

Interoffice Communication

To J. A. DeBernardi, VCM Plant, Lake Charles

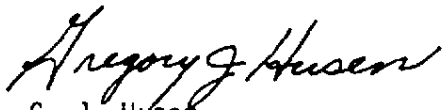
From G. J. Husen, Chemicals Research, Ponca City

Date May 5, 1977

Subject "Summary of Chemical Research Division Service Work for the Lake Charles VCM Plant," M. A. Newman

The attached report summarizes Chemical Research Division VCM Plant related research since 1970. Over one hundred seventy (170) reports and many other communications are summarized. This report should be helpful for acquainting new personnel with prior work. The reports and letters are available in Engineering Research Section files or the R&D Library.

Please contact us if we can assist in obtaining any additional information.



G. J. Husen
Research Group Supervisor
Engineering Research Section

lag/jw
CC + Enc
FK:WRS:DVP:WRBe:OCK:AJLu:RCL:MAN
CMS:RJC:DHS:GJF:RELe:RWM:RDG
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CWH 000008522

Technical Service Report

No. TSR - CEN - 77 - 1

To: J. A. DeBernardi

Title: Summary of Chemicals Research Divisions Service Work
for the Lake Charles' VCM Plant

Date: April 15, 1977

Objective: To present a summary of all the work Chemicals
Research has done for the Lake Charles' VCM plant
from 1970 through February, 1977.

Introduction

This report was compiled in order to organize the variety of work Chemicals Research and Development has done for the VCM plant since 1970. The major topics are arranged as follows:

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The general category includes such things as meetings where a wide variety of topics were covered, and subjects that were not easily categorized.

The types of communications included are: Interoffice Communications, Plant Progress Reports, Information Searches, Research Reports and VCM-related literature articles. None of R & D's Monthly Progress Reports are included in this summary, but they are available for further information. Most of the reports and communications are available in the Engineering Research Section files; however, reports not in the file are designated throughout the report as IN LIBRARY.

CWH 000008523

J. A. DeBernardi

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April 15, 1977

Also available from R & D is the report from the Stauffer Symposium held in 1976. The plant should have the notes from the first (1971) and second (1973) Stauffer Symposiums.

A handwritten signature in cursive script, appearing to read "M. A. Newman", with a horizontal line underneath.

M. A. Newman
Assistant Research Chemical Engineer
Engineering Research Section

lag/ck

Enc

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VCM SERVICE WORK SUMMARY

1970 - FEBRUARY, 1977

CWH 000008525

GENERAL TOPICS

STAUFFER VCM SYMPOSIUM; 839-0, 1971

This is a confidential report of the discussion of companies attending the Stauffer Symposium.

PROJECT REVIEW--VCM RELATED TOPICS, DECEMBER 8, 1971, 839-0, 1971

Subjects discussed were 1) plant studies on caustic washing of EDC to remove methyl chloride precursors, 2) laboratory investigation on the effect of conversion level and EDC contaminants on by-product formation in pyrolysis of EDC, 3) water washing of acid treated VCM, 4) reduction in tar production by separate chlorination of the EDC recycle from the VCM tower, and 5) the Stauffer experimental oxychlorination catalyst.

CONOCO VCM PRODUCT QUALITY MEETING, LAKE CHARLES, LOUISIANA; 839-0, 1971

Subjects discussed were 1) analytical methods for VCM, 2) the butadiene impurity problem, 3) light ends column plugging, 4) reactor modeling studies, 5) catalyst improvement, 6) diluent, 7) effluent treatment, and 8) process changes.

PROJECTS REVIEW MEETING, LAKE CHARLES VCM PLANT; 839-0, 1972

Projects discussed were 1) the EDC cracking studies, 2) butadiene removal from VCM, and 3) removal of copper from hydroblast water via centrifugation.

REVIEW OF VCM PLANT OPERATING AND QUALITY PROBLEMS, APRIL 7, 1972; 839-0 1972

Subjects discussed were 1) reduction of butadiene concentration in product VCM, 2) methyl chloride in the product VCM, 3) oxygenates in EDC tars, and 4) laboratory studies of EDC cracking.

WATER DISPLACEMENT OF VCM VAPOR AT ATMOSPHERIC PRESSURE--TEST OF WATER SAMPLING PROCEDURE; 839-0, 1975

Tests were conducted at PVC pilot plant on displacement of VCM vapor with heated water. Based on these tests, it did not appear that this method would be generally applicable for all vessels in VCM service.

DISTRIBUTION OF VCM PLANT CAUSTIC USAGE; 839-0, August-December, 1976

ACTION STEPS FROM JOINT MEETING OF VCM PLANT, CRD, PED, AND PRODUCT
MANAGEMENT--VCM PLANT, SEPTEMBER 29, 1976; 839-0, August-December, 1976

Too numerous to list here.

ACTION STEPS FROM JOINT MEETING OF VCM PLANT, CRD, AND PED--FEBRUARY 1,
1977; 839-0, 1977

Too numerous to list here.

VALUE OF PLANT LOSSES--1970; 839-0, 1970

NOTES ON MEETING WITH DON BUTTER AND OTHERS; 839-0, 1970

Many research projects were discussed.

REACTIONS OF CHLORINE AND ETHYLENE

Information Search 70-47. IN CONOCO LIBRARY

CWH 000008527

EDC

REACTIONS OF 1,2-DICHLOROETHANE; 839-0, 1971

An information search.

EDC DRYING; 839-0, 1968

Methods of drying EDC.

CWH 000008528

EDC CRACKING

PRELIMINARY DATA ON LABORATORY CRACKING EXPERIMENTS; 839-0, 1971

The report lists conclusions and data leading to those conclusions.
The conclusions are:

1. Impurities in EDC contribute to methyl chloride formation and cracking inhibition.
2. EDC cracking rate increases dramatically by increased surface/volume ratio in reactor.
3. A number of low-level by-products are formed in cracking reaction which generally inhibit the reaction.

A STUDY OF THE FORMATION OF BY-PRODUCTS DURING THE PYROLYSIS OF EDC-- A RESEARCH PROPOSAL; 839-0, 1971

PYROLYSIS OF 1,2-DICHLOROETHANE; 839-0, 1972

As conversion increased, it appears that by-product formation increases. A study of VCM and impurity production as a function of residence time was recommended.

EXPERIMENTAL STUDIES OF EDC PYROLYSIS; 839-0-1-72; Research Report

Experiments on pyrolysis of 1,2-dichloroethane to vinyl chloride are reported. The object of this work was to ascertain the side reactions which lead to the formation of by-products, particularly methyl chloride and butadiene. The level of all by-products formed, relative to vinyl chloride formed, increased exponentially with increases in 1,2-dichloroethane conversion level. Formation of methyl chloride is enhanced when 2-chloroethanol is present in the feed but does not go to zero when the 2-chloroethanol is removed. Formation of butadiene is enhanced when ethyl chloride is present in the feed. The effect of a number of possible contaminants on both by-product formation and 1,2-dichloroethane conversion level was examined. Increasing the surface area in the reactor was found to increase the pyrolysis rate and decrease by-product formation. IN CONOCO LIBRARY.

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EDC PURIFICATION

ADDITION OF PHOSPHITE OR PHOSPHORATE ESTERS TO EDC DISTILLATION COLUMN; 839-0, 1971

A copy of a patent was enclosed as a possible means of solving an EDC column reboiler fouling problem.

PLUGGING OF THE LIGHT ENDS COLUMN AT THE VCM PLANT; 839-0, 1972

There were two opinions on why the column was plugging: 1) ammonia working itself down the column causing a solids buildup and 2) an emulsion containing polymeric material upstream plugging the holes.

PURIFICATION OF 1,2-DICHLOROETHANE; 839-0, 1972

Amine treating might be a probable method for abbreviated EDC purification.

PROGRESS REPORT, NOVEMBER, 1971; 839-0, 1971

Work was continued on caustic treatment of EDC for removal of methyl chloride precursors and the VCM cloud problem.

PROGRESS REPORT; 839-0, 1971

Analysis of Competitor's EDC--Samples of EDC producers were compared with Conoco's furnace feed and sales grade EDC.

PROGRESS REPORT, DECEMBER, 1971; 839-0, 1972

1. Methyl Chloride Problem

Caustic strength in caustic wash vessel T-102 would have to be kept high for additional mixing between P-102 and T-102 to be effective in removing 2-chloroethanol.

2. Emulsion Problem in T-101

No conclusions reached on why emulsions are forming in the caustic wash vessel.

PROGRESS REPORT, OCTOBER, 1971; 839-0, 1971

1. Methyl Chloride Problem

Caustic washing of 2-chloroethanol was carried out in stages. The washing was to simulate a proposed reactor scheme

REMOVAL OF 2-CHLOROETHANOL (METHYL CHLORIDE PRECURSOR) FROM EDC VIA BAUXITE ADSORPTION; 839-0, 1971

The life of the bauxite was too short to be economically attractive to remove all of the 2-chloroethanol.

REDUCTION OF METHYL CHLORIDE IN LAKE CHARLES PRODUCT VCM VIA REMOVAL OF PRECURSOR 2-CHLOROETHANOL; 839-0, 1971

A linear relationship between concentration of 2-chloroethanol and methyl chloride was found. The intercept value of 44 ppm was not even close to the 10 ppm objective.

REMOVING 2-CHLOROETHANOL FROM DICHLOROETHANE; 834-0, 1971

This is a literature search.

PROGRESS REPORT, JULY, 1971; 839-0, 1971

1. Methyl Chloride Problem

Checks were made of different plant streams for 2-chloroethanol, a major methyl chloride precursor. Some experiments were also conducted for 2-chloroethanol removal.

2. Methyl Chloride-2-Chloroethanol Correlations; 839-0, 1971

A correlation of amount of methyl chloride from amount of 2-chloroethanol was given.

ENVIRONMENTAL ASPECTS

AIR POLLUTION AFTERBURNER PROJECT FOR VCM PLANT, 839-0, 1972

VCM plant considering installing an afterburner on C-500 vent stack to meet environmental requirements on ethylene.

CATALYTIC OXIDATION--VCM PLANT, 839-0, 1975

A list of questions that need to be answered by piloting a catalytic oxidation unit and review of progress on both catalytic oxidation and incineration.

REMOVAL OF VCM FROM VCM PLANT VENT STREAMS DESIGN AND RESEARCH PLANS AND OBJECTIVES, 839-0, 1975

DESIGN BASIS FOR DISPOSAL OF VCM PLANT VENT GAS AND LIQUIDS--ADDITIONAL DATA, 839-0, 1975

DESIGN BASIS FOR DISPOSAL OF VCM PLANT VENT GAS AND LIQUIDS, 839-0, 1975

PROJECT 839-0--REMOVAL OF VCM FROM VCM PLANT VENT STREAMS VIA CATALYTIC OXIDATION, 839-0, 1975

MINUTES--NOVEMBER 5, 1975, MEETING ON VCM EMISSION CONTROL PROJECT, 839-0, 1975

The purpose of the meeting was to review the overall status of the project and to examine the technical feasibility of developing and designing a catalytic oxidation unit within the anticipated EPA time limits.

PROGRESS REPORT--VCM EMISSION CONTROL PROJECT, 839-0, 1975

The two areas discussed were 1) Incineration and Quenching and 2) Scrubbing, Neutralization, and Offsites.

ORIGIN OF ACETALDEHYDE AND ETHYLENE OXIDE IN RECENT VCM PLANT STREAMS AND RESEARCH ON THEIR REMOVAL BY SOLID REAGENTS, 839-0, JAN.-MAY, 1976

VCM EMISSION CONTROL SERVICE--DEFINITION OF VENT STREAMS, 839-0, JAN.-MAY, 1976

R&D PROJECT--CATALYTIC OXIDATION OF VCM CONTAINING STREAMS, 839-0, JAN.-MAY, 1976

Catalytic Oxidation will not be feasible in time to meet EPA requirements.

PROJECT STATUS--CATALYTIC OXIDATION OF VCM CONTAINING STREAMS, 839-0, JAN.-MAY, 1976

Catalytic Oxidation will not be feasible in time to meet EPA requirements.

ENGINEERING AND COST STUDY OF AIR POLLUTION CONTROL FOR THE PETROCHEMICAL INDUSTRY--VOLUME 3--ETHYLENE DICHLORIDE MANUFACTURE BY OXYCHLORINATION, 839-0, JAN.-MAY, 1976

PROJECT 839-0--REMOVAL OF VCM FROM VCM PLANT VENT STREAMS VIA CATALYTIC OXIDATION--MEETING OF DEC. 17, 1975; 839-0, JAN.-MAY, 1976

Proposal and discussion of further work.

FREE CHLORINE EMISSION FROM VCM DESTRUCTION SYSTEM, 839-0, JAN.-MAY, 1976

General information concerning free chlorine formation and emission--EPA's requirements and impact on the system.

OXY BY-PASS TEST RUN, 839-0, AUG.-DEC., 1976

Analyses for different components in different streams. Other studies' data are also included.

VCM PLANT WASTE TREATMENT SYSTEM IMPROVEMENTS: ACTION PLAN, 839-0, AUG.-DEC., 1976

Factors determining action plan were:

1. EDC and 2-chloroethanol levels in biotreatment pond.
2. Soluble BOD removal across the treatment system, and
3. Suspended solids removal in final clarifier.

DETECTING AND MONITORING VINYL CHLORIDE IN AIR

Information Search 76-23. IN CONOCO LIBRARY

PROGRESS REPORT, OCTOBER, 1971; 839-0, 1971

1. Plant Vent Problems

Work on identifying the source of an objectionable odor from C-500 vent gas stack was done.

VCM ADSORPTION ON ACTIVATED CARBON PROCESS--ADSORPTION AND STEAM REGENERATION; 820-27-1-75, Research Report

Carbon adsorption was found to be a feasible method of reducing the VCM concentration in air or nitrogen streams to below 10 ppmv. The effect of temperature, VCM concentration, feed humidity, and residual water in the bed on loading was determined. Carbon activity was unchanged over 30 cycles, but a carbon attrition rate of 0.326 percent/cycle occurred. The adsorption was found to take place in a narrow zone in the bed. The effect of residual water, flow rate, and feed VCM concentration on the outlet VCM concentration preceding this zone was determined. A recommended regeneration time of 30 minutes and a steam rate of .0133 lb steam/lb carbon minute were determined. IN CONOCO LIBRARY

THE BIOTREATABILITY OF VCM PLANT WASTE WATER; 839-1-1-73, Research Report

With the exception of ethylene dichloride, which can be removed from VCM plant waste water by air stripping, the waste can be efficiently reduced by about 50 mg/l BOD₅ (a 95 percent reduction) and 350-450 mg/l COD (an 80 percent reduction) residuals by activated sludge biotreatment. The nature of the residual COD was not determined. The present oxidation basins are oversized by fourfold for most efficient operation, although this overexposure does allow EDC stripping to occur. The plant can be operated under the excessive residence conditions if a way can be found to settle the clarifier suspended solids with additives. To achieve adequate biotreatment, the raw waste must be supplemented with phosphate. COD and BOD loading must be carefully controlled within a rather narrow range for efficient COD and BOD removal. IN CONOCO LIBRARY

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HCl PURIFICATION

RESEARCH REPORT NO. 842-1-1-70--REMOVAL OF ORGANIC CONTAMINANTS FROM HYDROGEN CHLORIDE PRODUCED IN THE VINYL CHLORIDE MONOMER PLANT, 842-1, 1970

This report covers the building and operation of a pilot plant to evaluate removal of organic components from HCl.

HCl PURIFICATION, 842-1, 1970

HCl Purification of organics was dropped because the plant could buy its supply of HCl. If needed, the plant would blend the unpurified HCl directly.

TRIP REPORT--REVIEW OF PILOT PLANT PURIFICATION OF HCl FOR THE ANCON METHYLCHLORIDE PLANT, 842-1, 1969

HCl FOR ANCON, 842-1, 1969

A check of the ability of oxychlorination catalyst to convert residual acetylene in HCl to heavier chlorinated compounds. This was in consideration as a backup approach.

HCl CLEANUP PROJECT, 842-1, 1969

Liquid HCl must be free of chlorine before being returned to oxychlorination catalyst.

PLANS FOR OPERATION OF HCl PURIFICATION PLANT, 842-1, 1969

Temperature, space velocity, and chlorine addition were to be studied. If these were optimal, a catalyst life study would begin.

CHANGES IN DESIGN--HCl PURIFICATION PILOT PLANT FOR LAKE CHARLES VCM PLANT, 842-1, 1969

ECONOMICS OF HCl PURIFICATION--VCM PLANT, 842-1, 1969

PROJECT SCHEDULE--HCl FOR ANCON W.O. M-2288 LAKE CHARLES PETROCHEMICAL PLANT, 842-1, 1969

Tentative project schedule for mechanical design, procurement, and construction of the HCl purification system.

PILOT PLANT RESEARCH--HCl FOR ANCON, 842-1, 1969

Priorities of information to procure in pilot plant.

CHLORINATION PROCESS PILOT PLANT DESIGN--HCl FOR ANCON, 842-1, 1969

Actual design specs are given.

HCl FOR ANCON, MEETING APRIL 25, 1969, 842-1, 1969

Several routes for removing acetylene from HCl were discussed.

HCl CLEANUP VIA CHLORINATION/CONDENSATION, 842-1, 1969

Chlorination followed by condensation seems to be best method for removing contaminants. A pilot study is necessary to answer more thoroughly some questions.

TRIP REPORT--WORK BY AUTHOR AT VCM PLANT ON HCl CLEAN-UP MARCH 16, 1969 TO APRIL 4, 1969, 842-1, 1969

PRELIMINARY DESIGN AND ECONOMICS FOR HCl PURIFICATION-CHLORINATION PLUS DISTILLATION, 842-1, 1969

HCl CLEAN-UP PROJECT, 842-1, 1969

A critique of the HCl Clean-up design.

MEETING--OCTOBER 28, 1969, ANALYTICAL PROBLEMS OF THE HCl CLEAN-UP PILOT UNIT, 842-1, 1969

HCl CLEAN-UP PROCESS, 842-1, 1969

A presentation of plant scale design.

COMPLETE ANALYSIS OF HYDROGEN CHLORIDE GAS, 842-1, 1969

Provided by Hooker Chemical.

A NEW METHOD FOR ON-STREAM ANALYSIS OF HYDROCARBON CONTENT IN GAS-PHASE HCl, RESEARCH REPORT NO. 585-4-3-69, DECEMBER, 1969; 842-1, 1969

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OXYCHLORINATION

STEAM DILUENT ADDITION TO OXYCHLORINATION REACTORS; 839-0, 1971

Injection of steam into primary oxychlorination reactors reduces formation of ethyl chloride by-product.

OXYCHLORINATION REACTION IMPURITY FORMATION VERSUS PROCESS INPUT AIR DEW POINT VARIATIONS: A PLANT DATA TEST OF THE STARKS-HACKETT PROCESS; 839-0, 1971

The idea that ethyl chloride and other impurities might be a function of moisture is borne out, especially as catalyst ages.

FIXED-BED OXYCHLORINATION YIELDS 1,2-DICHLOROETHANE; 839-0, 1972

REMOVAL OF COPPER FROM HYDROBLAST WATER USED TO CLEAN THE VCM PLANT OXYCHLORINATION REACTORS; 839-0, 1972

A pilot centrifugation study was done to reduce the copper content in the reactor wash water at turnaround, when catalyst washed out.

DISCUSSION OF GOODRICH OXYCHLORINATION SYSTEM AT VCM PLANT ON MARCH 3, 1973; 839-0, 1974

Subjects discussed were:

1. Goodrich Royalties and Liabilities
2. Process Capital Costs
3. Catalyst
4. Process
5. Materials of Construction
6. Past Process Improvements and Problems

HIGH PRESSURE DROP IN OXYCHLORINATION REACTOR R-303; 839-0, Jan.-May, 1976

This report gives an explanation why the unusually high pressure drop. Research Report 839-0-2-71, "A Study of Ethylenic Carbon Formation versus Catalyst Activity in a $\text{CuCl}_2\text{-KCl-Al}_2\text{O}_3$ Oxychlorination Catalyst" is enclosed.

PROGRAM TO DETERMINE IF HCl FROM OLIN'S TDI PROCESS IS ACCEPTABLE AS FEED TO CONOCO'S OXYCHLORINATION PROCESS; 839-0, Aug.-Dec., 1976.

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HCl SPLITTING IN THE OXYCHLORINATION UNIT; 839-0, Aug.-Dec., 1976

Action steps to determine safety (due to flammability) were decided.

EXPLOSION LIMITS OF THE SYSTEMS $C_2H_4/O_2/N_2$ UP TO 26 ATM AND 20°C; 839-0, Aug.-Dec., 1976

FLAMMABILITY IN OXYCHLORINATION WITH HCl SPLITTING; 839-0, Aug.-Dec., 1976

HCl splitting does not cause a flammability problem.

FLAMMABILITY AND EXPLOSIBILITY OF ETHYLENE AND 1,2-DICHLOROETHANE; 839-0, Aug.-Dec., 1976

Literature search.

CONTINENTAL FLAMMABILITY STUDY--SOUTHWEST RESEARCH INSTITUTE MEETING, AUGUST 19, 1976; 839-0, Aug.-Dec., 1976

A meeting was held to examine facilities and talk with Southwest about studying flammability region of ethylene 1,2-Dichloroethane.

ETHYLENE/EDC/HCl FLAMMABILITY STUDY; 839-0, Aug.-Dec., 1976

A review of what would be expected from the flammability study.

EXPERIMENTAL STUDIES OF IRON-ALUMINA CATALYST BEHAVIORS IN R305, VCM PLANT, WESTLAKE, LOUISIANA; 839-0, Aug.-Dec., 1976

REMOVAL OF CHLORINE AND ETHYLENE FROM OXYCHLORINATION VENT GAS; 839-0, Aug.-Dec., 1976

OLIN HCl USAGE-OXYCHLORINATION UNIT; 839-0, Aug.-Dec., 1976

This IOC discusses problems of using Olin HCl in oxychlorination unit.

REDUCTION OF ETHYLENE AND HCl LOSSES FROM THE OXYCHLORINATION SYSTEM; 839-0, 1970

KINETICS OF THE OXYCHLORINATION OF ETHYLENE; 839-0, 1970

CWH 000008538

OXY VENT RECYCLE TO R-101 FOR C₂H₄ RECOVERY--VCM; 839-0, 1970

Economic benefits of recycling unreacted ethylene from oxychlorination to direct chlorination.

REDUCTION OF ETHYLENE AND HCl LOSSES FROM THE OXYCHLORINATION SYSTEM; 839-0, 1970

Ideas were suggested for investigations in order to make best recommendations on how to reduce ethylene and HCl losses significantly.

METHODS FOR INCREASED CONVERSION IN R-304 CLEAN-UP REACTOR; 839-0, 1970

THE PARAMETERS AFFECTING OPERATION OF THE STAUFFER OXYCHLORINATION REACTION SYSTEM; 839-0, Report dated September 22, 1969

R-302 TUBE FAILURES; 839-0, 1968

The tubes appear to have failed due to burning of carbon deposited in the catalyst.

REPORT OF WESTLAKE OXYCHLORINATION REACTOR MEETING, AUGUST 25, 1969; 842-1, 1969

PRELIMINARY EVALUATION OF STAUFFER'S SPHERICAL PARTICLE OXYCHLORINATION CATALYST; 839-0, 1971

The preliminary evaluation revealed 1) the alumina support for the spherical catalyst did not evolve from Conoco materials and 2) relative to the present oxychlorination catalyst the spherical particles possess:

- a. approximately equal surface area and catalytic activity.
- b. lower cumulative pore volume
- c. greater crush strength

A STUDY OF CATALYST AND REACTOR BEHAVIORS IN THE VAPOR-PHASE OXYCHLORINATION OF ETHYLENE; 839-0-2-69, Research Report

This report pertains to the theory and practice of the vapor-phase oxychlorination of ethylene. It is of specific relevance to the large-scale operation of the Stauffer-designed Westlake, La., VCM plant oxy unit, especially to the catalyst degradation phenomena therein. IN CONOCO LIBRARY

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A FURTHER STUDY OF CATALYST AND REACTOR BEHAVIORS IN THE VAPOR-PHASE OXYCHLORINATION OF ETHYLENE; 839-0-2-70, Research Report

This report describes our latest views and data on the vapor-phase oxychlorination of ethylene, especially to large-scale VCM plant operations. It concerns catalyst degradation, reactor changes and instrumentation. Several new methods of catalyst evaluation, certification and analysis are also given herein. IN CONOCO LIBRARY

AN OPERATIONAL STUDY OF R-303 OXYCHLORINATION REACTOR DECOKING; 839-0-7-70, Research Report

A restricted variables study was made of the in-plant, on-stream feasibility of the catalyst decoking of the R-303 $\text{Cu}_2\text{Cl}_2\text{-KCl-Al}_2\text{O}_3$ catalyst. While no decoking was observed, there did appear to be an improved reactor performance after this decoking test. The process conditions necessary for catalyst decoking are given in the report. A brief correlation of reactor behavior with impurity formations through the oxychlorination test interval is included. IN CONOCO LIBRARY

SINGLE-PELLET MICROREACTOR STUDIES OF GAS DILUENCY EFFECTS IN THE VAPOR-PHASE OXYCHLORINATION OF ETHYLENE; 839-0-1-71, Research Report

A reactor using a single oxychlorination catalyst pellet was used to study the effect on the pellet temperature of various gas-phase diluents to the feed gases of the reactor. It was found that gas dilution with compatible and feasible gases was an effective way of reducing individual pellet temperatures caused by the oxychlorination reaction. By using a means of reducing the individual pellet temperature during reaction, the catalyst degradation can be reduced and the life of the catalyst extended. IN CONOCO LIBRARY

A STUDY OF ETHYLENIC CARBON FORMATION VERSUS CATALYST ACTIVITY IN A $\text{CuCl}_2\text{-KCl-Al}_2\text{O}_3$ OXYCHLORINATION CATALYST; 839-0-2-71, Research Report

This experimental report explores the kinetics of carbon formation from ethylene pyrolysis within a Harshaw $\text{CuCl}_2\text{-KCl-Al}_2\text{O}_3$ oxychlorination catalyst. Since the oxychlorination activity is related to carbon formation, catalyst activity measurements were made upon these variously carbonized catalyst pellets, this via a $\text{C}_2\text{H}_4\text{-HCl-O}_2$ oxychlorination catalyst activity measurement. The time-temperature-catalyst activity as related to carbon formation are presented. These specific data pertain to oxychlorination catalyst design, reactor design, and process design. IN CONOCO LIBRARY.

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MICROREACTOR (SCANA) ANALYSES OF OXYCHLORINATION CATALYST PERFORMANCE FROM
THE NOVEMBER 1970 VCM PLANT SHUTDOWN; 839-0-4-71, Research Report

A study was made of the oxychlorination catalyst activity of samples of the Harshaw oxychlorination catalysts removed in November, 1970, from three ethylene oxychlorination reactors at our VCM plant at Westlake, Louisiana. The study was made using the single-pellet microreactor (SCANA) developed for catalyst activity determinations. It was found that the catalyst activity varied widely because of the extremes in operating environment and because of catalyst dusting and the resulting caking within a reactor tube. IN CONOCO LIBRARY

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NEW TECHNOLOGY

NEW ROUTE TO VINYL CHLORIDE--VINYL CHLORIDE PROCESS HAS WIDE RANGE OF BY-PRODUCTS; 839-0, 1971

An adiabatic reactor produces monomer from chlorine gas, ethylene, and recycle dichloroethylene. Process conditions control the ratios of chlorinated by-products.

ADVANCED MITSUI TOATSU VINYLCHLORIDE PROCESS; 839-0, 1972

A description of the Mitsui Toatsu Vinylchloride Process.

HYDROGENATION OF HYDROGEN CHLORIDE FROM AN EDC CRACKING UNIT

Information Search 76-25. IN CONOCO LIBRARY

ENGINEERS TELL HOW TO CUT COSTS; 839-0, 1972

A process for turning HCl wastes into chlorine was revealed.

CHLORINE MADE IN THE MODERN STYLE WITHOUT CAUSTIC; 839-0, 1972

A discussion of the Kel-Chlor Process for converting waste HCl into chlorine.

HYDROGEN CHLORIDE TO CHLORINE VIA THE KEL-CHLOR PROCESS; 839-0, 1973

LUMMUS "TRANSCAT" VCM PROCESS--VCM FRACTIONATION STUDY; 839-0, 1973

An evaluation of the fractionation section of the Lummus "Transcat" VCM process design was made as part of the overall "Transcat" process technical feasibility study.

TRANSCAT REVIEW WITH LUMMUS; 839-0, 1973

Subjects discussed were 1) the features of "Transcat," and 2) various production cases Conoco would need to analyze usefulness of the process.

TRANSCAT PROCESS; 839-0, 1973

A list of questions about the process to ask Lummus.

CWH 000008542

CALL REPORT--LUMMUS/CONOCO MEETING, DECEMBER 19, 1973--"TRANSCAT" VCM TECHNOLOGY; 839-0, 1974

Doubts about "Transcat" were discussed, especially the scarcity of ethane feedstocks.

SPECULATIVE ECONOMICS OF CHLORINATION AND OXYCHLORINATION OF ETHANE AND ETHYLENE--PART I: EDC PRODUCTION; 819-4-1-71, Research Report

Speculative economic calculations were made for seven alternate routes for producing ethylene dichloride (EDC) from ethane or ethylene by chlorination and/or oxychlorination. The seven routes and required netback prices for 18 percent DCF return are summarized as follows:

<u>PROCESS</u>	<u>RNP, ¢/Lb</u>
Chlorination of high purity ethylene	3.2
Chlorination of dilute ethylene	4.0
Dehydrohalogenation of ethyl chloride	3.6
Oxychlorination of ethyl chloride	3.8
Chlorination of ethane with HCl electrolysis	5.4
Oxychlorination of ethane to ethylene	4.0
Oxychlorination of ethane (hypothetical case)	3.3

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SPECULATIVE ECONOMICS OF CHLORINATION AND OXYCHLORINATION OF ETHANE AND ETHYLENE, PART II: VCM PRODUCTION; 819-4-2-77, Research Report

Speculative economic calculations were made for ten alternate processes for producing VCM from ethylene, ethane, and naphtha. Results indicated that two processes have a significant economic advantage over the conventional high purity ethylene process. These are:

1. Lummus process for oxychlorination of ethane to VCM.
2. "One-Step" process for oxychlorination of ethane to VCM.

Two other processes have a marginal economic advantage over the conventional process, but the advantage was in the same range as the accuracy of the calculations.

It was recommended that study be continued on the Lummus process and the "one-step" process. IN CONOCO LIBRARY

CWH 000008543

VCM

EXPLOSIVE AND FLAMMABILITY CHARACTERISTICS OF VCM

Information Search 75-10. IN CONOCO LIBRARY

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VCM INCINERATOR

✓ INCINERATION RECYCLE DUE VCM BY-PRODUCTS; 839-0, 1972

Incineration of chlorinated hydrocarbons replacing dumping at Akzo's plant in 1974.

✓ DISPOSAL OF CHLORINATED BY-PRODUCTS; 839-0, 1972

An article from Chem. Eng. on using incinerators for organic wastes.

VCM INCINERATOR DESIGN; 839-0, 1975

A listing of the information needed to produce a "Class A" design for VCM plant.

DESIGN BASICS--VCM VENT GAS INCINERATOR; 839-0, Jan.-May, 1976

✓ TRIPS TO JOHN ZINK COMPANY AND TRANE THERMAL COMPANY TO DISCUSS CHLORINATED HYDROCARBON INCINERATION DESIGN; 839-0, Jan.-May, 1976

✓ PLANT TRIP TO SHELL CHEMICAL COMPANY'S VCM INCINERATION SYSTEM AT NORCO, LOUISIANA; 839-0, Jan.-May, 1976

VCM INCINERATION-ENVIRONMENTAL IMPACT STUDY REFERENCES; 839-0, Jan.-May, 1976

RESEARCH PROPOSAL-CORROSION STUDY FOR EVALUATION OF INCONEL 600 AND CARBON STEEL USE IN INCINERATION WASTE HEAT BOILER; 839-0, Aug.-Dec., 1976

✓ CORROSION TESTING--COMPARATIVE CORROSION RATES ON CARBON STEEL AND INCONEL 600 IN Cl_2 AND HCl ATMOSPHERES; 839-0, 1977

This study was done in order to determine the expected corrosion rates on the waste heat boiler tubes in the VCM incinerator.

OPERATION OF CORROSION STUDY FOR EVALUATION OF INCONEL 600 AND CARBON STEEL USE IN INCINERATOR WASTE HEAT BOILER; 839-0, 1977

INCINERATION OF VCM CONTAINING GAS STREAMS

Information Search 76-5. IN CONOCO LIBRARY

CWH 000008545

VCM PLANT LIQUID BY-PRODUCTS

✓ VCM PLANT BY-PRODUCTS; 839-0, 1975

The different and most feasible methods of handling the by-products.

✓✓ VCM PLANT BY-PRODUCTS; 839-0, Jan.-May, 1976

To find new uses for by-product chemicals seems fruitless. Incineration with HCl and energy recovery was suggested.

DEHYDROCHLORINATION OF CHLORINATED HYDROCARBONS

Information Search 76-25.

CHLOROLYSIS OF ALIPHATIC AND AROMATIC COMPOUNDS TO CCl₄; 839-0, 1972

This paper is a method on converting VCM tars to CCl₄.

✓ FINDING PROFITS IN CHLORINATED HYDROCARBON WASTES; 839-0, 1972

Discussion of different firms' methods of handling chlorinated hydrocarbons.

STATUS REPORT--OXYGENATES IN EDC TARS; 839-0, 1972

The plant had to reduce the oxygenate levels in their tars to sell them. Three aspects of this problem were investigated: 1) removal of oxygenates from the tars, 2) identification of the oxygenate source streams, and 3) removal or prevention of oxygenate in the source streams.

DISPOSAL OF VCM TARS; 839-0, 1973

This IOC explains a scheme for converting VCM tars into ethane and muriatic acid by direct hydrogenation over molten zinc chloride catalyst.

✓ CHLORINATED TAR DISPOSAL; 839-0, 1973

A summary of three approaches to the VCM tar problem:

1. Decomposition of tars to HCl and carbon.
2. Burning tars to produce HCl feed for a new oxychlorination reactor.
3. Burn tars, feed effluent directly to existing oxy reactors.

DECOMPOSITION OF CHLORINATED TARS IN A NICKEL REACTOR; 839-0, 1973

PYROLYTIC DECOMPOSITION OF VCM PLANT CHLORINATED TARS; 5505-1-1-73,
Research Report

Chlorinated tars from the VCM plant were decomposed in a nickel reactor in the absence of air at 1,100°F and 260 psig to yield carbon, hydrogen chloride, methane, ethane, vinyl chloride and traces of incomplete decomposition products. No water is formed, allowing the HCl to be recovered for VCM production and the methane and ethane to be consumed as fuel. The carbon can be handled as a solid waste or burned. IN CONOCO LIBRARY

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VCM PURIFICATION

SELECTIVE HYDROGENATION OF VINYL CHLORIDE; 839-0, 1971, Information Search

This report sought to find literature to describe a process that selectively and catalytically removes hydrogenated olefinic compounds present as impurities in VCM. No specific process was found, but the report contains some general information.

LAKE CHARLES VCM PLANT BY-PRODUCT PROBLEMS

The topics discussed were 1) butadiene removal via oleum treatment of the VCM, 2) methyl chloride removal from VCM, and 3) laboratory pyrolysis of EDC.

RESEARCH REPORT NO. 839-0-6-71 "REMOVAL OF METHYL CHLORIDE FROM VCM" BY DR. RONNIE D. GORDON; 839-0, 1971

Two relatively expensive methods were found for reducing methyl chloride content in VCM from approximately 50-100 ppm to less than 15 ppm.

RESULTS OF TEST TO REMOVE RESIDUAL ACID FROM OLEUM-TREATED VCM; 839-0, 1971

Data were given on several test runs and it was concluded that probably some sort of water wash would be the best means of removing residual acid.

OLEUM TREATING SYSTEM FOR REMOVAL OF BUTADIENE FROM VINYL CHLORIDE MONOMER; 839-0, 1971

This is a rough draft of a Conoco paper to be presented at the annual Stauffer Symposium relating to butadiene removal technology.

SUMMARY OF WORK ON THE PROBLEM OF ACID CARRY-OVER ON THE BD TREATMENT PROCESS; 839-0, 1971

After oleum treatment of VCM, the residual acid was neutralized in the caustic wash, but was causing numerous problems. Pilot or plant water-washing system was suggested.

PROPOSAL FOR BRIEF PILOT STUDY OF RESIDUAL ACID IN OLEUM TREATED VCM; 839-0, 1971

A study would show whether the treatment system should be a coalescer or of the adsorption type.

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REACTION CONTROL FOR POSSIBLE BY-PRODUCTS FORMATION REDUCTION IN R-304;
839-0, 1971

If methyl chloride problem solely is R-304, maybe the hot spot value could be lowered.

VCM IMPURITIES PROGRAM; 839-0, 1971

Work was being done on analytical problem of analyzing butadiene in VCM samples that had been in contact with HCl in metal sample cylinders.

MARCH 11 MEETING WITH STAUFFER CHEMICAL COMPANY, OLEUM TREATING SYSTEM;
839-0, 1971

Planned submittal of oleum treating system paper was enclosed.

REMOVAL OF 1,3 BUTADIENE FROM VCM VIA ION EXCHANGE RESINS; 839-0, 1972

Sulfonic Acid ion exchange resin preferentially removes small quantities of 1,3 butadiene, but also undesirable levels of carbonyls are produced.

A REPORT ON WORK DONE TO REMOVE RESIDUAL ACID AND OTHER IMPURITIES FROM
OLEUM TREATED VCM; 839-0, 1972

Conclusions from this study are

1. Water washing the VCM prior to caustic washing will remove the residual acid and colored bodies.
2. In some cases the addition of the water wash lowered the aldehyde level; in other cases it did not.
3. Ion exchange would be more expensive than water washing for removal of the residual acid.
4. A plant test run should be conducted to investigate water washing using your present oleum treatment facilities and small laboratory equipment.

REMOVAL OF BUTADIENE FROM VINYL CHLORIDE MONOMER; 839-0, 1970

Research Report No. 859-0-5-70

REMOVAL OF 1,3 BUTADIENE FROM VINYL CHLORIDE MONOMER USING OLEUM AND
CONCENTRATED SULFURIC ACID; 839-0, 1970

Research Report No. 839-0-6-70

CWH 000008549

REMOVAL OF CONTAMINANTS FROM VCM IN A SIDEDRAW TOWER--VCM PLANT--
LAKE CHARLES, LOUISIANA; 839-0, 1970

TRIP REPORT AND RESULTS OF TEST RUNS FOR THE REMOVAL OF BUTADIENE FROM
VCM WITH OLEUM CONDUCTED AT THE VCM PLANT; 839-0, 1970

TRIP REPORT ON VCM PLANT TEST RUN TO REMOVE BUTADIENE; 839-0, 1970

The oleum treatment may be permanently used in the plant but will need some minor adjustments.

OPERATION OF BUTADIENE REMOVAL SYSTEM; 839-0, 1970

EXAMINATION OF VCM SAMPLES GRJ 2856-42-F THROUGH -42-S; 839-0, 1970

BD REMOVAL SYSTEM OLEUM TREATING PROCESS; 839-0, 1970

Flow diagram with some dimensions given.

REMOVAL OF BUTADIENE FROM VCM; 839-0, 1970

A general synopsis of the problem and the solution.

BUTADIENE IN VCM; 839-0, 1970

A program to reduce butadiene in VCM was outlined.

MATERIALS OF CONSTRUCTION FOR VCM OLEUM SEPARATOR; 839-0, 1970

A discussion of the pros and cons of different construction materials.

PRELIMINARY DATA FOR BUTADIENE REMOVAL FROM VCM USING PLEUM; 839-0, 1970

The pilot plant is explained and data given.

PILOT TESTING OF SULFURIC ACID TREATING OF VCM; 839-0, 1970

This is a preliminary survey of the requirements of a pilot plant.

BUTADIENE REMOVAL FROM VCM; 839-0, 1970

A discussion of two methods to remove butadiene from VCM: 1) contacting with concentrated sulfuric acid, and 2) use of oxy catalyst.

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VISIT TO VCM PLANT; 839-0, 1970

The bench scale sulfuric acid removal scheme was explained and the possibility of removing other impurities by the same process.

TRIP REPORT--MEETING ON VCM QUALITY AT LAKE CHARLES, JUNE 10, 1970; 839-0, 1970

Problems discussed were: 1) Cloudy Monomer, 2) Butadiene Problem, and 3) VCM specifications.

RESEARCH REPORT NO. 839-0-1-70 "CLOUD FORMATION IN VCM" BY DRS. C. M. STARKS AND R. D. GORDON; 839-0, 1970

PROGRESS REPORT, NOVEMBER, 1971

Work was continued on the VCM cloud problem and on the caustic treatment of EDC for removal of methyl chloride.

PROGRESS REPORT; 839-0, 1971

Methyl Chloride Problem-2-chloroethanol may be precursor of methyl chloride.

PROGRESS REPORT, OCTOBER, 1971; 839-0, 1971

1. Cloudy Monomer Problem

The oxygen content of some plant streams was measured since oxygen could cause clouding.

ECONOMICS OF MeCl REMOVAL FROM VCM USING MFA; 839-0, 1971

COST FOR PILOTING THE MeCl REMOVAL FROM VCM; 839-0, 1971

REMOVAL OF METHYL CHLORIDE FROM VCM; 839-0, 1971

The economics of a pilot size operation were given.

METHYL CHLORIDE REMOVAL FROM VCM; 839-0, 1970

A catalyst developed for methyl chloride removal deactivated much too soon to be economical.

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MEETING WITH VCM PLANT PERSONNEL CONCERNING METHYL CHLORIDE REMOVAL
FROM VCM; 839-0, 1971

REMOVAL OF METHYL CHLORIDE FROM VCM; 839-0, 1970

Research work and a proposed pilot study were discussed.

METHYL CHLORIDE-2-CHLOROETHANOL CORRELATION; 839-0, 1971

A correlation of amount of methyl chloride from amount of 2-chloroethanol was given.

REMOVAL OF METHYL CHLORIDE FROM VCM; 839-0-6-71, Research Report

Methyl chloride (MeCl) has been removed from vinyl chloride monomer (VCM) using two different methods. Treatment of VCM vapor with metal oxides on alumina at ca 150°C or treatment of liquid VCM with 10 w/o monoethanol amine (MEA) at 60-70°C successfully removes MeCl from VCM. IN CONOCO LIBRARY

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Rough Yeast Cals 12/3/81

Comp Dry Yeast Wet Yeast Total

EDC	900	2935	3835
VCN	100	494	594
ETC	210	4662	4872
CHL ₃	—	3700	3700
CH ₄	—	1921	1921
CH ₄	12,000	—	12,000
C ₂ H ₆	900	673	1573
H ₂	1500	—	1500
O ₂	3000	—	3000
N ₂	7520	—	7520
CO	15	—	15
CO ₂	1500	—	1500
H ₂ O	—	—	—
(C ₂ H ₆)	(300)	—	(300)
(H ₂ O)	(316)	—	(316)

CUM 00008553

James O. Gibson



To

Date

HEAT OF COMBUSTION SAMPLES

EDC T/L P/O - START - LCVCP-M-76-2
" " " - END - LCVCP-M-76-3
TANK T-405 - LCVCP-M-76-4

C.E. DRAW 5-27-76 - LCVCP-M-76-5
L.E. DRAW 5-27-76 - LCVCP-M-76-6
C.E. DRAW 5-28-76 - LCVCP-M-76-7

E.W. SAYBOLT & CO. INC.

1404 S. HOUSTON

HOUSTON, TX 77502

5-27-76 1530

5-27-76 1930

5-28-76 0100

	T.C. mult type conversion factor	area X F	corrected wt %		Area X F	Corrected wt %		Area X F	Corrected wt %
C ₂ ⁻	.54	492 492	.025		936	.037		403	.041
VCM	.894	24,892	1.270		363,40	1.429		11,946	1.223
ETCE	.7323	497,099	24.854		682,10	26.755		26,7448	27.378
?	1.0 ?	3,392	0.173		8,543	.335		—	—
H ₂ C=CCl ₂	1.074	55,480	2.831		74,662	2.928		28,993	2.968
?	1.0 ?	1619	.083		2,619	.103		991	.101
ED + acetald.	1.0 ?	13,272	.677		32,180	1.267		8166	.836
Trans	.9949	81,572	4.162		114,418	4.487		42,597	4.361
CCl ₄	1.2148	232,940	11.886		287,335	11.269		115,347	11.808
1,1	.8375	55,229	2.818		74,094	2.906		26,738	2.737
CH ₂ Cl ₂	1.0 ?	8,275	.422		12,117	.415		4,083	.418
Cis (EOC part)	1.0 ?	73,262	3.738		94,109	3.691		36,394	3.726
CHCl ₃	1.095	701,602	35.800		887,416	34.803		336,226	34.411
EDC	1.000	209,653	10.698		241,875	9.486		97,539	9.98
2 CCl ₃ OH	.9838	1,026	.052		882	.035		present ?	—
		1,959,805	99.489		3,221,836	100.001		976871	100.01
					2,541,836				

Transmitted to John Campbell 6-11-76 via phone

CWH 000008555