate pilot plant was operated at Ponca City in 1962. The Baltimore plant was converted to "NALKYLENE" alkylate production in 1965. Production increased from 150 MM pounds per year in 1967 to 220 MM pounds per year in 1976. The increase was made possible by debottlenecking projects in the fractionation section and more recently, the chlorination and HCl absorption sections. Muriatic acid and aqueous aluminum chloride are produced as byproducts of the process. The bottoms from fractionation are sold as feedstock for oil soluble sulfonate production.

The Specialty Alkylate Unit (SAU) in the Baltimore plant produced high molecular weight alkylbenzenes that were used for the production of oil soluble sulfonates. Baltimore Base Oil (BBO) and DN-500 (a low pour lubricating oil) were produced in the SAU. This unit was shut down in 1979 and the technology for producing specialty alkylate was transferred to Witco Chemical Company. BBO and DN-600 are toll processed by Pilot Chemicals.

The Baltimore plant also includes the STXS Unit, a small batch reaction process that is used to produce sodium toluene and xylene solfonates and ammonium xylene sulfonate (STS, SXS, STXS, and AXS). These materials are known as hydrotropes and are used as anticaking agents for powdered detergents and clarification agents for liquid detergents. Total production is about 3 MM pounds per year.

WASTE TREATMENT

Baltimore's waste treatment system consists basically of:

1. API Separator
2. Dissolved Air Floating Unit
3. Gravity Dewatering System
4. Storm Water Diversion System
5. Acid Neutralization Pit

WASTE TREATMENT (CONTINUED)

Effluent from the acid neutralization pit is transferred to the API separator. Once this system is operated as designed, the Baltimore plant will tie into a new minicipal water treatment plant. Doing this will eliminate the need for further additions to Baltimore's waste treatment system.

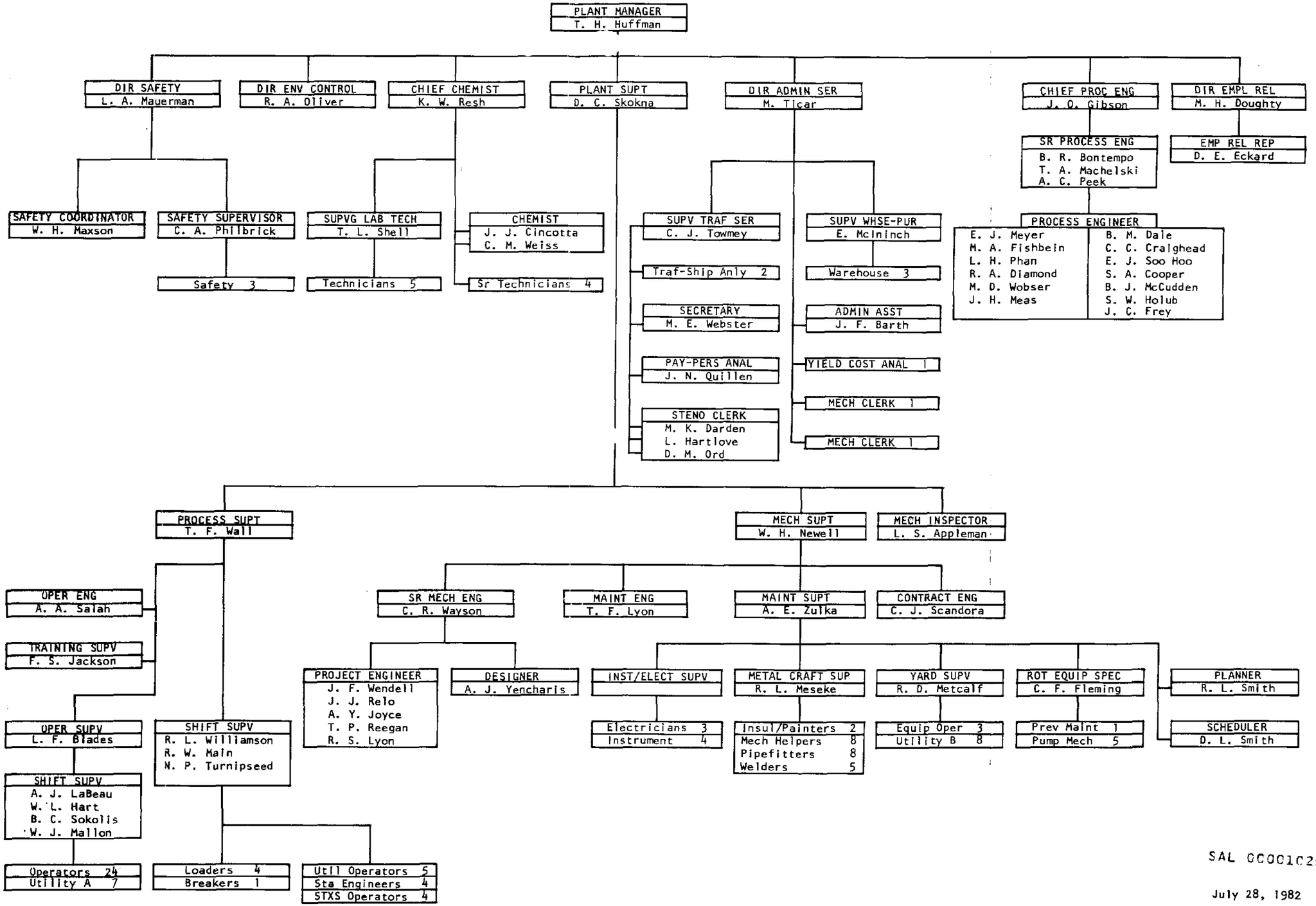
When the entire system is operating, waste water is neutralized before it enters the API separator. In the API separator, oil and some solids are removed. The effluent from the separator is saturated with air at about 40 psig. The air-saturated effluent enters the flotation cell. Air bubbles encapsulate solids and bring them to the top of the cell where they are skimmed off. A polymer is added to the flotation cell to enhance solids coagulation. Water from the flotation cell enters a holding pond. From the holding pond, the water is discharged to the Patapsco Rover or the municipal treatment plant.

Solids from the flotation cell enter a holding tank. From here they are pumped through a rotating drum filter where some of the water is removed. The solids are then trucked to a disposal site.

The acid neutralization pit is banked with limestone. Muriatic acid is neutralized when it is off-specification or when the storage capacity is exhausted. Effluent from the acid pit is pumped to the API separator where it joins the waste water from the rest of the plant.

The storm water diversion system consists of four 3,000 gpm pumps and a storm water holding tank. When heavy rain increases waste water flow above the capacity of the treatment system, water is automatically pumped on level control to the holding tank. Water in the holding tank is later purged on control through the treatment system.

CONOCO CHEMICALS
BALTIMORE PLANT



SAL 000010203

July 28, 1982

SAL 00001047C9

PED ORIENTATION PROGRAM

"NALKYLENE" ALKYLATE UNIT

BALTIMORE CHEMICAL PLANT

BALTIMORE, MARYLAND

The "NALKYLENE" Alkylate (NAB) Unit became operational in 1965. It was constructed in the midst, and using some equipment, of its predecessor, the "NEOLENE" Alkylate Unit. The latter had been in operation since 1947 and had been Conoco's first petrochemical plant. The nucleus of the "NEOLENE" Alkylate Unit was then converted to a Specialty Alkylate Unit (SAU) described in a later section.

The "NALKYLENE" Alkylate Process was developed by Conoco to meet the need for a fully biodegradable detergent feedstock. The original unit had a production capability of approximately 50 MM pounds per year. It has subsequently been expanded several times to its present capacity of 220 MM pounds per year. Feedstocks for the unit are normal paraffins from the Lake Charles Chemical Plant, benzene, and chlorine. The plant operates on blocked-out operation to produce alternately a 10-12 carbon chain alkylbenzene (N-500) or a 12-14 carbon chain alkylbenzene (N-600). Plant throughput capability is essentially the same with either product and is divided approximately 70 percent N-500 and 30 percent N-600. In addition to these flexibilities, the plant also can vary alkylation reaction conditions (basically benzene/chloroparaffin mol ratio) to yield various dialkylbenzene bottoms products. These are sold to Witco and other companies to produce oil soluble sulfonates.

GENERAL DESCRIPTION

Crude normal paraffins are fractionated into desired cuts each having a three carbon range. Each paraffin cut is alternately (blocked-out operation) reacted in excess with chlorine at elevated temperatures. The resulting chloroparaffins are reacted with benzene in the presence of an aluminum chloride catalyst to yield the desired alkylbenzenes. By-product HCl is recovered and marketed as food-grade muriatic acid. The chemistry of the "NALKYLENE" Alkylate Process is represented as follows:

$$5\ R\text{-}H + Cl_2(g) \longrightarrow R\text{-}Cl + 4\ R\text{-}H + HCl$$

PROCESS DESCRIPTION

The attached Overall Process Flow Diagram illustrates the "NALKYLENE" Alkylate Process.

PROCESS DESCRIPTION (CONTINUED)

Section 150 - Paraffin Fractionation

Normal paraffins are received from the Lake Charles plant by tanker either as a C_{10} to C_{16} crude or as C_{10} to C_{12} and C_{12} to C_{14} cuts. The C_{10} to C_{12} cut is used for the production of N-500 and the C_{12} to C_{14} cut for the production of N-600. The crude is fractionated into these cuts in two towers which comprise Section 150.

Section 200 - Chlorination

The paraffins are chlorinated in Section 200. The feed is heated by exchange with the chlorinated product and then by steam up to 250°F. From the steam heater, the paraffin enters a packed glass-lined steel column where it absorbs residual chlorine from the HCl gas leaving the chlorination reactor. The paraffin is then pumped to the first stage (top) of the chlorination reactor.

Chlorine is received in tank cars, vaporized by steam, and fed to the reactor. The chlorination reactor is a five-section, glass-lined steel tower. Chlorine is fed to each of the sections, and the reaction temperature is maintained between 250°F and 330°F. Temperatures above 330°F cause coking in the reactor. The partially chlorinated paraffin overflows from the first stage through a downcomer into the second stage and out of the chlorinator into the first of two HCl separator drums. The chloroparaffin is cooled from 330°F to 260°F by forced circulation through an air-cooled exchanger and put back into the third stage of the chlorinator. Overflowing through downcomers, the chloroparaffin passes through third, fourth, and fifth stages of the chlorinator. HCl gas from the first four stages and the first separator drum enters a second knockout drum. Chloroparaffins from the fifth stage enter the second knockout drum at 330°F.

Chloroparaffin product goes to the alkylation section after going through a feed-to-product interchanger. HCl gas from the chlorine absorber goes to the benzene absorber in Section 450.

Section 300 - Alkylation

The reaction of chloroparaffins and benzene occurs in Section 300. This is a conventional aluminum chloride-catalyzed Friedel-Krafts reaction. The chloroparaffins are fed to one of two agitated batch reactors on flow control along with aluminum chloride sludge from the slurry drum. Benzene is dried with molecular sieves and fed to the batch reactors on flow control. About 90 percent of the chloroparaffins are reacted at an alkylation temperature of about 165°F. This reaction does not appear to be either exothermic or endothermic.

The reaction mixture is pumped from the batch reactors to the multistage reactor. Upon leaving the multistage reactor, greater than 99+ percent of the chloroparaffins have been reacted. A second multistage reactor is available and can be used to increase the reaction time. However, the second multistage reactor is generally used only as a spare for the

PROCESS DESCRIPTION (CONTINUED)

Section 300 - Alkylation (Continued)

first, since the reaction is essentially complete in the first multistage reactor. Product from the multistage reactor is pumped to a large horizontal drum where most of the catalyst sludge settles out. The crude "NALKYLENE" alkylate product overflows the supersettler and goes to Section 400. HCl gas from the batch and multistage reactors is compressed, absorbed, and purified in Section 450.

Some of the catalyst sludge from the supersettler is purged to the aluminum chloride liquor unit. The remainder of the sludge flows to the slurry drum. Just enough fresh dry aluminum chloride is added to the slurry drum to maintain the unreacted chlorides out of the multistage reactor around 0.2 percent by weight. The catalyst slurry is then pumped back to the batch reactors.

The aluminum chloride liquor unit dilutes the sludge that is purged from the reaction system with water. The aluminum chloride is then recovered as a 26 percent water solution, and the hydrocarbons that were complexed with the aluminum chloride are separated out in a decanter. The considerable amount of heat generated in the process is removed by circulating the mixture through a water-cooled exchanger. This process generally works very well when processing sludge from the "NALKYLENE" Alkylate Unit. However, sludge from the SAU Unit is also processed and sometimes causes problems because of emulsions. The sprung oil is used for fuel after benzene is stripped out. Most of the aluminum chloride liquor produced is sold to sewage treatment plants as a coagulating agent.

Section 400 - Crude Cleanup

Crude "NALKYLENE" alkylate from the supersettler is fed to the HCl stripper which vents to the suction of one of the HCl compressors. Most of the dissolved HCl is removed in the stripper. The crude alkylate then flows to the secondary settler where a small additional amount of catalyst sludge settles out. This sludge is pumped back to the primary settler along with a small amount of the crude alkylate. Thus no sludge layer accumulates in the secondary settler.

Crude alkylate is subjected to a water washing operation to neutralize any remaining aluminum chloride. A settling stage is used to separate the aqueous phase and an emulsion which forms at the interface.

Crude alkylate flows from the phase separator to the first caustic settler through an orifice mixer. A dilute recycle caustic stream from the bottom of the second stage caustic wash is added to the crude just before it enters the mixer. The crude alkylate overflows from the top of the first caustic settler, is mixed with a 5 percent NaOH solution, and then goes to a second stage settler. Neutralized crude from the top of the second stage settler is ready for fractionation and goes to crude storage. Spent caustic is purged from the first stage caustic settler.

PROCESS DESCRIPTION (CONTINUED)

Section 500 - Fractionation

Neutralized crude alkylate is pumped from storage to Section 500 - Fractionation. The crude alkylate is heated by interchange with the paraffin tower vapor before going to the benzene vaporizer. The benzene vaporizer is a kettle reboiler in which some of the excess benzene is flashed. The remaining stream is then fed to the benzene tower where benzene is removed. The bottoms from the benzene tower are pumped to the paraffin tower where the excess paraffin is removed. The alkylate then flows from the bottom of the paraffin tower to the product tower. Product is removed as a sidedraw and pumped to intermediate storage. The bottoms from the product tower are fed to the bottoms stripping tower. The overhead diphenyl alkylate stream from the bottoms tower goes to fuel. The dialkyl-benzene bottoms from the stripping tower are high boiling by-products from alkylation. Some of this material is sold to producers of oil soluble sulfonates.

The paraffin tower, product tower, and bottoms stripping tower all operate under vacuum. The product tower operates at about 10 mm Hg.

Section 600 - Product Cleanup

The distilled "NALKYLENE" alkylate product is pumped from intermediate storage to Section 600 for final cleanup. The product is contacted twice with sulfuric acid which is then separated out with electrostatic separators. After acid separation, the alkylate is contacted with dilute caustic which is also separated in an electrostatic separator. The final cleanup step is to pass the alkylate through a bed of caustic pellets, a bed of activated clay, and a cotton filter before going to finished product storage. The caustic removes traces of water, the clay some of the color, and the filter any remaining foreign particles. The spent acid is returned to the supplier. Most of the dilute caustic that is purged from the system is sold. If necessary, the waste caustic can be neutralized with muriatic acid.

Section 450 - HCl Recovery Section

HCl gas from Section 200 goes directly to the benzene absorber. HCl gas from Sections 300 and 400 is compressed and then sent to the benzene absorber where HCl gas is contacted with some of the chloroparaffin before it goes to Section 300. This removes most of the benzene from the HCl gas.

HCl is absorbed in water in a two-stage water-cooled absorber system. The muriatic acid produced still contains a small amount of benzene, most of which is removed in deoilers. Each deoiler contains three stages of plastic mesh pads. The last traces of benzene are removed from muriatic acid in a large carbon bed familiarly known as a jolly green giant. When the carbon bed is exhausted, as is shown by a significant amount of benzene passing through the bed, the carbon in the bed is dumped. The first muriatic acid through a new bed is high in benzene and is normally run to the neutralization pit. The muriatic acid produced is of very high quality. When sales of muriatic acid do not keep up with production, the excess is neutralized.

Spec. No. 01-3000

CONOCO C_{10}-C_{12} Normal Paraffins

Special Identification: C_{11} average n-paraffins

Test	Specification	Typical Property	Test Method
Total n-Paraffins, Wt. %	97.5 min.	97.7	1.072
Homolog Distribution, Wt. %			1.053
C_9 and Lower	2.0 max.	0.4	-
C_{10}	25.0 max.	18.3	-
C_{11}	-	38.9	-
$C_{10} + C_{11}$	50.0 min.	-	-
C_{12}	25.0 min.	33.9	-
$C_{10} + C_{11} + C_{12}$	85.0 min.	-	-
C_{13}	15.0 max.	8.0	-
C_{14} and Higher	2.0 max.	0.5	-
Aromatics, %	1.0 max.	0.75	1.036
Bromine Number	0.2 max.	0.01	ASTM D-1491-60
Color, Saybolt	25+ min.	30+	1.011
Molecular Weight		160	1.053
Pounds/Gallon (60°F)		6.22	-

3-1-79
(Replaces 4-30-76)

SAL 000010214

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Spec. No. 01-3011

CONOCO C_{12}-C_{14} Normal Paraffins

Special Identification: C_{13} Average n-Paraffins

Test	Specification	Typical Property	Test Method
Total n-Paraffins, Wt. %	97.0 min.	97.5	1.072
Homolog Distribution, Wt. %			1.053
C_{10} and Lower	2.0 max.	0.8	-
C_{11}	-	1.7	-
C_{10} + C_{11}	5.0 max.	-	-
C_{12}	-	10.6	-
C_{13}	-	55.3	-
C_{14}	45.0 max.	30.4	-
C_{13} + C_{14}	70.0-90.0	-	-
C_{15} and Higher	5.0 max.	1.2	-
Aromatics, %	1.2 max.	0.82	1.036
Bromine Number	0.2 max.	0.02	ASTM D-1491-60
Color, Saybolt	25+ min.	30+	1.011
Molecular Weight		186	1.053
Pounds/Gallon (60°F)		6.34	

3-1-79
(Replaces 4-30-76)

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Spec. No. 01-3012

CONOCO C_{12}-C_{16} Paraffins

Special Identification: C_{13} Average n-Paraffins

Test	Specification	Typical Property	Test Method
Total n-Paraffins, Wt. %	97.0 min.	97.3	1.072
Homolog Distribution, Wt. %			1.053
C_{11} and Lower	1.0 max.	0.5	
C_{12}	8-25	11.0	
C_{13}	-	53.0	
C_{14}	-	27.0	
C_{15}	-	7.0	
C_{16}+	-	1.5	
Aromatics, Wt. %	1.5 max.	0.9	1.036
Bromine Number	0.2 max.	0.02	1.012
Color, Saybolt	-	15+	1.011

7-16-79
(Replaces 3-1-79)

SAL 000010216

Spec. No. 01-3020

CONOCO C_{14}-C_{16} Normal Paraffins

Special Identification: C_{14}-C_{16} Normal Paraffins (W-9 Tower Bottoms)
Made at Baltimore Plant

Test	Specification	Typical Property	Test Method
Total n-Paraffins, Wt. %	96.0 min.	96.5	1.053
Homolog Distribution, Wt. % (100% n-paraffins basis):			1.053
C_{13} and Lower	2.0 max.	1.0	
C_{14}	25.0 max.	20.0	
C_{15}	-	61.9	
C_{16}	-	13.1	
C_{17} and Higher	6.0 max.	4.0	
Aromatics, Wt. %	3.0 max.	2.2	1.036
Isoparaffins, Wt. %	5.0 max.	2.4	1.053
Bromine Number	0.15 max.	0.07	1.012
Color, Gardner	7 max.	5	1.022
Appearance, @ 100°F	Clear and free of suspended matter	same	visual
Specific Gravity, @ 60°F		0.7762	1.023
Flash Point, Pensky-Martens, °F		250	ASTM D-93
Melting Point, °F		47.0	1.050
Molecular Weight (on n-paraffins)		215	1.053
Distillation Range, °F			ASTM D-86
IBP		503.0	
90%		518.0	
FBP		546.0	

3-1-79
(Replaces 4-30-76)

SAL 0C0C1C217

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Spec. No. 01-3100

NALKYLENE 500 Linear Alkylbenzene

Special Identification: Linear Dodecylbenzene

Test	Specification	Typical Property	Test Method
Alkylate Homolog Distribution, Wt. %			1.087
$<C_{10}$	0.5 max.	0.1	
C_{10}	25.0 max.	19.2	
$C_{10} + C_{11}$	50.0 min.	57.3	
C_{12}	25.0 min.	33.2	
$C_{10} + C_{11} + C_{12}$	85.0 min.	90.7	
C_{13}	15.0 max.	7.8	
C_{14} and Higher	2.0 max.	0.9	
2-Phenyl Isomer, Wt. %	25.0-35.0	28.3	
Total Material Lighter Than C_{10} LAB, Wt. %	0.5 max.	0.4	1.087
Bromine Number	0.01 max.	0.003	ASTM D-2710-72
Molecular Weight	231-241	237	1.087
Saybolt Color	29 min.	30+	1.011
Completeness of Sulfonation, %	98.0 min.	98.4	1.059
Specific Gravity @ 60°F	0.850-0.870	0.8660	1.023
Water, Wt. %	0.1 max.	0.004	1.016
Organic Chloride, Wt. %	0.1 max.	0.05	5.004
Color of Sodium Sulfonate, Klett (5% AI) (SO_3 Derived)	35 max.	33	1.042
Biodegradability	must pass	pass	SDA test[1]
Pounds/Gallon, 60°F	-	7.214	

[1] Soap and Detergent Association procedure for the determination of ABS and LAS biodegradability (JAOCS, Nov. 1965 issue, Vol. 42, No. 11, pages 986-993).

10-15-79
(Replaces 9-4-79)

SAL 0C001C219

This sheet is CONFIDENTIAL and should not be distributed outside Conoco Inc.

Spec. No. 01-3101

NALKYLENE 550 Linear Alkylbenzene

Special Identification: 244 Molecular Weight Linear Alkylbenzene

Test	Specification	Typical Property	Test Method
Alkylate Homolog Distribution, Wt. %			1.087
$<C_{10}$	0.5 max.	nil	
C_{10}	5-15.0	12.7	
C_{11}	-	29.5	
$C_{10} + C_{11}$	30-50.0	42.2	
C_{12}	-	30.5	
C_{13}	-	18.3	
C_{14}	10.0 max.	7.4	
$C_{13} + C_{14}$	30.0 max.	25.7	
$>C_{14}$	2.0 max.	0.9	
2-Phenyl Isomer, Wt. %	25-35.0	26.5	
Total Material Lighter Than C_{10} LAB, Wt. %	0.5 max.	0.3	1.087
Average Molecular Weight Range Preferred	240-248 244	244	1.087
Bromine Number	0.01 max.	0.003	ASTM D-2710-72
Saybolt Color	29 min.	30+	1.011
Completeness of Sulfonation, %	98.0 min.	98.3	1.059
Doctor Test	negative	negative	ASTM D-484
Specific Gravity (60°F/60°F)	0.860-0.870	0.8658	1.023
Water, Wt. %	0.1 max.	0.004	1.016
Organic Chloride, Wt. %	0.1 max.	0.05	5.004
Color of Sodium Sulfonate, Klett (5% AI) (Oleum Derived)	60 max.	50	1.042
Biodegradability	must pass	pass	SDA test[1]
Pounds/Gallon, 60°F	-	7.205	

[1]Soap & Detergent Association procedure for the determination of ABS and LAS biodegradability (JAOCS, Nov. 1965 issue, Vol. 42, No. 11, pages 986-993).

10-15-79
(Replaces 9-4-79)

SAL 000010219

This sheet is CONFIDENTIAL and should not be distributed outside Conoco Inc.

Spec. No. 01-3104

NALKYLENE 600 Linear Alkylbenzene

Special Identification: Linear Tridecylbenzene

Test	Specification	Typical Property	Test Method
Homolog Distribution, Wt. %			1.087
C_{10}	2.0 max.	0.7	
$C_{10} + C_{11}$	5.0 max.	4.2	
C_{14}	45.0 max.	26.5	
$C_{13} + C_{14}$	70-90	75.7	
$>C_{14}$	5.0 max.	3.2	
2-Phenyl Isomer, Wt. %	20-30	23.5	
Total Lighter Than C_{10} LAB, Wt. %	0.5 max.	0.3	1.087
Average Molecular Weight Preferred	258-266 262	261	1.037
Bromine Number	0.01 max.	0.003	ASTM D-2710-72
Saybolt Color	29 min.	30+	1.011
Completeness of Sulfonation, %	97.5 min.	98.0	1.059
Doctor Test	negative	negative	ASTM D-484
Specific Gravity @ 60°F	0.860-0.870	0.8651	1.023
Water, Wt. %	0.1 max.	0.004	1.016
Organic Chloride, Wt. %	0.1 max.	0.05	5.004
Color of Sodium Sulfonate, Klett (5% AI)[1] (Oleum Derived)	90 max.	76	1.042
Biodegradability	must pass	pass	SDA test[2]
Pounds/Gallon, 60°F	-	7.206	

[1]Not for release outside Conoco Inc.

[2]Soap & Detergent Association procedure for the determination of ABS and LAS biodegradability (JAOCS, Nov. 1965 issue, Vol. 42, No. 11, pages 986-993).

10-15-79
(Replaces 9-4-79)

SAL 000010220

Spec. No. 01-3400

Hydrochloric Acid

Special Identification: Technical Grade 20° Baume, 31.45% HCl

Test	Specification	Typical Property	Test Method
Assay, Baume	20.0°-20.4°	20.2°	1.035
Sulfite, ppm[1]	10 max.	4	1.035
Sulfate, ppm	15 max.	10	1.035
Iron, ppm (as Fe)	1.0 max.	0.2	1.035
Free Chlorine, ppm	1.0 max.	*	1.035
Arsenic, ppm	0.05 max.	*	1.035
Sodium, ppm[1]	6 max.	5	1.035
Aluminum, ppm[2]	1 max.	0.5	1.035
Heavy Metals, ppm (as Pb)	1.0 max.	0.5	1.035
Color, APHA	15 max.	10	1.035
Nonvolatile Matter, ppm	200 max.	90	1.035
Residue on Ignition, ppm	100 max.	70	1.035
Organic Matter, ppm[3]	2.0 max.	0.5	1.035
Odor	Characteristic		

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*Nondetectable.
[1]Specification for Foote Mineral only.
[2]Specification for Diamond Shamrock only.
[3]One ppm maximum for J. T. Baker only.

3-1-79
(Replaces 5-16-78)

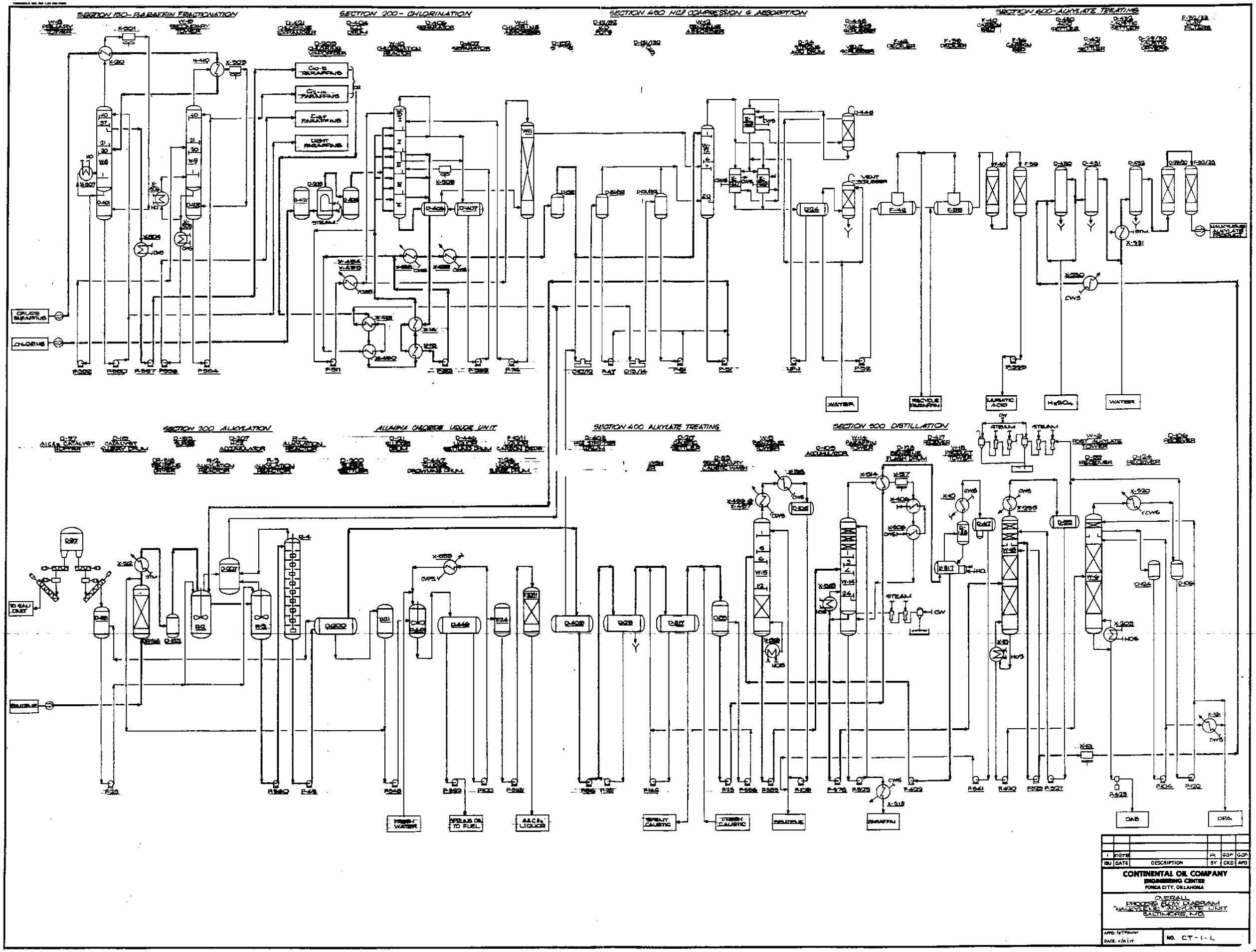
Spec. No. 01-3450

Aqueous $AlCl_3$ Regular Grade

Special Identification: Aluminum Chloride

Test	Specification	Typical Property	Test Method
Aluminum Chloride, Wt. %	23.0 min.	24.0	1.077
Hydrogen Chloride, Wt. %	3.0 max.	1.5	1.077
Organic Matter, ppm	30 max.	5	1.077
Iron, ppm	800 max.	300	1.077

3-1-79
(Replaces 8-15-73)



SAL 000010223

PED ORIENTATION PROGRAM

STXS UNIT

BALTIMORE CHEMICAL PLANT

BALTIMORE, MARYLAND

The STXS Unit manufactures hydrotropes for use in the production of liquid detergent formulations. The unit is run batchwise in several steps on blocked-out operation to produce one of three products. Production capability is 13-15 MM pounds per year of sodium toluene sulfonate (STS), sodium toluene xylene sulfonate (STXS), and ammonium xylene sulfonate (AXS).

GENERAL DESCRIPTION

The purpose of this unit is to produce a 40 percent aqueous solution of sodium toluene sulfonate by sulfonation of toluene with 99 percent sulfuric acid and neutralization of the sulfonic acid with 50 percent caustic. One hundred percent excess toluene is used in the reaction to assure that most of the sulfuric acid is consumed, thereby minimizing the Na_2SO_4 formed on neutralization.

Sulfonation

$$R\text{-}H + H_2SO_4 \longrightarrow \underset{\text{Sulfonic Acid}}{R\text{-}SO_3H} + H_2O$$

where R-H is toluene or xylene.

Neutralization

$$R\text{-}SO_3H + NaOH \longrightarrow \underset{\text{SXS or STS}}{R\text{-}SO_3Na} + H_2O$$

$$R\text{-}SO_3H + NH_4OH \longrightarrow \underset{\text{AXS}}{R\text{-}SO_3NH_4} + H_2O$$

PROCESS DESCRIPTION (The following description is typical for STS production)

Sulfonation Section

There are two parallel systems with the same function and capacities in the sulfonation section. Each consists of a standard glass-lined reactor, an overhead condenser, and a water separator.

The reactants are measured by means of positive displacement meters with automatic shutoff valves. The reaction mixture is heated to the boiling point (230°F) with steam and held there during the course of the reaction. Water of reaction and toluene are removed overhead as vapors which are condensed. The condensate flows into the separator from which toluene overflows continuously and passes back to the reactor by gravity. The

PROCESS DESCRIPTION (CONTINUED)

Sulfonation Section (Continued)

water flows continuously out the bottom and is collected in measuring drums. Heat is applied to the reaction mixture until 80 percent of the water of reaction and all of the water in the sulfuric acid are removed. The reaction mixture is then cooled to 100°F and transferred to the nuetralizer.

Neutralization Section

Water is added to the sulfonic acid and toluene in the neutralizer in order to make a 40 percent aqueous solution of the final product. Heat is evolved, and the temperature increases to 160°F. Fifty percent NaOH is injected into the sulfonic acid for neutralization, and the material is circulated through a heater-cooler to hold it at 200°F, thereby avoiding vaporization of the excess toluene. The sulfonate formed separates from the toluene on settling as the bottom layer, which is pumped out to the slurry rundown tank. The intermediate layer, or cuff, is withdrawn to an accumulator for the recovery of any good product and toluene. The top toluene layer is cooled and pumped to storage. The toluene is separated mechanically instead of by distillation to avoid a concentration of ditolyl sulfone in the product which causes insolubility in water. The sulfonate product is returned to the neutralizer and heated to remove any dissolved toluene. The toluene vapor passes overhead and is condensed by direct contact with a cold water spray. The product is then cooled and pumped to the slurry rundown tank. It is transferred to heated storage after control tests are run.

The same operational procedures are used for the production of sodium xylene sulfonate and ammonium xylene sulfonate. For the latter case, ammonium hydroxide is used as the neutralizing agent.

PRODUCTS

The hydrotrope products are sold as 40 percent solutions in water to liquid detergent manufacturers. They use the hydrotropes at levels of 1-3 percent to maintain other components of the formulations in solution. Hydrotropes also contribute wetting and cleansing characteristics. They are clear solutions with a slight yellow tint.

Spec. No. 01-3200

CONOCO AXS
Ammonium Xylene Sulfonate

Special Identification: Hydrotrope

Test	Specification	Typical Property	Test Method
Active, Wt. %	40 min.	42.1	1.018
Active/Total Solids	0.93 min.	0.93	Calculated
Iron, ppm (as Fe) (Solids Basis)	5.0 max.	0.2	1.019
Copper, ppm	1.0 max.	0.1	-
Sulfate (as SO_4), Wt. %	-	2.9	1.052
Water, Wt. %	-	55.0	1.018
Chloride (as Cl^-), Wt. %	0.1 max.	0.03	1.010
Free Oil, Wt. %	0.10	0.03	1.002
pH, "as is"	7.0-8.5	7.7	2.008
Color, Klett	40 max.	21	1.042
Flash Point (Pensky-Martens, °F)	Shall not flash @ 200	Above 210	ASTM D-93
Odor	Pass	Pass	Baltimore
Davis Vaportester Reading	0.5 max.	-	Baltimore
Pounds/Gallon @ 60°F	-	9.5	1.023

3-1-79
(Replaces 10-1-72)

Spec. No. 01-3210

CONOCO SXS
Sodium Xylene Sulfonate

Special Identification: Hydrotrope

Test	Specification	Typical Property	Test Method
Active, Wt. %	40.0 min. 42.0 max.[1]	41.4	1.018
Active/Total Solids[2]	0.93 min.	0.97	Calculated
Water, Wt. %	-	57.7	1.018
Chloride (as Cl^-), Wt. %	0.1 max.	0.03	1.010
Sodium Sulfate, Wt. %	-	1.16	1.052
Free Oil, Wt. %	0.1 max.	0.01	1.002
Iron (as Fe) (solids basis), ppm	5 max.	0.20	1.019
Color, Klett	40 max.	21	1.042
pH, "as is"	7.5-10.5	9.7	2.008
Flash (Pensky-Martens, °F)	Shall not flash @ 200	210	ASTM D-93
Odor	Passes	Pass	Baltimore (Lever)
Pounds/Gallon, 60°F	-	9.84	1.023
Davis Vaportester Reading	0.5 max.	Nil	Baltimore (Lever)

[1]For Texize only.
[2]Total solids - active + inorganic salts.

3-1-79
(Replaces 3-1-74)

Spec. No. 01-3250

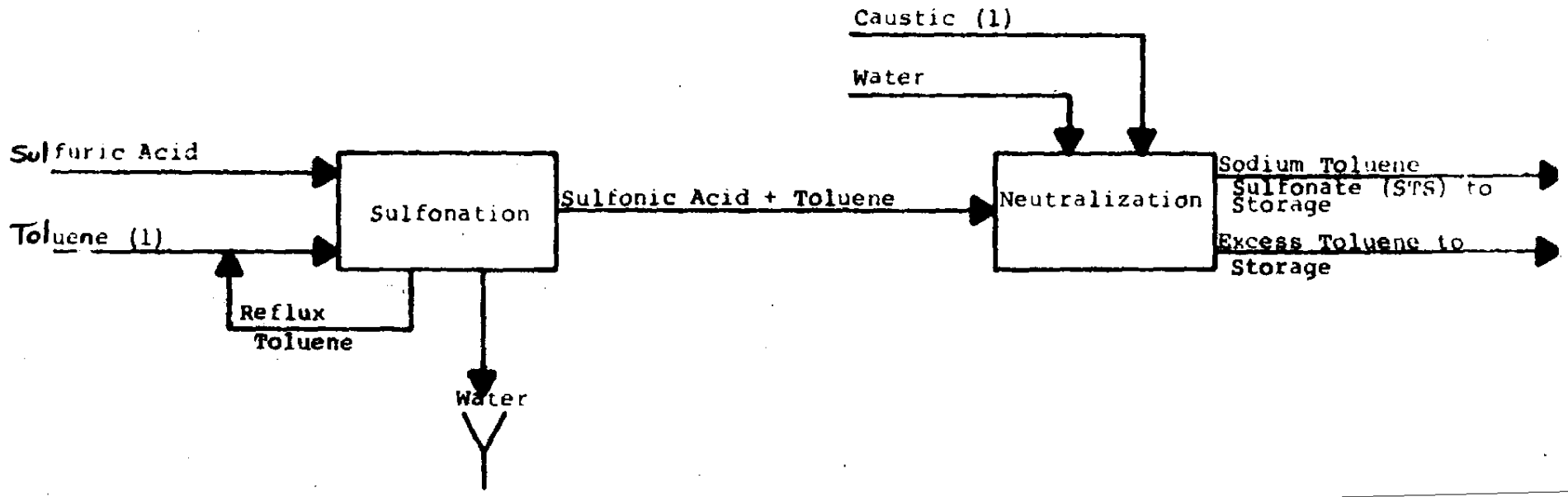
CONOCO STXS
Sodium Toluene Xylene Sulfonate

Special Identification: Hydrotrope

Test	Specification	Typical Property	Test Method
Active, Wt. %	40.0 ± 2.0	41.5	1.018
Water, Wt. %		56.4	1.018
Sodium Sulfate, Wt. %		2.0	1.052
Chloride (As Cl^-), Wt. %	0.1 max.	0.03	1.010
Free Oil, Wt. %	0.1 max.	0.03	1.002
Iron, ppm	5.0 max.	0.20	1.019
Copper, ppm	1.0 max.	0.20	1.045
Crystallization Temperature, °C	5-15	14	1.020
Color, Klett, "as is"	40 max.	26	1.042
Ph, "as is"	7.5-10.5	9.7	2.008
Flash (Pensky-Martens, °F)	Shall not flash @ 200	Above 210	ASTM D-93
Active/Total Solids[1]	0.88 min.	0.92	Calculated
Pounds/Gallon, 60°F	-	9.94	1.023
Sodium Toluene Sulfonate, % of Active	25 max.	20	1.074
Odor	Pass	Pass	Baltimore
Davis Vaportester Reading	0.5 max.	Nil	Baltimore

[1]Total solids = active + inorganic salts.

3-1-79
(Replaces 10-1-72)

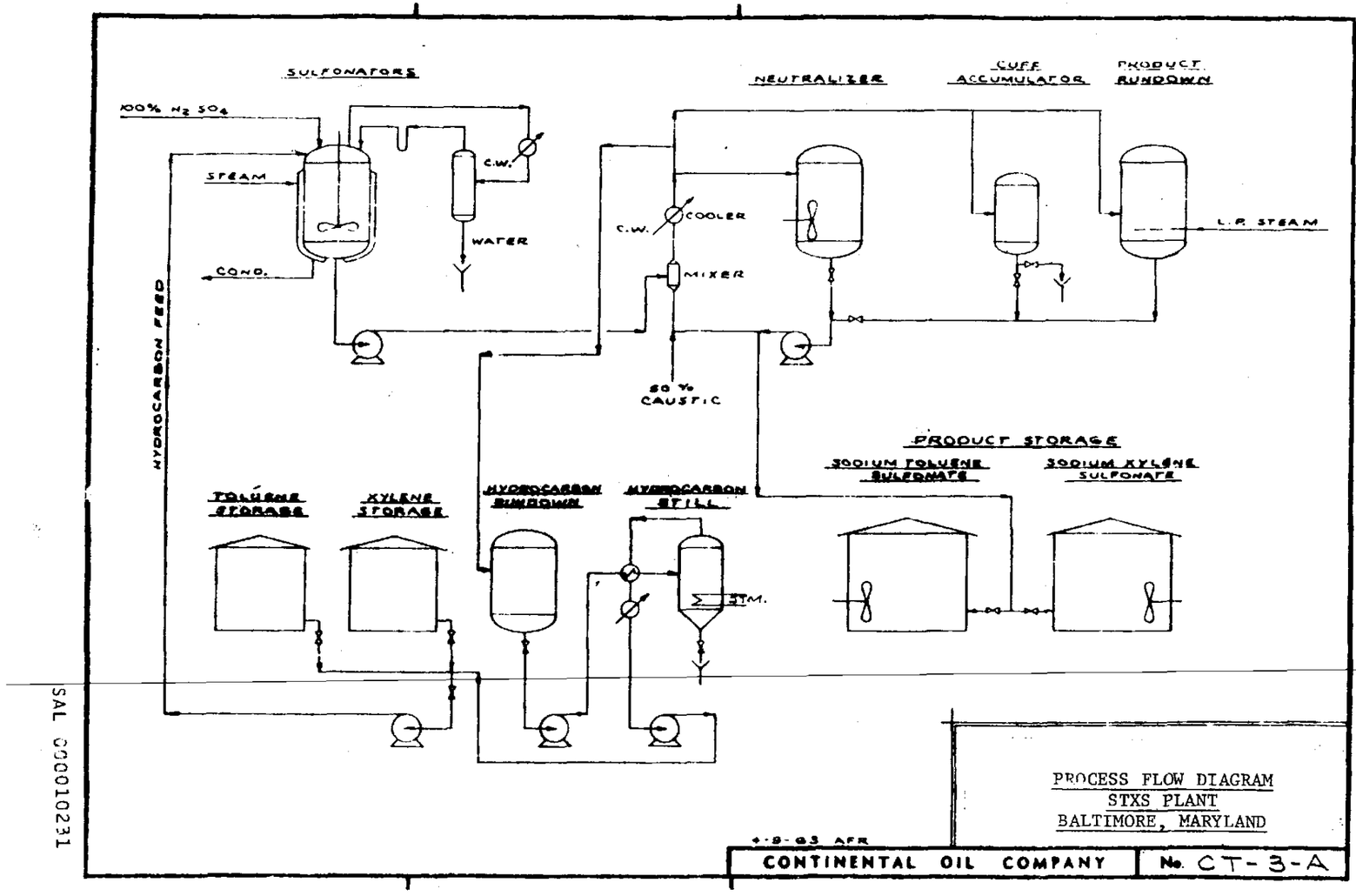


(1) Type of Sulfonate Dependent on Feedstock

Hydrocarbon Feedstock	Neutralizing Agent	Product
Toluene	Caustic	Sodium Toluene Sulfonate (STS)
Xylene	Caustic	Sodium Xylene Sulfonate (SXS)
Xylene	Aqua Ammonia	Ammonium Xylene Sulfonate (AXS)

BLOCK FLOW DIAGRAM
STS, SXS, AXS PRODUCTION
STXS UNIT
BALTIMORE, MARYLAND

CT-50-A



SAL 00010231

Hammond Plant

SAL 0000010232

PED ORIENTATION PROGRAM

HAMMOND CHEMICAL PLANT

HAMMOND, INDIANA

The Hammond plant began production in 1969 using the new Chemithon Sulfonation Process. Operation had existed at the site using a batch process since 1958. This operation was discontinued when the new continuous process came on-stream. Throughput capability has been increased to 70 MM pounds per year from the original 30-40 MM pounds per year. The increase was achieved both through optimization of the process and through evolution of different product slates. The plant uses as feedstocks linear alkylbenzenes from Baltimore and "ALFONIC" ethoxylates from Lake Charles. Sulfur trioxide is purchased from an adjacent Stauffer Chemical Company plant. Other feedstocks include sodium hydroxide, ammonium hydroxide, ethyl alcohol, and "ALFOL" alcohols.

GENERAL DESCRIPTION

Sulfonation/Sulfation

Feedstock (alkylbenzene, ethoxylate, or alcohol) is charged to parallel thin film reactors. Sulfur trioxide, in a carrier air stream, is injected into the reactor also. The exothermic reaction mass is cooled by water in the reactor jackets. Reactor effluent, consisting of liquid sulfonation products and air, is phase separated in a cyclone. Effluent gases are normally passed through knockout drums and transferred across the fence to Stauffer Chemical Company where SO_2 and SO_3 mists are burned to recover the sulfur. When the Stauffer unit is not running, the effluent gases are vented to the atmosphere through an SO_2 scrubber.

Crude sulfonates (alkylbenzene derivatives) from the cyclone are subjected to further contacting in "digestors" to complete the reaction of any dissolved sulfur trioxide. Following the digestors, alkylbenzene derivatives are contacted with water in a "hydrator." This converts any anhydrides back to the sulfonate form. The sulfonate or sulfonic acid is then sent to storage. Sulfates (alcohol or ethoxylate derivatives) are immediately neutralized to avoid chemical decomposition of the unstable sulfate.

Sulfonation/Sulfation Chemistry

a. Alkylbenzene Sulfonation

b. Ethoxylated Alcohol Sulfation

$$R\text{-}(C_2H_4O)X\text{-}CH_2\text{-}OH(L) + SO_3(g) \xrightarrow{Air} R\text{-}(C_2H_4O)CH_2\text{-}O\text{-}CH_3H(L)$$

Neutralization

Neutralization is accomplished through injection of NaOH or NH_3OH into the sulfonate or sulfate as it enters a centrifugal pump. The pump and downstream piping constitute the "reactor". The heat of reaction is removed in water-cooled exchangers. Sodium hypochlorite is then injected to bleach the stream. Sodium xylene sulfonate may also be injected at this stage, depending on the product being made. Sodium carbonate or sodium bicarbonate is added to the ether sulfate product as a pH buffer.

Neutralization Chemistry

a. Alkylbenzene Sodium Sulfonate (Sulfonic Acid)

b. Ethoxylated Alcohol Sodium Sulfate (Ether Sulfate)

$$R\text{-}(C_2H_4O)_X\text{-}CH_2\text{-}O\text{-}SO_3H + NaOH \longrightarrow R\text{-}(C_2H_4O)_X\text{-}CH_2\text{-}O\text{-}SO_3Na + H_2O$$

PRODUCTS

Sulfonates and sulfates are utilized as surface active agents (surfactants) in biodegradable laundry powders, dishwashing liquids, and shampoos. Hammond's products include:

Conoco SA-597 Sulfonic Acid	- Derived from N-500 Alkylbenzene
Conoco C-550 Slurry	- Sodium Sulfonate of SA-597
Conoco C-560 Slurry	- C-550 Blend with Sodium Xylene Sulfonate
"ALFONIC" 1412-A	- Ammonium Salt of Alcohol Ether Sulfate
"ALFONIC" 1412-S	- Sodium Salt of Alcohol Ether Sulfate

This sheet is CONFIDENTIAL and should not be distributed outside Continental Oil Company.

Spec. No. 02-3300

CONOCO SA-597 Sulfonic Acid

Special Identification: SO_3 sulfonated NALKYLENE 500 alkylate

Test	Specification	Typical Property	Test Method
Active, Wt. % a.	96 min.		1.001
b.	97.5 min.	97.5	Calculated (1)
Sulfuric Acid, Wt. %	1.6 max	1.3	1.052
Free Oil, Wt. % (2)	2.5 max		1.021
Unsulfonated Oil, Wt. %	1.0 max	0.7	1.063
Water, Wt. %	1.0 max	0.4	1.016
Sulfur Dioxide (SO_2), Wt. % (4)	0.06 max	0.01	1.066
Acid Color, Klett (5% active acid)	70 max	55	1.054
Iron, ppm (3)	10 max	2	1.019

(1) Active, Wt. % = 100% - (sulfuric acid, wt. % + unsulfonated oil, wt. % + water, wt. %)

(2) Internal specification—not to be used outside Conoco.

(3) For B. F. Goodrich only.

(4) For P & G only.

7-15-77
(Replaces 4-10-77)

SAL 000010235

Spec. No. 02-3351

CONOCO C-550 Slurry

Special Identification: Sodium sulfonate of SA-597

Test	Specifications	Typical Property	Test Method
Active, Wt. %	50-53 (1)	52	1.001
Sodium Sulfate, Wt. %	1.3 max.	1.05	1.052
Free Oil, Wt. %	1.2 max.	1.1	1.021
	1.1 max. (1)	1.05	-
Color, Klett (5% solution)	50 max.	32	1.014
pH, 5% aqueous solution	7-8	7.5	1.047
	7.0-8.5 (1)	-	1.047

(1) For Gateway Only - Add buffer, 0.05%-0.1% Sodium Bicarbonate

4-10-77
(Replaces 10-1-72)

Spec. No. 02-3353

CONOCO C-560 Slurry

Special Identification: Blend of sodium sulfonate of SA-597 and sodium xylene sulfonate

Test	Specification	Typical Property	Test Method
Total Active, Wt. % (LAS + SXS)	57-60	59	Calculated[1]
Solids, Wt. %	63.1 max	61.2	1.064
Sodium Xylene Sulfonate, Wt. %	1 min[3,4] 4 max	2	1.040
Free Oil, Wt.%	1.6 max[2]	1.35	1.021
Sodium Sulfate, Wt. %	1.5 max	1.0	1.052
pH, 5% aqueous solution	7-8	7.5	1.047
Color, Klett, 5% active	50 max	40	1.042
Iron, ppm	5 max	3	1.019
Phosphate (as P_2O_5) ppm	40 max	2	1.067

(1) Active, Wt. % = Solids, Wt. % - (Sodium Sulfate, Wt. % + Free Oil, Wt. %)

(2) 1.2 Max. for Colgate and Wyandotte

(3) For Colgate only -- 1.5-2.5% SXS

(4) For Wyandotte only -- 2.0-2.5% SXS

7-15-77
(Replaces 2-25-77)

Spec. No. 06-4560

ALFONIC 1412-A Alcohol Ether Sulfate

Special Identification: Ammonium Salt of ALFONIC 1412-40 (Spec. No. 06-4360)

Test	Specification	Typical Property	Test Method
Active, Wt. %	58.0-).0	59.2	1.009
Solids (Active + Free Oil + Salts), Wt. %	62.0 ± 1.5	61.0	Calculated
Ethanol, Wt. %	14.0 ± 1.0	13.7	1.057
Unsulfated (Ethoxylate + Alcohol)	3.0 max.	2.2	1.038
Total Inorganic Salts, Wt. %	2.5 max.	<1.0	Calculated
Alcohol Insoluble, Wt. %	0.75 max.	0.3	1.052
Cl as NH_4Cl, Wt. %	0.25 max.	<0.1	1.010
Iron, ppm	5 max.	2	1.019
Copper, ppm	0.5 max.	0.3	1.045
Peroxide Content, ppm	10.0 max.	nil	1.044
pH	7.0-7.5 (1)	7.2	1.043
Color, Klett, 5% Solution	50 max.	30	1.042
Color, Gardner (Purex)	3 max.	-	
Corrosion Test	Pass	-	
Odor	Equal to Standard	-	
Appearance	Clear to slightly viscous liquid		
Molecular Weight	423-444	436	
Ca + Mg as $CaCO_3$	5 max.	<5	Colgate Method
Water, Wt. %	20.0-29.0	25	Calculated

(1) 0.2% Na_2CO_3 as buffer

1-26-76
(Replaces 2-7-73)

This sheet is CONFIDENTIAL and should not be distributed outside Continental Oil Company

Spec. No. 06-4565

ALFONIC 1412-S Alcohol Ether Sulfate

Special Identification: Sodium Sulfate of ALFONIC 1412-40 (Spec. No. 06-4360)

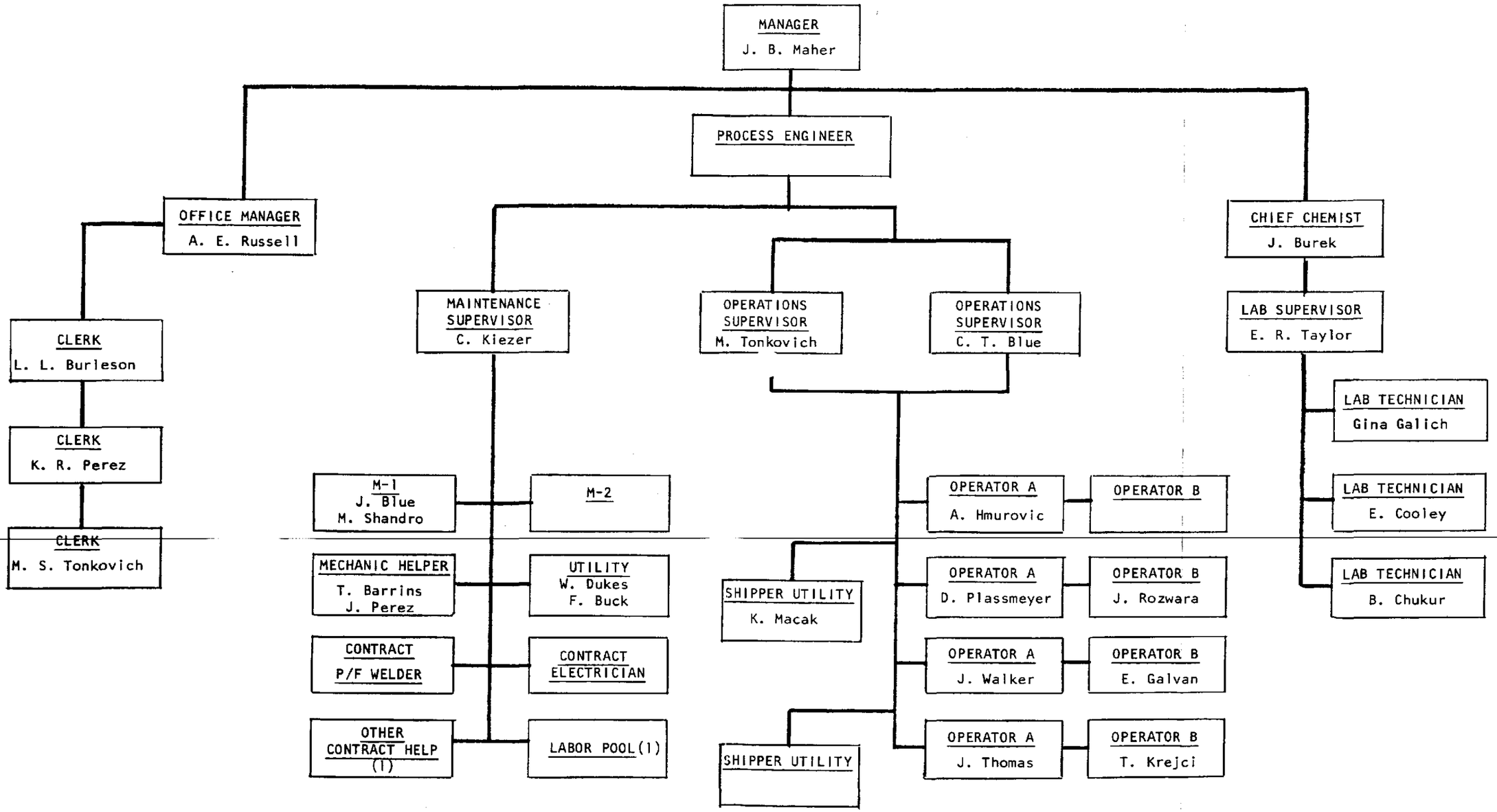
Test	Specifications	Typical Property	Test Method
Active, Wt. %	58.0-60.0		1.009
Ethanol, Wt. %	13.0-15.0		1.057
Unsulfated (Ethoxylate + Alcohol), Wt. %	3.0 max.		1.038
Total Inorganic Salts, Wt. %	2.5 max.		Computed
Na_2SO_4, Wt. %	1.0 max.		1.052
NaCl, Wt. %	1.5 max.		1.010
Iron, ppm	5 max.		1.019
Copper, ppm	0.5 max.		1.045
pH, 1% Solution	8-9 (1)		1.047
Color, Klett, 5% Solution	50 max.		1.042

(1) Buffer required: 0.2% to 0.3% Na_2CO_3 recommended

10-1-72

ORGANIZATIONAL CHART

AMMOND C .ICAL PLANT



(1) There may or may not be any people in these positions.
They are listed to show reporting lines.

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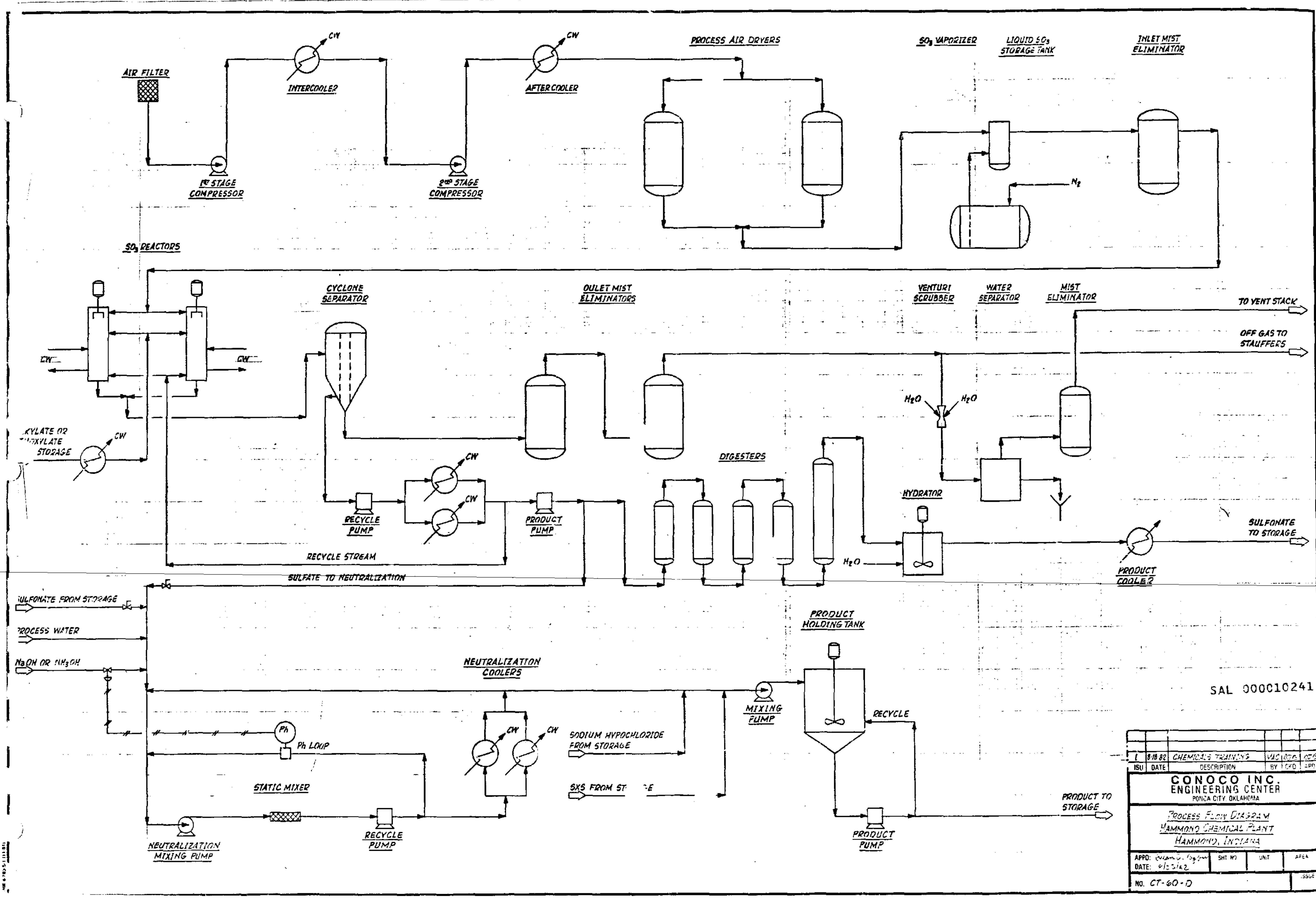
AIR FILTER
1ST STAGE COMPRESSOR
INTERCOOLER
CW
2ND STAGE COMPRESSOR
AFTERCOOLER
PROCESS AIR DRYERS
SO_3 VAPORIZER
LIQUID SO_3 STORAGE TANK
N_2
INLET MIST ELIMINATOR
SO_3 REACTORS
CYCLONE SEPARATOR
OULET MIST ELIMINATORS
VENTURI SCRUBBER
WATER SEPARATOR
MIST ELIMINATOR
TO VENT STACK
OFF GAS TO STAUFFERS
H_2O
KYLATE OR
KYLATE STORAGE
RECYCLE PUMP
PRODUCT PUMP
RECYCLE STREAM
DIGESTERS
HYDRATOR
SULFONATE TO STORAGE
PRODUCT COOLER
SULFATE TO NEUTRALIZATION
ULFONATE FROM STORAGE
ROCESS WATER
NaOH OR NH_3OH
PRODUCT HOLDING TANK
NEUTRALIZATION COOLERS
MIXING PUMP
RECYCLE
Ph
Ph LOOP
SODIUM HYPOCHLORIDE FROM STORAGE
SXS FROM ST E
STATIC MIXER
NEUTRALIZATION MIXING PUMP
PRODUCT TO STORAGE
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8-18-82
CHEMICALS TRAINING
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DESCRIPTION
BY
CONOCO INC.
ENGINEERING CENTER
PONCA CITY, OKLAHOMA
PROCESS FLOW DIAGRAM
HAMMOND CHEMICAL PLANT
HAMMOND, INDIANA
APPD:
DATE:
SHT NO
UNIT
AREA
NO. CT-60-D
ISSUE

Newark Plant

SAL 0000010242

PED ORIENTATION PROGRAM

NEWARK CHEMICAL PLANT

NEWARK, NEW JERSEY

The plant, owned originally by Reilly Tar and Chemical Company, was purchased by Consolidated Coal Company (CONSOL) in August 1955 as part of CONSOL's broad program to create a group of satellite plants which would refine and market the crude chemical streams derived from the processing of coal. It was contemplated that the Newark plant would fit into this program by first refining and marketing cresylics derived from petroleum industry feedstock (spent caustic from gasoline washing), with this raw material being supplemented at a later date by coal-derived feedstocks.

At the time of purchase from Reilly, the plant produced only about 7 MM pounds per year of cresylic acid using an old cumbersome process; however, in 1956, a new facility, the "natural acid" plant, was built utilizing a novel extraction process. The new plant was designed for 14 to 15 MM pounds per year of cresylics; but with relatively minor changes, this capacity was increased to over 33 MM pounds per year. The new extraction process also separated a sizable quantity of aromatic sulfur compounds from the feedstock. Unfortunately, a market was not immediately available for these by-products, so they had to be incinerated. Attempts to market the thio-compounds as rubber peptizers were unsuccessful due to the materials' extremely unpleasant odor. Research and Development efforts showed that this problem could be solved by alkylating the compounds. A new plant, the "alkylation plant," was built in 1961 where these compounds were alkylated. Some cresylics were also upgraded by alkylation in this plant.

Advances in the technology for desulfurizing gasoline in the petroleum industry reduced the available cresylic feedstock supply, forcing the plant to consider synthetic processes for producing the cresylic acids. A grass roots "synthetic unit" was built in 1966 to synthesize cresylics from methanol and phenol.

In 1969, the plant came under the management of Conoco's Chemicals Division.

Unlike the "natural plant," the "synthetic unit" primarily produced orthosubstituted compounds with very little of the meta and para compounds. In order to convert some of the product from the synthetic plant to meta and para type material, an "isomerization unit" was built in 1969. This unit, however, never did go into commercial operation because low-priced meta/para cresols became available from Japan at the time the unit was scheduled to go on stream, rendering operation of the unit unprofitable.

Increasing pressure from environmental agencies, decreasing availability of spent caustic feedstock from the petroleum industry, and low product sales volumes resulted in shutdown of the "natural plant" and reduced operation in the alkylation plant in 1971. Coal-derived feedstock never did become available to the "natural plant" and the plant remains idle.

A new high pressure methylation unit was started up in mid-1978. It was designed to produce 4.5 MM pounds per year of 2,3,6-trimethylphenol by reacting 2,6-xylenol with methanol. It was designed to operate alternatively, producing ortho-cresol from phenol and methanol. This reaction step is conducted in the liquid phase rather than the vapor phase as in the "synthetic unit."

The alkylation unit was shut down in late 1980 as product sales prices were unable to keep abreast of rising material and operating costs.

GENERAL DESCRIPTION

Only the Synthetic Unit and Liquid Methylation Unit are currently being operated. However, processes formerly carried out in the equipment now idle (the "Natural Plant" and the "Isomerization Unit") are also described here.

A. Natural Plant

The Natural Plant employed a process for extracting cresylic acids and aromatic mercaptans from the caustic feedstocks. Therefore, it principally contained only separation equipment and did not have a reactor. A block flow diagram of the process is shown in drawing CT-4-A.

The feedstock was generally obtained from caustic washing of gasoline to remove cresylic acids and mercaptans. Those compounds were removed from gasoline to prevent gum deposits in engines during combustion. The principle reaction in the washing step was:

$$\underset{\text{Cresylic Acids}}{\text{R-OH}} + \underset{\text{Aromatic Mercaptans}}{\text{R-SH}} + \text{NaOH} \longrightarrow \text{R-ONa} + \underset{\text{Carbolate}}{\text{R-SNa}} + H_2O$$

The resultant product mixture was referred to as carbolate or spent caustic and usually contained 10 to 20 percent by weight cresylic acids and mercaptans.

At Newark, the first step in the refining process was to remove the sodium. In a combination springing tower and acid decanter, the carbolate was countercurrently contacted with the boilerhouse flue gas which had been scrubbed free of SO_2. The principal reaction was:

$$\text{R-ONa} + \text{R-SNa} + CO_2 \xrightarrow{\text{Heat}} \underset{\text{Sprung Acid}}{\text{R-OH} + \text{R-SH}} + Na_2CO_3$$

As the sprung acid left the decanter, it contained about 18 percent by weight sodium carbonate. This was removed by two water washes operated in series. The contaminated acid was mixed with an equal quantity of water and the carbonate-water and acid phases allowed to separate in a settling drum. Then the acid was withdrawn from the bottom and the procedure repeated in the next drum. Carbonate recovered from the first drum was decanted and sent to storage to be used for flue gas scrubbing. The very dilute carbonate solution recovered from the second drum was used as wash water for the first drum.

GENERAL DESCRIPTION (CONTINUED)

A. Natural Plant (Continued)

The washed sprung acid was charged to a distillation tower for topping and the removal of heavy salts and petroleum fractions. All overhead and bottom waste streams were incinerated. The product, or heart-cut, was then ready for extraction.

A 35-stage Scheibel liquid-liquid extraction tower was used to separate the cresylic acids from the mercaptans. Each stage was composed of a twelve-inch calming zone packed with stainless steel mesh and a six-inch high mixing zone agitated by an impeller. The feed entered near the top and was contacted with aqueous methanol and hexane which were introduced at the top and bottom, respectively. The cresylic acids were picked up by the methanol and the mercaptans by the hexane.

In the solvent recovery area, methanol and hexane were fractionated from their respective product streams. Water in a fixed ratio was added to the methanol. The hexane was scrubbed with caustic to remove traces of mercaptans and was then water washed. Both solvents were returned to storage for reuse.

The final acid products were obtained by continuous or batch fractionation. Process conditions could be changed enough to allow considerable flexibility in product composition. Some of the products were then upgraded by alkylation while others were ready for shipment.

The aromatic mercaptans from the hexane recovery tower were sent to the alkylation plant for further processing.

B. Alkylation Plant

The Alkylation Plant was composed of a continuous distillation column, batch still column, batch still reboiler, and batch reactor as shown in drawings CT-5-A and CT-6-A.

Fractionation of the aromatic mercaptan stream from the natural plant (shown in drawing CT-4-A) was accomplished in the system shown in drawing CT-5-A. This stream was fed continuously into the first column from which the overhead went to storage, and the bottoms were either further fractionated or stored, depending upon the desired product. All preparation of the mercaptans for the reactor occurred in the columns shown.

The plant alkylated many different compounds, mostly on a small volume basis. Therefore, a batch reactor offered the most flexibility. (The reactor section will be discussed on a general basis.) There were three possible types of feedstocks: aromatic mercaptans, cresylic acids, or phenol. The catalysts used depended on the feedstock and product required. As shown in drawing CT-6-A, the feedstock was alkylated with isobutylene which was bubbled into the reactor beneath the agitator. The operating conditions were mild, usually 300-400°F, and a few inches of water vacuum.

GENERAL DESCRIPTION (CONTINUED)

B. Alkylation Plant (Continued)

If fractionation of the reactor products was required, the batch still was used.

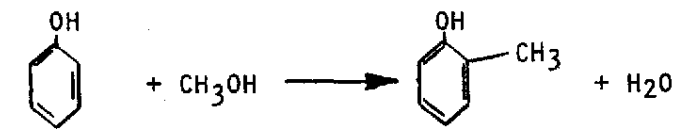
Most recently, the alkylation unit was operated on a limited basis for alkylating some phenol and cresylic acids, mainly, o-cresol and 2,4-xylenol. The unit is currently shutdown.

C. Synthetic Unit (See Drawing CT-56-D, "Overall Process Flow Diagram")

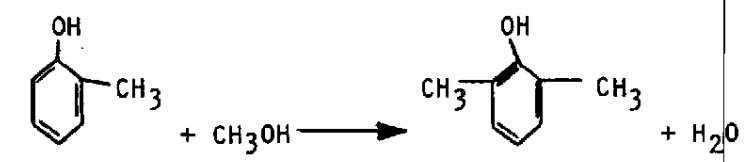
The Synthetic Unit is comprised of two process sections--reaction and fractionation.

1. Reaction Section

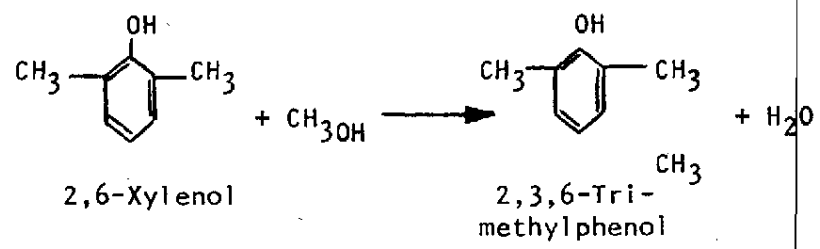
The main reaction carried out is the vapor phase alkylation of phenol by methanol to yield o-cresol. The reaction is conducted over alumina catalyst. 2,6-Xylenol is formed to an appreciable extent and the tri- and higher methylated phenols to a lesser extent follows:



Phenol o-Cresol



o-Cresol 2,6-Xylenol



2,6-Xylenol 2,3,6-Tri-methylphenol

The catalyst, "CATAPAL" alumina, is highly "ortho-selective," hence, the preponderance of ortho-substituted compounds such as o-cresol and 2,6-xylenol. Meta and para-substituted compounds are also formed to a much lesser extent as a result of isomerization of the ortho-substituted compounds.

GENERAL DESCRIPTION (CONTINUED)

C. Synthetic Unit (Continued)

1. Reaction Section (Continued)

The plant currently has two reactors operating in parallel. The reactors are cooled by molten salt circulation and both reactors share a common salt system (drawing CT-7-A).

The methanol-phenol feed to a reactor is vaporized in a series of feed-product and feed-salt exchangers. Product from the reactor is cooled and partially condensed in the feed-product exchangers and finally in a water-cooled condenser. The circulating molten salt serves as a cooling medium for the reactor as well as a heating medium for the feed. Provision of a coolant in the reactor is important in keeping reactor temperatures down to minimize coking that tends to deactivate the catalyst.

The product from the final condenser goes to a storage tank from which it is fed to the fractionation section.

After about 100-200 hours of operation, conversion in the reactors decreases and pressure drop across the reactor increases to a point such that the reactor has to be shut down and the catalyst regenerated. Because they share a common salt system, both reactors are shut down and regenerated simultaneously.

Periodically, the catalyst in a reactor has to be discarded and the reactor recharged with fresh catalyst.

2. Fractionation Section

The reactor product is purified by fractionation. The first column in the fractionation train is the "dehydrator." Water and light ends removed off the top of this column are condensed and phase separated with the water phase being sent to the sewer and the hydrocarbon phase recycled to the tower.

The other three columns in the fractionation section are used on a blocked-out basis for two operations, the "straight-run" operation and the "rerun" operation. Bottoms from the last fractionator on the "straight-run" operation serves as feed to the fractionation train on the "rerun" operation.

Overhead products from the three fractionators obtained during the "straight-run" and "rerun" operations and their disposition are indicated below:

GENERAL DESCRIPTION (CONTINUED)

C. Synthetic Unit (Continued)

2. Fractionation Section (Continued)

	STRAIGHT-RUN OPERATION		RERUN OPERATION	
	Overhead Product	Disposition	Overhead Product	Disposition
Fractionator No. 1	Phenol	Recycled to Reactor	2,6-Xylenol	Product
Fractionator No. 2	o-Cresol	Product	Mixed Xylenols	Batch Still for Further Purification or Blending
Fractionator No. 3	2,6-Xylenol	Product	80% 2,3,6-Tri-Methyl Phenol	Batch Still for Further Purification

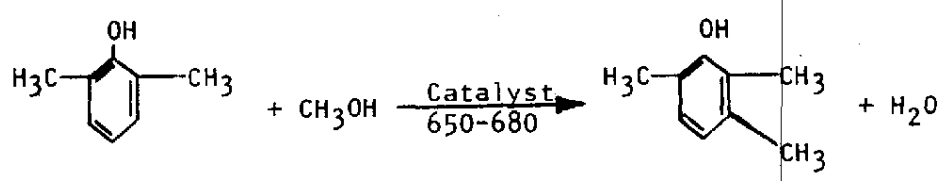
The bottoms product from the rerun operation is burned in the plant boilers. As noted above, further purification of some of the overhead products from the continuous fractionation is conducted in batch stills. Two batch stills are used for this purpose.

D. Isomerization Unit

In the isomerization reactor, predominantly ortho-substituted compounds were isomerized in the vapor phase to meta and para-type compounds over low silica porocel catalyst. Unlike the reactors in the synthetic plant, which have a cooling medium, the isomerization reactor was operated adiabatically. A flow diagram of the reaction system is shown in drawing CT-8-A.

E. High Pressure Methylation Unit (See Drawing CT-56-D)

Methanol and 2,6-xylenol feeds are combined and elevated to 500 psig. The material is preheated to 600°F in a reactor effluent-feed exchanger and elevated to 650°F against Dowtherm "A" in a second exchanger. Trim heat is applied to the feed as necessary in an electric heater prior to reactor entry. Feed enters the vertical shell and tube type reactor in a near critical state. Fluid is directed downward through alumina-packed tubes with cooling/heating provided by hot oil on the shell side. Crude product exits the reactor at 680°F. The stoichiometric reaction is:



2,6-Xylenol Methanol 2,3,6-Trimethylphenol

GENERAL DESCRIPTION (CONTINUED)

E. High Pressure Methylation Unit (Continued)

Reaction products are cooled to 290°F in the effluent/feed exchanger and to 150°F in a water-cooled unit. Pressure is reduced and crude reaction products are transferred to storage. At this stage, the material contains 2,3,6-TMP, 2,6-xylenol, methanol, dimethyl ether, water, and higher boiling phenol compounds.

Crude reaction products are similar in nature, but different in composition, than product from the reaction section of the Synthetic Unit. The two crude products are, therefore, stored separately and periodically processed through the continuous fractionation train of the Synthetic Unit on blocked-out operation.

The High Pressure Methylation Unit also has the capability of producing ortho cresol via the methylation of phenol. This operation requires more severe operating conditions than described above for 2,3,6-TMP.