Region-6

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Industrial Hygiene Walk-Through Survey Report of E.I. duPont deNemours and Company Pontchartrain Works LaPlace, Louisiana

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DISCLAIMER

Mention of company or product name in this report does not constitute endorsement by the National Institute for Occupational Safety and Health (NIOSH).

ACKNOWLEDGEMENTS

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The analysis of the bulk samples was conducted by Ardith A. Grote, Chemist, Division of Physical Sciences and Engineering, National Institute for Occupational Safety and Health.

PURPOSE OF SURVEY:	To perform a walk-through industrial hygiene survey of a 1,3-butadiene polymer producing plant and determine the suitability for inclusion in an in-depth exposure survey regarding this substance.
EMPLOYER REPRESENTATIVES CONTACTED:	Darrel Wild, Senior Chemist, Environmental Control Division
EMPLOYEE REPRESENTATIVES CONTACTED:	Employees are not unionized.
STANDARD INDUSTRIAL CLASSIFICATION (SIC) CODE:	2822 - (Synthetic Rubber [Vulcanizable Elastomers)

ABSTRACT

A walk-through industrial hygiene survey was conducted at the Pontchartrain Works of E.I. duPont de Nemours and Company in LaPlace, Louisiana on August 26, 1985. The purpose of the survey was to obtain information on the neoprene polymer production process and assess the potential for occupational exposure to 1,3-butadiene. This information will be used in determining the suitability of including this plant in an in-depth industrial hygiene survey.

The plant, which opened in 1964, began producing neoprene from 1,3-butadiene in 1968. Both chloroprene and neoprene are currently produced from 1,3-butadiene at the plant. In addition to use in the synthesis of neoprene, chloroprene is also shipped by rail cars to another duPont facility. The incoming 1,3-butadiene is received by pipeline. The company maintains personnel records on current and past employees.

No monitoring data was provided by the company. However, the company reported that they have conducted personal monitoring for 1,3-butadiene in those dichlorobutene and chloroprene areas where 1,3-butadiene exposure was thought likely. Of the 74 personal samples taken by duPont, 72 were less than 10 ppm.

The results of the NIOSH analysis of the bulk sample of neoprene for residual 1,3-butadiene were non-detectable. The limit of detection was 0.04 ng/mg by weight. A bulk sample of chloroprene was not obtained.

The facility is considered to be a potential candidate for an in-depth industrial hygiene survey for the determination of the extent of exposure to 1,3-butadiene. This consideration is based on an evaluation of the reported historical industrial hygiene data.

INTRODUCTION

Inhalation exposure of rats and mice to 1,3-butadiene induced a carcinogenic response at multiple sites. Mammary fibroadenomas/carcinomas, uterine sarcomas, Leydig cell adenomas of the testes, thyroid follicular cell adenomas, exocrine tumors of the pancreas, and Zymbal gland carcinomas were identified in rats exposed at concentrations of 1000 to 8000 ppm of 1,3-butadiene. Mice exposed to 625 or 1250 ppm of 1,3-butadiene developed a high incidence of malignant lymphomas; and increased incidence of other tumors, including hemangiosarcoma; and testicular and ovarian atrophy.[1,2]

The offspring of pregnant rats exposed to 1,3-butadiene at 8000 ppm had major skeletal defects. In addition, fetal toxicity was observed when pregnant dams were exposed at 200 ppm, 1000 ppm, and 8000 ppm.[3]

Epidemiological studies of workers employed in facilities producing styrene-butadiene rubber have indicated an increased, but not statistically significant, risk of mortality from neoplasms of the lymphatic and hematopoietic tissues and from leukemia.[4,5]

Based on these data, the National Institute for Occupational Safety and Health (NIOSH) recommends that 1,3-butadiene be regarded as a potential occupational carcinogen and teratogen and as a possible reproductive hazard.[6] Due to the number of workers potentially exposed to 1,3-butadiene and the resulting potential health risk, NIOSH researchers are conducting an extent-of-exposure study of workers potentially exposed to the monomer during production of 1,3-butadiene based products.

EXPOSURE EVALUATION CRITERIA

The current Permissible Exposure Limit (PEL) enforced by the Occupational Safety and Health Administration (OSHA) for 1,3-butadiene is 1000 ppm for an 8-hour time-weighted average (TWA).[7] The American Conference of Governmental Industrial Hygienists (ACGIH) has included 1,3-butadiene in their Notice of Intended Changes for the 1985-1986 Threshold Limit Values (TLVs) based upon reported animal carcinogenicity data.[8] The intended change identified 1,3-butadiene as an "A2" industrial substance suspected of carcinogenic potential in man. A numerical TLV of 10 ppm was proposed in connection with the notice.

NIOSH in their Current Intelligence Bulletin recommends that 1,3-butadiene be regarded as a potential occupational carcinogen and teratogen and as a possible reproductive hazard.[6]

HISTORY AND DESCRIPTION OF THE PLANT

The duPont Pontchartrain Works was constructed in 1964 and occupies 1700 acres in LaPlace, Louisiana. The plant initially produced adiponitrile. Neoprene production was added in 1968. An isomerization reactor was added to handle this change. Adiponitrile production ceased in 1978.

The present facility uses 1,3-butadiene to produce 250 million pounds per year of chloroprene, of which 100 million pounds is shipped off site. The remainder is used to produce 95 million pounds per year of neoprene (polychloroprene). The neoprene is sold to a large number of customers, and finds application in the manufacture of gloves, hoses, and automobile products. Hydrochloric acid (HCl) is also produced at the facility as a by-product of the dichlorobutene synthesis.

PROCESS DESCRIPTION

Figures 1, 2, and 3 are flow diagrams of the neoprene process employed at the Pontchartrain Works. Figure 1 is a schematic of the entire process while Figure 2 and 3 are detailed schematics of several process steps. This process also produces HCl and chloroprene as marketable products. The 1,3-butadiene is transported to the plant by pipeline from Shell Chemical Company's 1,3-butadiene monomer plant in Marco, Louisiana. 1,3-butadiene is stored in spheres in the tank farm. The duPont Pontchartrain Works consumes 160 million pounds per year of 1,3-butadiene. The 1,3-butadiene (gas) is first dried and fed, along with chlorine gas, into the dichlorobutene (DCB) reactor. Unreacted 1,3-butadiene and HCl are stripped off the crude dichlorobutene. The resulting crude dichlorobutene is either sent to storage or directly to dichlorobutene refining. During dichlorobutene refining, 3,4-dichlorobutene isomer is separated from the other isomers and fed to chloroprene synthesis. Hydrochloric acid is recovered from the by-product and sold or used on site.

The liquid chloroprene produced at the facility is inhibited to prevent polymerization before being shipped by rail to a duPont plant in Louisville, Kentucky or used to make neoprene at the site. The rail cars are dedicated to chloroprene shipment and are equipped with magnetic gauges. The remaining annual production of 95 million pounds of chloroprene is captively used on site for polymerization to neoprene (polychloroprene). Prior to polymerization the chloroprene is refined and modifiers, water, and sodium hydroxide are added to the chloroprene. After polymerization, unreacted chloroprene is recovered and returned to the chloroprene refining step. The polymerized neoprene is then washed, cut, dried at 300°F and packaged. The finished solid neoprene is bagged and warehoused on site.

Quality Control

Quality control (QC) samples for 1,3-butadiene are collected in gas bags at the tank farm and analyzed via gas chromatography. Liquid 1,3-butadiene samples are taken using a dry ice cold trap. The cold trap contains acetone which maintains the temperature at approximately -80°F. The exit tubing of the bath has a slit (bunsen valve) to control the pressure. No QC samples for 1,3-butadiene are taken past the DCB refining stage. The DCB QC samples typically contain less than 0.1 percent 1,3-butadiene.

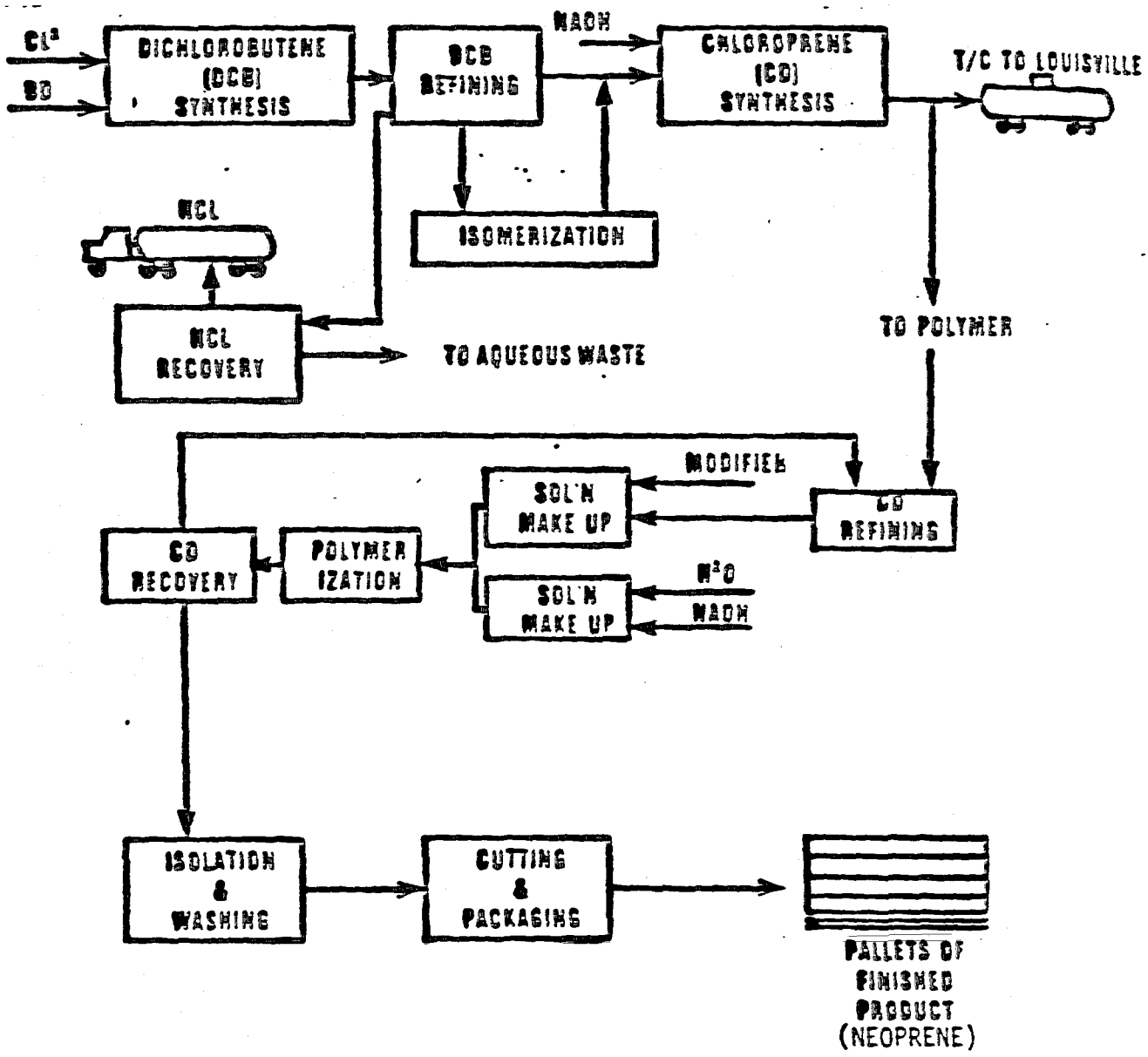

Figure 1. Process flow diagram for the production of chloroprene and neoprene at the duPont Pontchartrain Works, Laplace, Louisiana.

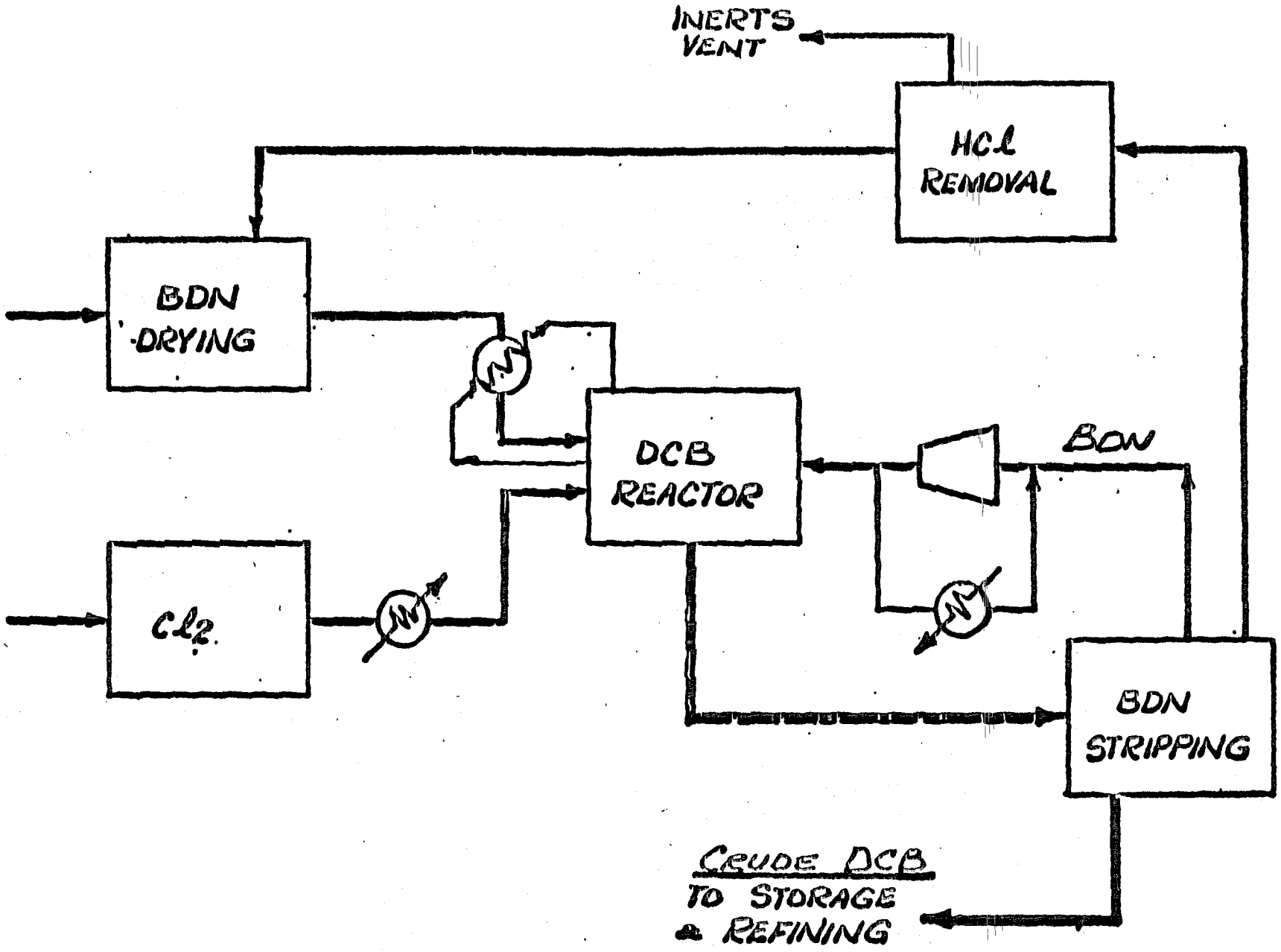

Figure 2. Process Flow Diagram for the Synthesis of DCB.

DCB SYNTHESIS

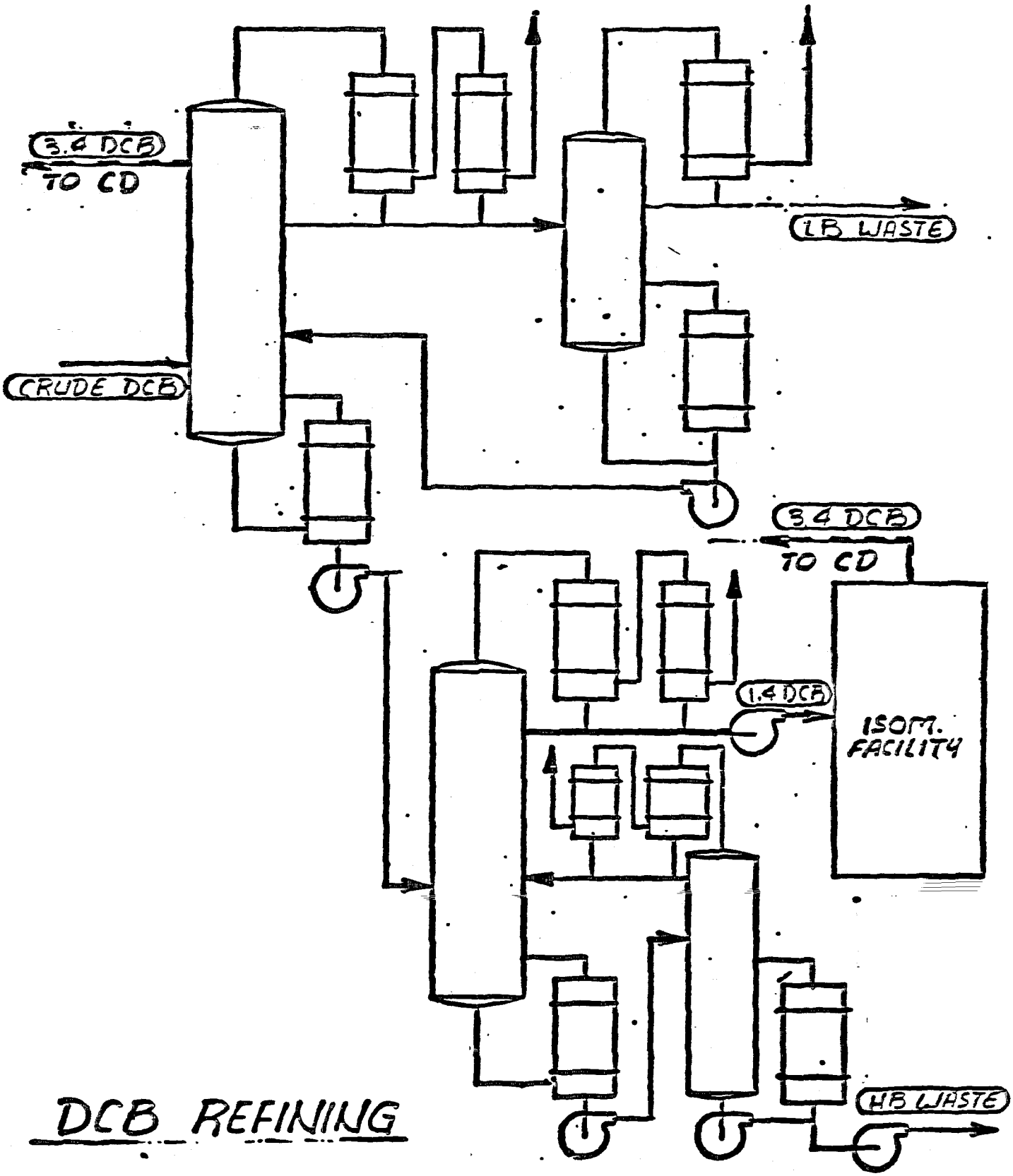

Figure 3. Process Flow Diagram for DCB Refining

DESCRIPTION OF THE WORKFORCE

Presently, there are 610 employees at the LaPlace site; 220 are in administrative jobs, 225 in process jobs, and 165 in maintenance. The average age of the workforce is 39 years. The process employees work during four rotating 12-hour shifts. Maintenance personnel work during the 8-hour day shift. There are five persons per shift involved in DCB synthesis and refining operations. One of these employees work in the 1,3-butadiene end of these process steps. The production employees with potential for exposure to 1,3-butadiene are divided into the following job categories:

Board Operator:	Control room operator. One per shift.
Tank Farm Operator:	Takes QC samples and monitors conditions in the tank farm. One per shift.
DCB Synthesis Operator:	Works in synthesis area of DCB, monitors process, takes QC samples, decontaminates equipment prior to maintenance. One per shift.
DCB Refining Operator:	Monitors process readings, obtains QC samples, and decontaminates equipment prior to maintenance. One per shift.
Isomerization Operator	Works in the isomerization area, takes QC samples, and decontaminates equipment prior to maintenance. One per shift.
Chloroprene Synthesis Operator:	Works in synthesis area, takes QC samples, decontaminates equipment prior to maintenance. One per shift.
Chloroprene Tank Car Loading Operator	Loads chloroprene tank cars. One per shift.

DESCRIPTION OF PAST WORKER EXPOSURES

1,3-butadiene has been the primary feed to the plant since its opening in 1964, and the dichlorobutene synthesis process has remained essentially the same since startup. Personal monitoring for 1,3-butadiene is reportedly performed primarily with the DuPont Protek® badge. In brief, the analytical method involved sample desorption with carbon disulfide and analysis by gas chromatography with flame ionization detector. The chromatograph utilizes a 60 meter capillary column and splitless injection. As a result of the splitless injection technique, butadiene is not resolved from other C_4 and C_3 hydrocarbon impurities. In 1985, 74 personal samples were obtained in the DCB and chloroprene areas of the plant where

1,3-butadiene exposure was thought likely. No area monitoring for 1,3-butadiene has been conducted at the Pontchartrain Works. Of the 74 personal samples in Table 1, 72 were reported by DuPont to be below 10 ppm. Two synthesis operators had 1,3-butadiene exposures of 19 and 10.1 ppm. However, the company was able to identify the source of the exposure and correct the problem.

Table 1
DuPont 1,3-butadiene Air Sampling Results

Job Assignment	No. of Samples	Avg. Conc. of Butadiene	Max. Conc. of Butadiene
DCB Synthesis Operator	52	1.7 ppm	19.1 ppm
Mechanics	11	0.6	2.3
Others	11	0.1	0.4

In an effort to reduce exposure during maintenance activities the company decontaminates and blinds prior to maintenance on process equipment. The system is purged three times with nitrogen and vented to a flare. The process is closed during this operation, therefore, special personal protective equipment is not required.

Engineering Controls

All pumps handling 1,3-butadiene and most of those handling the reaction products have single mechanical seals. Seals need to be replaced approximately every 2 to 6 months. Since 1,3-butadiene is handled at ambient temperature and at pressures slightly above atmospheric, no compressors are required which reduces the number of mechanical pieces of equipment requiring maintenance. Relief valves on reactor vessels are vented to the atmosphere, not to a flare. Since 1,3-butadiene is under pressures slightly above atmospheric, samples are taken by the gas bag or with old trap techniques rather than by a sample "bomb". Rail cars used for shipment of the chloroprene product are equipped with magnetic gauges.

DESCRIPTION OF MEDICAL, SAFETY, AND INDUSTRIAL HYGIENE PROGRAMS

Medical Program

The company conducts pre-employment and semi-annual physicals for all employees. Tests given during the pre-employment and annual physicals include hearing, vision, lung function, blood, and urine tests. A chest x-ray is taken on all employees at every physical.

The plant has one full-time physician as well as one licensed nurse. The physician and nurse are duPont employees. There are 15 people per shift organized and trained as a first-aid response team. The closest hospital, the River Parish Medical Center, is located 3 miles from the plant.

Safety Program

The company maintains an organized safety program. The program is headed by a full-time Safety Supervisor. Additional staff include two employees who, for 50% of the time, engage in activities related to the safety program. A Safety and Health Committee meets monthly and has eight subcommittees with different responsibilities. The committee consists of fourth-level (and higher) supervisors and has between 8 to 10 people. The Plant Manager is the Committee chairman.

As of September, 1985, Nomex® fire resistant clothing is required for all production employees. Production workers' clothes are washed daily. Safety glasses (including prescription), and hard hats are required and are provided by the company. Goggles are provided and required during QC sampling. Safety shoes are required for process workers and are company supplied. Other employees receive shoes at half price up to two pairs per year. PVC gloves are available and are required for specific jobs such as QC sampling. In the dichlorobutene section, employees are provided with full-face MSA chin, full-face air-supplied, and escape respirators. These respirators are provided because of potential exposure to dichlorobutene rather than to 1,3-butadiene. The company has a respiratory protection program.

Cigarette smoking is only permitted in designated smoking areas. The company also provides showers, clothing change areas, and separate lunchrooms.

Industrial Hygiene Program

The company has a formal industrial hygiene program headed by one environmental health employee and one full-time lab technician. Any sampling results that exceed one-half of the standard are investigated. The DuPont internal performance standard for 1,3-butadiene is 10 ppm. Personal measurements are taken for 1,3-butadiene, chloroprene, dichlorobutene, hydrochloric acid, and toluene. Samples are also obtained for asbestos in the power plant area. Ventilation measurements are taken on the lab hoods and a noise standard of 87 dB is reportedly to be not exceeded in order to protect the 12-hour-shift workers. An OVA Century Analyzer with a detection limit of 10 ppm for hydrocarbons is used to spot leaks. Once leaks are identified, the process leak is repaired and hydrocarbon (which may include 1,3-butadiene) levels are reduced.

DESCRIPTION OF PERSONNEL RECORD SYSTEM

The company maintains personnel records on terminated and current employees. A total of 1264 workers have worked at the plant site. The

records date from the plant opening and no records have been purged. The personnel records for the last 6 years are maintained on a computer and provide the following information:

1. Name
2. Social Security Number
3. Employee number
4. Job description code

Records prior to 1980 are available through medical where the work location was recorded at the time of the physical.

Since 1980, employees' job activities can be tracked on a daily basis by cross checking dates with time sheets. The time sheet specifies the unit the employee is working. The company provides insurance coverage with death benefits for the employees.

SAMPLING AND ANALYTICAL METHOD FOR BULK POLYMER SAMPLES

Because polymers are further processed into finished products, it was one of the intentions of this study to analyze the polymer(s) produced at the survey site to determine the potential for release of 1,3-butadiene monomer at temperatures typical of various fabrication processes employed in the manufacture of finished products which used the polymer. Therefore, a bulk polymer sample(s) was obtained at each site and then analyzed for emanation of free monomer at three predetermined temperatures: 1) ambient, 2) highest polymer process temperature, and 3) highest estimated end use temperature.

The method for analysis of the bulks was developed by the Measurement Research Support Branch of the Division of Physical Sciences and Engineering at NIOSH.

Sampling System Description

A Tekmar Model 4000 Automatic Dynamic Headspace Concentrator combined with the Model 4100 Heated Sampler Module and Model 1000 Capillary Interface was used throughout this study for the bulk sample analysis. The basic operating principle of this system is as follows: residual organic compounds diffusing from the bulk matrix (placed in an enclosed sampling tube) are removed by purging the enclosed sampling tube with inert gas (helium) followed by subsequent analysis in a gas chromatograph. The Heated Sampler Module allows the bulk matrix to be heated at a specified controlled temperature variable from ambient to 200°C. The organics removed from the sampling tube are next swept to a porous polymer adsorbent (Tenax) and trapped. The adsorbent is then heated and backflushed to release the organics, which are then swept onto the head of a capillary column via the Capillary Interface Unit. This capillary interface operates on the principle of cryofocusing. The interface freezes (using liquid nitrogen) the desorbed sample from the Concentrator into a narrow band on the injection end of a fused silica precolumn. The focused sample is then flash heated and injected into a gas chromatograph.

Sampling System Conditions

The system described involves the setting of numerous temperature and time parameters that had to be predetermined before any analytical work could be accomplished. After preliminary work with the 1,3-butadiene standard the settings listed in Table 2 were chosen and used throughout the study.

TABLE 2
SAMPLING TIME AND TEMPERATURE VARIABLES
USING THE DYNAMIC HEADSPACE CONCENTRATOR, TEKMAR MODEL 4000

Sample Chamber Temperature	Variable 30°C (ambient) to 200°C
Sample Transfer Lines & Valves	150°C
Sample Chamber Preheat Time	0 min (ambient); 5 min (heated samples)
Sample Wet Purge Time	5 min
Purge Flow	40 cc/min
Trap Desorb Temperature	200°C
Trap Desorb Time	4 min
Trap Bake Out Temperature	220°C
Trap Bake Out Time	15 min

Due to the high sensitivity of this system, sample size, especially with heated samples, had to be kept small to avoid overloading or contaminating the Tenax trap irreversibly with generated organic compounds. Twenty to 30-mg portions of the bulk polymers were weighed and used for sampling. (Even with these small amounts the system was often found to be heavily contaminated with higher boiling organic material after analysis of a sample.)

Analytical Instrumentation and Conditions

All bulks were initially screened using the Dynamic Headspace Concentrator interfaced directly to an HP 5840 gas chromatograph (GC) equipped with a flame ionization detector (FID). A 30-meter DB-1 fused silica capillary column, 0.25 mm I.D., and 1.0 um film thickness was used for all analyses. The column was temperature programmed from 35°C to 260°C at a rate of 15°/min after an initial hold time of 2 minutes. 1,3-butadiene eluted at about 2.0-2.2 minutes under these analytical conditions.

Positive identification of the presence of butadiene in selected samples was accomplished by interfacing the headspace unit and GC column directly into an HP 5982A mass spectrometer (MS). Samples were reanalyzed under the same concentrator conditions except that the GC effluent was passed into the mass spectrometer ion source rather than a FID. Samples were scanned from 35 to 200 atomic mass units (amus) to obtain the mass spectra. The presence of 1,3-butadiene was specifically look for by monitoring for the m/e 54 ion. A 1,3-butadiene gas standard was run by mass spectrometry to obtain a standard reference spectrum and GC/MS retention time data for comparison. Standards in the same range as used for the GC/FID calibration were also analyzed by GC/MS.

Calibration and Standards

Quantitation of 1,3-butadiene released from the polymer bulks was performed by GC-FID. GC/MS was used for confirmation and identification only. Certified (± 2%) 37-liter Scotty IV cylinders of 1,3-butadiene in nitrogen were used for standards (obtained from Scott Specialty Gases). A one-liter Tedlar bag was filled from this cylinder for use in obtaining the standard aliquots. This bag was evacuated and refilled with new 1,3-butadiene standard every 2-5 days. The 1,3-butadiene standard appeared stable in the Tedlar bag for at least 5 days. Various 0.1-5.0 cc aliquots of 1,3-butadiene from the bag were taken using gas tight syringes and injected directly into the purge stream of the heated module sample tube. Standards were subjected to the same purge and trap conditions as the samples. An initial calibration curve was constructed using multiple runs of varying amounts of a 9.51 ppm calibrated 1,3-butadiene gas standard. Each day at least two standard runs were made and amounts calculated against this curve to make sure the system was performing satisfactorily.

At the lower range of an analytical method, it may not be possible to confidently attribute an instrument response to the substance in question. The point at which instrument response can confidently be attributed to the contaminant being measured is called the "limit of detection" (LOD). If an instrument response is attributed to the contaminant, it may be present at such low levels that the confidence interval for the results reported may be excessive. The point at which the range of possible values are within acceptable limits is called the "limit of quantitation" (LOQ). These limits were calculated from the statistics of the calibration curve.

Under the analytical conditions previously described the limit of detection (LOD) for butadiene was approximately 1 ng per injection. Based on an initial sample weight of 25 mg (actual weights used varied from about 20 to 30 mg for solids), the LOD per sample was about 0.04 ng/mg or 0.04 ppm by weight. The limit of quantitation (LOQ) was 0.2 ppm by weight.

Sample Analysis

The following general procedure was used for the bulk samples: One 20-30 mg portion of the bulk was weighed out and put into the sample tube. An initial ambient run was made on all samples at 30°C. If little or no butadiene was detected at this temperature, the same portion of the bulk was subjected to the next higher predetermined temperature and reanalyzed. The procedure was repeated for a third temperature if applicable.

If 1,3-butadiene was detected in the sample at a certain temperature, that same portion of the bulk was then reanalyzed at the same temperature again, two or three times if necessary, until little or no additional butadiene was evolved. The sample then progressed to the next higher temperature and the process repeated if necessary.

Only samples suspected of containing 1,3-butadiene at a level above the LOQ (0.2 ppm) were reanalyzed at a later date using GC/MS to positively confirm the presence of 1,3-butadiene.

Analytical Results

The analytical results of the bulk analysis of neoprene were below the limit of detection of 0.04 ng/mg by weight at all analytical temperatures. The temperatures at which the bulk sample was analyzed were: ambient, 86°F (30°C) and process, 320°F (160°C). A bulk sample of chloroprene was not obtained from the company because of the lack of stability of the compound and Hazardous Material Regulations for transporting the material to the NIOSH analytical laboratory.

DISCUSSION

The duPont Pontchartrain Works manufactures chloroprene and neoprene from 1,3-butadiene with hydrochloric acid produced as a by-product. The process occurs in a closed system, tightly maintained for both economic and fire hazard reasons.

In 1985 the company conducted some industrial hygiene sampling for 1,3-butadiene in the dichlorobutene and chloroprene process areas. The majority of the exposure data (96%) is below 10 ppm.

Since the system operates at pressures only slightly above atmospheric, QC sampling is done using gas bags in the tank farm. Single mechanical seals are employed on all pumps in 1,3-butadiene service.

CONCLUSIONS

Based on the historical industrial hygiene information provided by DuPont, the Pontchartrain Works plant qualifies for further consideration as a candidate for site selection for an in-depth industrial hygiene survey. The purpose of the survey, if DuPont is selected, would be to develop an extent of exposure profile for all job descriptions associated with the production and distribution of 1,3-butadiene based polymers or products.

RECOMMENDATIONS

The company should consider retrofitting dual mechanical seals on all process pumps with single mechanical seals.

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