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FINAL

## CONTROL TECHNOLOGY ASSESSMENT

FOR

## COAL GASIFICATION AND LIQUEFACTION PROCESSES

Cresap Test Facility Cresap, West Virginia

Report on Site Visit of April 16-19, 1979

Contract No. 210-78-0084

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Submitted to:

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## FOREWORD

During the period April 16-19, 1979, Enviro Control, Inc., conducted a site visit to the Cresap Test Facility at Cresap, West Virginia. The purpose of the visit was to study the technology used to control worker exposure to hazardous chemical and physical agents at the facility.

The Cresap Test Facility is operated by the Liquefied Coal Development Corporation (LCDC), a wholly-owned subsidiary of Fluor Engineers and Constructors, Inc. The following LCDC personnel were present for this initial meeting and/or subsequent meetings:

Michael Barrick, Assistant Safety/Health Supervisor Robert Blake, Safety/Health Supervisor A.G. Draeger, Plant Manager Robert K. Geery, Process Control William Kittlewell, Maintenance Administrator Donald Kaslof, Mechanical Engineer James Swindler, Operations Superintendant Maurice Turgeon, Engineering and Instrumentation Supervisor

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## I. INTRODUCTION

## A. Background

The objective of the "Control Technology Assessment for Coal Gasification and Liquefaction Processes" program is to study the control technologies available for preventing occupational exposure to hazardous agents in coal conversion plants. This report details the control technology and industrial hygiene information gathered at the Cresap Test Facility (CTF) located at Cresap, West Virginia during the site visit of April 16-19, 1979.

Operation of the DOE-owned CTF was being carried out by Fluor Engineers and Constructors, Inc., to test the various critical mechanical components required for coal liquefaction and to demonstrate the Consol Synthetic Fuel (CSF) liquefaction process for the production of low-sulfur liquid fuel. The CTF was included in this study because the equipment and process problems (corrosion, erosion) being encountered in other more commercially viable liquefaction processes (such as Solvent Refined Coal and Exxon Donor Solvent) were also being experienced at the CTF.

## B. Project History

In 1957, the Consolidation Coal Company (Consol) began a laboratory investigation of the Consol Synthetic Fuel (CSF) process to produce a low-sulfur, high-boiling aromatic fuel oil. Consol continued funding the investigation through continuous bench-scale operations until August 30, 1963. At this time a contract was signed with the Office of Coal Research (OCR) for further development of the process through the bench-scale and pilot plant. The OCR-sponsored project was given the code name "Project Gasoline" although in effect experimental development was aimed only at production of low-sulfur synthetic crude oil distillate.

In May 1962, OCR executed a contract with Ralph M. Parsons Company to conduct an engineering and economic evaluation of the process. The Parsons Company was also retained as design consultants to OCR in connection with the proposed contract with Consolidation Coal Co. to build and operate a pilot plant to test the process.

In 1963, a second contract was executed with Consol to further conduct laboratory development of the process and prepare design specifications for the construction of a pilot plant. The design of the 20 ton per day coal processing pilot plant was completed in 1965 by C.W. Nofsinger Co. The construction contract was awarded to Dravo Corp. and the plant, located in Cresap, West Virginia, was completed in 1967.

The first run at the pilot plant was begun on May 23, 1967, one week before the plant was dedicated. Between July 1, 1968 and February 10, 1970, the plant achieved 1818 hours of dissolved coal (extract) production, 842 hours of high-pressure hydrogenation, and 550 hours of integrated plant operation on Pittsburgh No. 8 coal.

Plant operation was terminated for budgetary reasons in February 1970, 2-1/2 years ahead of schedule, before satisfactory integrated operation was achieved. However, operation of the pilot plant showed that the process could be scaled-up from bench-scale units with no unexpected process-related problems (though an extensive effort in equipment development was required).

Reactivation of the Cresap facility as an integrated process test center to produce low-sulfur liquid fuel from coal was begun in May 1974 when a contract was awarded to Fluor Engineers and Constructors, Inc., to organize, coordinate, and direct the testing program. Since then, the facility has been renovated to meet environmental requirements and current design standards of piping, electrical, and mechanical equipment codes. The Liquefied Coal Development Corporation (LCDC), a wholly-owned subsidiary of Fluor, was formed during 1975 to operate the reactivated pilot plant.

Construction activities continued through 1976 and were completed by September 1977. Upon completion of the plant construction, the Fluor field office was closed. Care, custody, and control of the plant was returned to LCDC in August and start-up activities began by the middle of the month. The Ralph M. Parsons Company was retained as a consultant to D.O.E. prior to the LCDC start-up. By the end of 1977, four extraction runs had been carried out with the longest coal feed run being 144 hours. Conversion averaged 62 percent with more than 5500 gallons of extract-rich intermediate being produced.

Fires and equipment plugging prevented extended operations in 1978 and early 1979. Integrated operation of the CTF was carried out in December 1978 and the operational capability of the hydrogenation and related systems demonstrated significant improvement over previous operations in January and February 1979. Efforts to activate other tests were delayed by a fire in the extraction section. After repairs were completed, three periods of integrated operation were conducted in March but were terminated due to blockages in the carbonizer and hydrogenation lines.

Decommissioning of the plant began in May, 1979, immediately after our site visit.

### C. Process Description

The Consol Synthetic Fuel (CSF) process used at the Cresap Test Facility to produce a low-sulfur liquid fuel involves a three step process: (1) coal preparation (Area 100) and extraction (Area 200); (2) undissolved coal and minerals separation from dissolved coalsolvent mixture; and (3) hydrogenation and fractionation of the dissolved coal-solvent mixture. A schematic of this process is given in Figure 1.

At the Cresap Test Facility, coal is first crushed to a minus 100mesh size and mixed with aromatic solvent to form a coal-solvent slurry. The slurry, pressurized and preheated, passes through an extractor where coal dissolution occurs. The slurry is then treated in a solvent deashing unit where the coal extract/solvent mixture is separated from the undissolved coal and minerals. The undissolved solids are removed as a thickened slurry and sent to a carbonizer for conversion into char, oil, and gas.

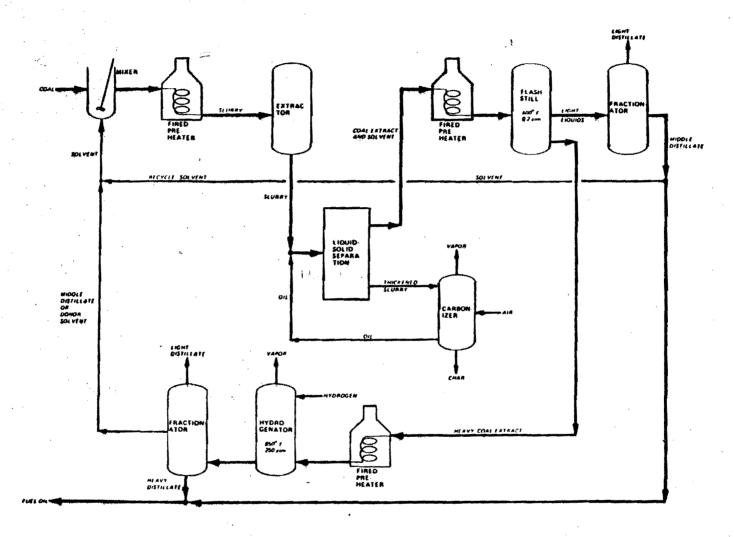


Figure 1. Consol Synthetic Fuel Process Schematic Cresap Test Facility Cresap, West Virginia From the separator the coal extract/solvent mixture enters the flash still where it is separated into light and heavy components. The light component is fractionated into a light distillate and middle distillate product. The middle distillate is used either as recycle solvent for slurry mixing or as product fuel. The light distillate is stored as naphtha for product blending.

The heavy component is hydrogenated and fractionated into three distillate fractions. The light distillate is stored as naphtha for product blending while the middle distillate is used as recycle aromatic solvent for slurry mixing. The heavy distillate is used as product fuel.

#### D. Plant Layout

The Cresap Test Facility uses the Consol Synthetic Fuel (CSF) process to produce a low-sulfur, high-boiling, aromatic residual fuel oil. The unit operations are housed in two open, multi-level structures with support facilities in separate buildings (the third level of the 300 area is an enclosed structure containing the filtration system).

These unit operations are segregated into two groups, which are separated by a service road about 20 feet wide.

Numerical area designations are used at the Cresap Test Facility in reference to the various unit operations. Numerals from 100 to 700 represent process unit operations and 900 the support and utility operations. These numerals and the unit operations they represent are given in Table 1. This designation system is also employed in Figure 2, a layout of the plant, to identify the unit operations and relative locations.

The ground level of all units was constructed of concrete with all flooring being diked to contain solvent spills. A majority of the pumps were located on the ground level, which simplifies monitoring and maintenance of the pumps.

## TABLE 1 AREA DESIGNATIONS

## Cresap Test Facility Cresap, West Virginia

AREA	DESCRIPTION
100	Coal preparation
200	Solvent extraction
300	Solids separation
500	Solvent recovery
600	Hydrogenation
700	Fractionation
800	Carbonization
900	Support/utilities
1100	Tank farm
1200	Environmental units

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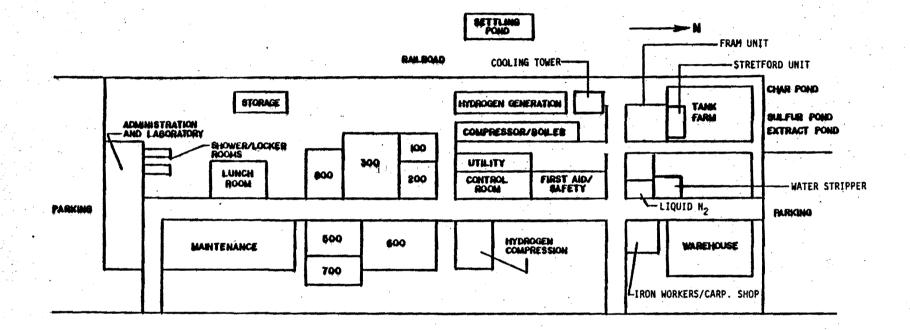


Figure 2. Plant Layout, Cresap Test Facility, Cresap, West Virginia

## E. Potential Hazards

The aromatic coal-derived liquids generated in the CSF process are considered the major health hazard in the facility. Worker exposure to this solvent can potentially occur in each of the process areas except the coal preparation area. Potential hazards found at the CTF are shown by process area in Table 2.

## TABLE 2 POTENTIAL HAZARDS BY UNIT OPERATION Cresap Test Facility Cresap, West Virginia

UNIT OPERATION	HAZARD
Coal preparation	Coal dust Noise Trace elements Ionizing radiation
Solvent extraction Solids separation Solvent recovery Hydrogenation Fractionation	Polynuclear aromatics (PNAs) Aromatic amines Benzene, toluene, xylene Hydrogen Sulfide
Carbonization	Polynuclear aromatics Aromatic amines Benzene, toluene, xylene Trace elements Ionizing radiation
Environmental units	Hydrogen sulfide Mercaptans Noise

### II. ENGINEERING CONTROL TECHNOLOGY

### A. Introduction

A two part discussion of each process area in the Cresap Test Facility is presented. The first part consists of an area process description. The second part is a discussion of the potential hazards associated with that process area, and the engineering controls used to mitigate those hazards. The term engineering control means the use of hardware (e.g., ventilation systems, mechanical seals, or special metallurgy) to eliminate or reduce an occupational safety or health hazard. Work practices (e.g., protective clothing, monitoring instruments and work scheduling) as a means of mitigating occupational safety and health hazards are discussed later in the report.

The engineering controls discussed in the following sections are largely equipment, piping, and instrumentation modifications that have resulted in increased plant reliability. Increased plant reliability means less fugitive emissions, fewer accidental releases, and less equipment maintenance. For the plant operating and maintenance personnel, this means less exposure to potentially hazardous substances. Unless stated otherwise, this is the health benefit implied by the engineering controls discussed below.

### B. Coal Storage and Preparation (Area 100)

(1) Process Description

Pittsburgh No. 8 coal is trucked into the plant from the Ireland mine which is located about 2 miles north of the CTF plant. Coal is dumped from the truck into either of two 10-ton bins located below ground level. The coal is transferred from the unloading bins to a bucket elevator by a Syntron feeder and a belt conveyor with the belt- and elevator-feed port being located about 15 feet below grade in a fan-ventilated and heated sump. A bucket elevator lifts and dumps the coal into a hammermill for coarse grinding, below which is a three-ton covered surge bin. The coal from the surge bin is ground to 100 percent minus 28 mesh, 70 percent minus 100 mesh particle size at the rate of 3 to 5 tons/hr in a Williams Patent crusher. Hot 1500 F (816 C) combustion gas is mixed with recycle gas to give an outlet temperature of 180 F (82 C). The gas sweeps the ground coal from the mill through a spinner separator to a cyclone collector at the top of the structure and dries it to 1 to 2 percent free moisture. The major portion of the clean gas from the cyclone is recycled back to the roller mill and a minor portion is purged through a baghouse. The mill and cyclone operate below atmospheric pressure while the remainder of the system operates at positive pressure. The entire system is monitored to make certain that the oxygen concentration remains below 15 percent. Nitrogen is available for purging should the oxygen concentration exceed 15 percent. Part of this system is continuously blanketed with nitrogen.

Coal from the cyclone drops through a rotary feeder to a screw conveyor. Fine coal from the baghouse drops through another rotary feeder to the same screw conveyor which conveys it to a Syntron vibrating screen. Over-size coal is returned to the mill and product grind drops to a 70-ton storage bin. Screen, conveyor, and bin are all purged with stored nitrogen, or with inert gas generated on site.

(2) Control Technology Assessment

Most of the CTF plant is positioned in above ground, open spaces that do not permit the collection of potentially toxic gases in "dead" spaces. However, the coal bins that feed the vertical bucket elevator to the crusher are located about 15 feet below grade. To avoid collection of hazardous gases the sump is both heated and fan ventilated. A two-sided shed above the bins reduces dust emissions.

Coal is pneumatically conveyed through the crushing, classifying, and drying system by an inert gas. This system is arranged so that, in the event of failure of the inert gas generator, the unit automatically switches over to a back-up nitrogen system. A portion of the coal crushing and drying system is operated at a slight vacuum. To keep oxygen out of the system, as much equipment as possible has been welded rather than flanged to reduce leaks.

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Another safety feature of the coal pulverizing and drying system is an interlock that automatically shuts down the pulverizer when oxygen levels reach 15 percent oxygen. At 16 percent oxygen a coal dust explosion is possible.<sup>1</sup> The inert gas generator at times produces a gas with over 1 percent oxygen. Feeding this gas to the pulverizer does not cause an automatic shut down.

The interlock system and the nitrogen back-up system are designed to prevent a coal dust explosion. If these systems fail, blowout doors are located in the baghouse to safely release the explosion and direct it away from the process area.

The plant did experience a small fire in the baghouse, but there was no explosion and the plant minimized the damage by quickly extinguishing the fire. The fire occurred when a baghouse door was opened to allow workers to enter the baghouse to change the filter bags.

An induced draft fan is used to pull coal drying gases through the baghouse. If this fan creates a negative pressure in the baghouse, outside air will be drawn in possibly producing an ignitable or explosive mixture with the coal fines. To prevent this occurrence, inert gas is supplied to the baghouse to keep the pressure above atmospheric. A safer and more efficient means of solving this probelm would be to install an oxygen monitor on the discharge of the baghouse.

## C. Solvent Extraction (Area 200)

(1) Process Description

Coal is dropped from the storage bin through a rotary valve into a stirred, baffled, slurry mix tank which is supported by weigh cells. There it is blended with recycle solvent and fresh hydrogenated solvent in predetermined amounts. In the tank, the slurry is mixed with an agitator and blended

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'Plant Engineering Handbook, 1950, p. 2-20.

from bottom to top with a centrifugal recycle pump. The residence time in the slurry mix tank is 30 minutes. Once mixed, the slurry is fed to the continuous withdrawal system feed tank. A centrifugal slurry circulating pump feeds the slurry through either of two White triplex reciprocating pumps (or alternately through a Wilson-Snyder triplex reciprocating pump) with excess slurry circulating back to the mix tank.

The mix tanks are kept under approximately 15 psig inert gas pressure at all times. The plant is designed for a coal feed rate of about 1700 lbs/ hr (20.4 TPD). The feed rate through the White pump to the high-pressure section (550 psig) of the plant is approximately 8 gal/min. Coal concentrations range up to 40 percent, but usually consist of 15 to 25 percent of the slurry.

The slurry is heated by radiant heat in a natural gas fired preheater. This unit contains a heating coil 704 feet long, made of 3/4-inch pipe. No hydrogen is added to the slurry either before or after the preheater. Should a pump fail, the slurry is immediately diverted to a holding tank, and the preheater is blown with highpressure nitrogen to prevent coking.

From the preheater the 800 F (425 C) slurry passes to the top of the extractor (dissolver) and flows downward to the bottom of the vessel. The extractor is a vertical, compartmentalized, stirred vessel with sufficient volume for approximately 1/2-hour residence time. The extractor runs at approximately 450 psig or at sufficient pressure to insure minimum vaporization of the solvent at 725 to 750 F (385 to 400 C). The residence time in the vessel is normally sufficient to complete all dissolution that will take place at the particular conditions of operation and thus stabilize the slurry. Downstream from the extractor the slurry flows through a knockout pot for removal of agglomerated solids. After the knockout pot removes the solids, the slurry is passed through a flow control/pressure reduction valve, where pressure is reduced to approximately 150 psig. This pressure reduction permits some of the dissolved gas to flash at the valve and can also cause some solvent flashing.

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Hydrocarbon vapors and non-condensable gases are formed as a result of thermal cracking and hydrogenation of the dissolved coal/solvent mixture in the extractor. This gas-vapor mixture includes methane, the higher hydrocarbons, hydrogen sulfide, carbon monoxide, and carbon dioxide and amounts to about 3 wt percent of the coal fed to the extractor on a moisture-ash-free basis. It also includes 2 to 5 percent water.

These gases, water vapor, and light hydrocarbon solvent vapors are exhausted from the top of the reactor and cooled. The gases go to the Stretford unit, and the condensed light oil and water forms an emulsion which is sent to the Carbonization Section (800 Area).

(2) Control Technology Assessment

The coal slurry tank is arranged on load cells. The proper slurry content is achieved by weight calculation. Solvent is pumped into the slurry mix tank to the proper weight and then the proper amount of coal is fed to the mix tank. Residence time is approximately 30 minutes and the use of agitators maintains the coal in a suspended state so that it can be pumped to the extractor. Currently, mixing of coal and solvent is a batch process. There would be less line plugging if a method for directly measuring and adjusting the slurry density and viscosity was available; however, densiometers and viscometers failed in this service.

The CTF installation uses Wheatley and Wilson-Snyder pumps to transfer the material from the slurry mix tank to the extractor. Both pumps have spring loaded ball checks, however, the check valves on the Wilson-Snyder pump stick more frequently. This may be because the Wilson-Snyder pump operates intermittently. Other problems with the Wilson-Snyder pump includes:

- Cavitation, and as a result, loss of flow to downstream equipment.
- Leaking seals because of solids in the slurry.

The only problem incurred with the Wheatley pumps was leaking packing. The packing is replaced as a part of the plant's routine maintenance program.

Durmetallic seals, double units with a tungsten carbide face, are used on the slurry mix tank circulating pumps. These seals work well.

Durmetallic seals are also used on the stirrer in the slurry mix tank. Double mechanical seals are used at the top of the unit and a single seal with an oil flush is used at the bottom. The plant experienced erosion problems with these seals.

In order to prevent coking in the preheater the procedure is to blow the slurry out of the heater coil with nitrogen immediately when the flow stops. If coking does occur, a steam and air mixture is introduced to burn off the material that has coked.

Nuclear level controls were used in the slurry mix tank, but foaming caused false readings and this method of measuring the level in the tank has been abandoned.

There were problems with the shaft of the extractor whipping. The problem was solved by substituting a larger diameter shaft. The new shaft is a hollow 6-inch diameter tube with 3-inch stubs. Another problem with the extractor was that coal fines were getting into the shaft seals and causing them to leak. Part of the problem in maintaining the integrity of the shaft seals may be due to the fact that the shaft expands 4 to 6 inches in length going from ambient to operating temperature. The solvent leaking through the shaft seals gets into the bearings on the extractor shaft, causing a loss of lubrication in the bearings. Plant personnel were still working on these problems at the time of the site visit. Because the plant was decommissioned shortly after the visit these problems were never resolved. Several procedures have been tried to eliminate solids buildup at the liquid-vapor interface of the extractor. Two nozzles were installed on top of the extractor to reduce or knock down the emulsions formed at this interface. These nozzles were designed to use hot solvent. This hot solvent spray was subsequently replaced with a cold spray system which eliminated the problem. There was also a solids buildup in the bottom of the extractor. Previously nitrogen was used to blow out solids buildup. Recently, additional paddles ("stubs") were added to the bottom of the agitator shaft. This has eliminated the solids buildup in the bottom of the extractor.

The plant has attempted to measure the torque on the agitator shaft on the 200 extractor. The high temperature in the extractor caused the probe shoe running on the shaft to gall, making it impossible to take any measurements.

Most transmitters in the 200 area are purged with nitrogen, hot flush, or solvent. If the purged lines become partially plugged, the purges can cause the instrument to give inconsistent and inaccurate readings.

A major maintenance problem in the CTF are the letdown valves. The difficulties of the letdown valves are twofold: (1) there is too much pressure drop across these valves; and (2) the flow is too low, allowing solids to settle out and causing the valves to plug up. To reduce erosion of the letdown valves in the 200 area, a new letdown valve made of Cerametal K-701 is being tested. The existing letdown valves in this area are standard 87 percent tungsten carbide, 12 percent cobalt, and 1 percent chrome. With larger size pipes and valves and a greater flow rate, the commercial plant will probably not have plugging problems.

Emulsions from the extractor are dumped into the chemical sewer. This may present a worker safety problem due to the release of hydrogen sulfide, and solvent vapors from the emulsion.

Slugs of solids cause pluggages in the knockout pot system following the extractor. A commercial operation would use a centrifuge to remove solids which would eliminate this problem.

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D. Solids Separation (Area 300)

## (1) Process Description

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The slurry from the extraction section flows to the feed surge solids separation section. Here, dissolved gas is separated i slurry. The solids in the feed surge tank are kept in suspens agitator and by recirculating the contents of the tank with a pump. The contents of the feed surge tank can be circulated t gas-fired heater, if necessary, to maintain temperature.

Slurry from the feed surge tank is fed to the 4200-gallon Cons settling tank at approximately the center of the unit. This v relatively large in diameter, thus providing a quiescent pool slurry. The solids (about 95 percent minus 325 mesh) agglomer settle, thus speeding the settling action. Clear product, a m 20 percent liquified coal and 80 percent solvent containing le percent solids is decanted from the overflow weir at the top c vessel. This clear product flows to the 500 Section product f

AnswerThe solids are swept toward the bottom discharge of the settlenow (a)a scraper moving at a rate of 2 rpm.: Underflow material, contapprox (a)approximately 25 percent solids, goes to a heated circulatingmaterial is fed to the low-temperature carbonization (LTC) secapproximatesolow (LTC) secapproximaterate of 3 gal/min.

(2) Control Technology Assessment

The biggest probleme in this area is plugging of the bubbler-tycontrollers used in the settlers.

Area 300 is running on the straight gravity settling system we per value to 321 C to 321 C). The settler mode allows the solids concentration is settler to average around 15 to 20 percent. The solids concer

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material going to Area 800 can run as low as 6 percent and as high as 40 percent in some streams. The overflow runs from 0.5 to 0.7 weight-percent solids.

There is considerable erosion in Area 300 because streams with high solids content are being pumped. Double mechanical seals are used on the pumps in this area and on the shaft of the rakes in the settlers.

There is no method of determining the percent of solvent in the extract product directly. At the present time, samples are taken to the laboratory for analysis. One of the problems is that the extract must be kept hot. Aging has an effect which is unknown at the present time.

The settler in the 300 Area contains hydrocarbon liquid at 600 F (316 C) and 150 psig which will vaporize if a leak develops. With a source of ignition, this could lead to a fire. There have been a few solvent releases in Area 300, with one incident of ignition. A 30# dry chemical extinguisher was used to put out the fire which was so small that only six pounds of chemical was needed to extinguish it. However, a fire in the 300 Area is particularly dangerous because the third level of the 300 Area is enclosed and the other process areas are in close proximity. This creates a problem in being able to properly combat and contain a fire if one should occur. Solvent leaks are easy to detect because they show as smoke.

Vapor streams from the 300 Area containing process solvent and water are condensed and collected in the separator drum, Tank 318. Tank 318 is not a very efficient separation unit because the density of oil is very close to that of water. Fortunately, this does not affect the process until Area 800.

The 302 emergency dump tank is equipped with a full orifice. This orifice is effective; however, it does away with the flexibility that may be required in the operation of this unit.

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Drum-out procedures in the Cresap plant allow fumes to escape into the area even though an enclosure as been erected around the dump to remove the fumes. In a commercial plant, this system would be eliminated and a method devised to handle materials directly from the bottom of the flash still.

Maintenance must obtain a line-breaking permit, and Plant Modification Request (PMR) before beginning work on any process lines. Plant modification requests (PMR) must be obtained to ensure that piping and valve specifications as established by Fluor for the Cresap Test Facility are adhered to. As many lines as possible are welded instead of flanged to reduce the amount of leakage. In the event of repairs, the lines are cut with a saw and then rewelded.

No plugging problems have occurred in the Area 500 feed tank because solids are removed from the bottom of the tank. Originally the area used LaBour pumps, but most of these have been replaced with Pacific pumps which have a better maintenance history. Hot slurries cause frequent leaks from the pump packing and seals in this area.

There are no pumps on the commercial market which are designed for pumping hot slurries. Turbine Metal Technicians from Burbank, California, diffused titanium (from titanium bromide) into the casing metal. Due to the doping that is required for the different metals, porous castings result. An increase in temperature increases porosity of the metals in the pump, causing a leakage of the process fluid through the pump casing. In addition, flakes of metalized material peel off the casing resulting in erosion at the exposed surface. Normally the leaks on the pumps are seal fluid leaks which are usually at much lower temperatures than the bulk liquid in the tank.

E. Extract Solvent Recovery (Area 500)

(1) Process Description

In Area 500, liquor from Area 300 is separated into light, medium, and heavy fractions. The liquified coal/solvent product is pumped to the

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extract solvent recovery section where the material is first preheated to 600 F (325 C) at 80 psig, then flashed across a pressure control valve into a flash still at minus 5-inches water pressure (14.3 psia). A part of the unflashed liquid, now rich in dissolved coal, is recycled back to the flashstill heater while the remainder is pumped to the catalytic hydrogenation section (Area 600). If the hydrogenation section is not operating, the product may be stored as 50 percent dissolved coal, 50 percent solvent in either of two stream-heated, 10,000-gallon storage tanks. Alternately, flashing conditions can be made more severe to vaporize most of the solvent, so that the dissolved coal may be drummed out or instantaneously solidified and shattered (granulated). It can then be pumped as a water slurry to the disposal pond at the north end of the plant.

The vapor from the flash still is taken off overhead and fed to the 50-foot vacuum column at the sixth tray level. A light distillate is taken off at the top of the column while the heavier middle distillate is taken off at the reboiler. The middle distillate recycle solvent is either pumped to the tank farm for storage or recycled to the coal mix tanks. A light distillate cut is taken off at the twentieth tray and is pumped to the tank farm for storage.

The overhead material from the vacuum tower is condensed and separated from any water or gas that is in the flash still feed. The hydrocarbon portion is recycled to the tower at the twentieth tray. Water and steam condensate from the steam ejectors, which create the vacuum in the vacuum column, are drained to a hot well. Because this water may contain hydrogen sulfide (H S), it is pumped to the utilities section for further processing.

(2) Control Technology Assessment

Area 500 is controlled based upon the pour-point of the solvent-extract mixture. The lab checks the percent of solvent in the extract and, on this basis, the plant's operating parameters are established. The percent solvent in the extract is checked with a vacuum heat scale. Samples are taken to the lab every two hours while the plant is being put on stream. Then one sample is taken every four hours. Solidification of the sample

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makes this sampling difficult. Because samples are collected in one-quart open cans, drawing and analyzing samples exposes field operators and laboratory technicians to potentially harmful organic materials. In addition an operator received second degree thermal burns as a result of a fall while carrying a sample. Enclosed sample bombs should be used in collecting samples. This is also a poor control procedure because of the lag time between drawing the sample and receiving the analysis, and because information concerning the performance of the 500 area is available only twice per shift. This has led to the buildup of solids in this system. Removing these solids increases the potential for maintenance worker exposure to harmful process constituents.

As a general rule, the extract is not stored after Area 500. If interim storage is required, the storage tanks are maintained at 300 F (149 C) or higher. The hot extract, up to 50 weight percent, can be moved to the heavy-oil tanks where it is circulated in the tanks at approximately 340 F (171 C). If the extract is maintained at this temperature, it can be moved back to Area 600 for recovery. On one occasion, the extract floated on the top of the tank's contents, and as it cooled this material precipitated, necessitating a clean-out of the tank.

Close-fitting API pumps are used at the extractor granulator; however, these pumps do not have the capacity to move this material out of the quench tanks to the tank farm, and consequently these lines plug up. Cavitation also is a problem with these pumps.

Cooling coils in the extractor granulator exhibit polythionic acid stress corrosion. When the plant was shut down in May, the polythionic acid was neutralized by circulating an alkaline material in order to avoid the extra maintenance noted above.

In the 500 Area, the biggest instrumentation problems are the venturi flow indicators. These venturis usually have a hot flush arrangement so that the flush is directed upstream and downstream at the point of injection. Injection occurs at a number of locations in the line.

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In addition to the flow indicators, there are plugging problems with the level control float in the flash still in Area 500 because of the heavy material being handled. Level indicators that operate on the differential pressure arrangement have never worked satisfactorily because of the heavy material which clogs the end of the differential pressure tubes. A nuclear high-level indicator has been used; because of drift, it has not worked satisfactorily. At the present time, the unit needs resetting.

In order to prevent the solidifcation of process materials in the plant piping, especially during the winter, lines containing high boiling point material are electrically traced and lines containing lower boiling material are steam traced. All piping would be electrically traced except that there is not enough electrical capacity in the plant to electrically trace all the lines.

The vacuum still must be a leak tight system because air leakage into the still can create an explosive mixture. The integrity of the system is checked each time the plant is shut down. There is no preventive maintenance program for the vacuum towers. This is so that the integrity of the vacuum system is not disturbed unless repairs are actually required.

To date there have been no problems with air leakage into the vacuum tower. Oxygen levels in the vacuum tower are checked by means of a gas sample which is analyzed in the laboratory.

Vapors are controlled at the drum-off station by using an exhaust fan to pump the off-gas vapors to the atmosphere at the top of Area 500. However, in a commercial plant, the material would be handled in a completely enclosed system so the problem of vapors affecting the personnel would be minimized.

Pacific pumps are used in this area, with the exception of one LaBour pump. The fact that the pumps in this area must operate against a vacuum causes some pump problems in this area. Pump seals are being replaced with double mechanical seals which are flushed with a seal oil.

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## F. Extract Hydrogenation (Area 600)

## (1) Process Description

The solvent dissolved coal product from the extract solvent recovery area (extract) is stored in the extract feed tank which is a stirred, electrically heated tank mounted on weigh cells. Material from this tank is fed to the Mattin-Goulin reciprocating high-pressure charge pump. The maximum design feed rate is about 2.6 gal/min or 930 lb/hr dissolved coal in a mixture containing 76 percent dissolved coal and 24 percent solvent. (The normal feed mixture under present operating conditions has been about 30 percent dissolved coal and 70 percent solvent.)

The reactors in this process are 1.0 feet inner diameter and 33 feet, 8-inches high. Feed enters the first reactor under the grid plate along with recycled reactor overflow and hydrogen. Reactor operating conditions are 800-850 F (428-455 C), 3000 to 3500 psig, 80 percent hydrogen partial pressure, and a superficial velocity on the order of 0.4 to 0.5 ft/sec depending on temperature. The first reactor contains no catalyst and acts as a preconditioner for the feed charge to eliminate light ends from the feed. Some hydrogenation does take place in the first reactor. Height of the fluid (ebullating) bed is determined by an offtake weir. Material leaves the reactor in two ways; either it is carried overhead as a vapor along with the unreacted hydrogen and product gases, or it overflows the weir. Because the heat generated by the exothermic hydrogenation reaction is insufficient to offset heat losses from the reactor system, the overflow liquid is passed through a fired heater before it is recycled back to the reactor by a Chempump-brand centrifugal pump at a rate of about 25 gal/min. In contrast to the H-Coal reactor recycle arrangement, all recycle operations are external to the reactor in order to reduce the reactor volume. Excess overflow (equal to feed rate less vaporized and gasified product) is pumped to the second reactor, which contains the catalyst charge.

Final velocity in the second reactor is determined by the expansion of the reactor catalyst bed (1/16-inch diameter extrudates of cobalt and molybdenum on an alumina base) with 50 percent expansion judged to be maximum. Bed expansion is determined by two nuclear density gauges located on the sides of the reactor. Experience indicates that fresh catalyst is less dense than catalyst which has been exposed to the product for several runs. (Operating conditions and sequence in the second reactor are similar to those in the first.)

Liquid overflow product from the second reactor is reduced from reactor pressure to ambient pressure of the feed surge tank in the hydroproduct fractionation section (Area 700) through a single control valve.

Vaporized product and gas from both reactors is passed through two sequential cooling systems. In the first cooler the gases and vapors are cooled to about 350 F (175 C) and the condensed heavy hydrocarbons are separated from the gases. The remaining gas is cooled to 100 F (38 C), and the cooled gas is separated from the light hydrocarbons and water. Water and light oil are separated in a decanter. The water is pumped to the water treatment section. Both the light oil and the heavy product oil are depressurized in three stages. Dissolved gas from all stages is sent to the Stretford unit. The liquid hydrocarbon product is accumulated in the feed surge tank in the hydroproduct fractionation section.

To maintain a high hydrogen partial pressure in the recycle gas stream, a portion of the gas stream is purged to the Stretford unit. The rest of the recycle gas is passed through an absorber train to remove light aliphatic hydrocarbons in the  $C_1$  through  $C_5$  range. The gas flows counter-current to the absorber oil, which is a  $C_6$  boiling range fraction (320 F (160C)) recovered from the hydroproduct. The gas is then recompressed and returned to the reactors. Sufficient fresh hydrogen is added at this point to maintain the 85 percent hydrogen partial pressure. The light aliphatic hydrocarbons are desorbed from the absorber oil is recycled back to the absorber. Desorbed gases are sent to the Stretford unit.

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Catalyst is loaded into the reactor only when the unit is offstream and cool. When catalyst is removed from the reactor, it is first washed with a light oil, then fluidized. A valve at the bottom of the reactor is opened and the fluidized slurry flows in a controlled manner into a drum. The catalyst is disposed of in an appropriate landfill.

(2) Control Technology Assessment

The heavy fraction from the distillation area is hydrogenated in this area.

Material is fed from the extract feed tank to the J607, Mattin-Goulin charge pump. This material is composed of approximately 76 percent coal extract and 24 percent solvent. To improve the reliability of the instrumentation in the feed drum system, a solvent, which is a 50/50 mixture of toluene and filtered recycle solvent is used to purge the instrument lines.

Chevron packing was originally used on the charge pumps; however, the packing, which was changed as needed, tended to tear at the hot end of the pump. U-Tex glass-filled Teflon molysulfide-impregnated packing has been used for one year.

The plunger on the charge pumps has been scored by solids. To prevent this scoring, oil is injected at a number of points; however, the process flowrate is so small that the process cannot handle the extra flow of lubricant that would be necessary to prevent scoring.

The reactor contents are controlled by apparent density which can be changed by temperature and gas flows. Temperature excursions in the reactor are controlled by the recycle rate, hydrogen purity, and the temperature of the recycle which normally runs at 850 F (454 C). The level in the reactor is maintained by differential pressure cells.

In the second reactor a preheated feed is used which is fed directly to the catalyst reactor to avoid carryover of the catalyst. There is no catalyst carryover if the temperature is 500 F (260 C) or higher. There is no water injection in the overhead system. Catalyst is added during shutdown when

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the equipment is depressurized. The catalyst is poured through a funnel into an eductor. Nitrogen is used to transport the catalyst into the reactor at the 20' level. An automatic catalyst charge system that introduced catalyst into the reactor at the 38' level is not used because the 38 foot drop to the bottom of the reactor at the start of the charging operation caused too much catalyst breakage. On occasion, the catalyst has been entrained and carried through the recycle compressors. This backflow can be reduced by capping over the pipe which is used for flow up through the extractor. GRAYLOC flanges work well; however, they are expensive and have a long delivery time. Ring joint flanges have been used successfully.

In the event of excessive bed expansion in the catalytic reactor, catalyst is carried over, causing pluggage in the lines, letdown valves, and pumps in the 600 Area. Nuclear level indicators were installed to monitor the height of the catalyst bed and prevent catalyst carryover.

Rockwell letdown valves were used in Area 600 but Foxboro valves are now being used. In order to reduce the wear on the letdown valves, a tungsten carbide solid plug was brazed to the stem. This plug was not successful because the difference in the coefficient of expansion of the two materials caused the valve trim to shatter when a tight shutdown was required. Therefore, this brazing technique was replaced with a pinned assembly technique which eliminated the problem. An experimental letdown valve was also used. This valve was too large for the flowrate through it, causing it to operate too close to the fully closed position and making it difficult to control flow with this valve.

A Chempump is being used to recycle overflow back to the reactor. This Chempump has been modified by cutting a groove in the rotor for better lubrication. The oil used for lubrication of this Chempump is externally cooled. At present, Legion 55 oil is used. The oil which is used to lubricate the seal goes into the process. The seals on the Chempump are Teflon discs with stainless steel support. In order to reduce the amount of wear on the pump, vibrations on the unit were measured and necessary adjustments made. Occasionally product backs up into the seal oil, and the very fine solids suspended in the process fluid damage the seals of the pump.

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The hydrogen compressor has experienced considerable problems with water in the inlet gas. Currently, it is on an hourly blowdown schedule.

Knockout pots have been used to prevent liquid from getting back to the recycle gas compressors. A letdown valve lowers the pressure from 3000 psig to 150 psig. The knockout pot uses a seal ring which is a Viton o-ring with a backup Teflon ring. Installation of the oring is critical because the knockout pot handles hydrogen sulfide and care must be taken when assembling it. When exposed to high temperature, the knockout pot locks tightly onto the o-ring. A high temperature lubricant is used to help with the o-ring installation.

The 602 tank, which is a water/liquor separator, separates gas, water and organics. The instruments on this unit were adjusted to eliminate foaming. There is no evidence of ammonium sulfide crystallization.

Hills McCanna units are used on the instrument purge pumps. These units give much better service because there is no maintenance except for packing adjustments.

G. Hydroproduct Fractionation (Area 700)

(1) Process Description

The hydrogenated coal extract is fractionated into light, medium and heavy fractions in this area. This section consists of two columns and the appropriate heaters, pumps, and storage. The first column is a stabilization column used for removing hydrocarbon gases, particularly  $C_3$  through  $C_5$  gases, from the hydroproduct. (In the pilot plant these gases are sent to the Stretford unit.) The bottoms from the stabilizing column are then sent to a 30-plate fractionation column which is capable of producing three product cuts--a light oil, a middle distillate, and a heavy distillate. The lighter fractions are stored in the tank farm. The heavy distillate fraction is recycled to the coal mix tank as about 20 percent of the total slurry oil feed to that tank. It is this fresh heavy distillate fraction which supplies the fresh hydrogen donor capabilities of the coal slurry solvent. Excess donor solvent is also stored in the tank farm.

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## (2) Control Technology Assessment

The greatest problem in this process area is leaking pump seals. The process product in this area is gummy and adheres to the pump seals, causing leaks when this material is oxidized and becomes hard. Experience indicates that mechanical seals are best for this service. In addition, the plant has experienced problems with the charge pump and the circulation pumps at the cyclones. Catalyst has not been observed in the system beyond the pumps except on two occasions.

Multitube, fin-fan coolers are used in Area 700. When the tubes plug, they cool and pull from the tube sheet. (This problem also exists in Area 600 but is greater here because of the potential for the movement of liquids to the overhead tubes and the settling out of solids in the lower tubes.)

In Area 700, electrically heated reboilers are used because of the high temperatures required in this operation. Solids build up on the heating coil of the vacuum reboiler. These solids absorb acidic materials resulting in holes in the coils. In addition, because low-boiling point compounds have boiled from solution, it is necessary to steam trace the storage tanks to keep the remaining high-boiling point compounds liquid.

## H. Low-Temperature Carbonization (Area 800)

(1) Process Description

The low temperature carbonization section, which operates at 875 to 925 F (470 to 495 C) and up to 15 psig, is designed to recover solvent from the mineral residue separated from the extracted product in the solids separation section (Area 300). This residue, containing solvent, coal extract, and very fine coal particles, is sprayed into the fluid bed of char. The heat of the bed causes the solvent to flash off. The extract and minus 325 mesh particles coat the particles in the fluid bed. The large surface area of the bed particles means that each particle receives only a thin coating of extract which rapidly pyrolyzes, leaving behind char particles. If

pyrolysis does not occur rapidly, the bed particles will agglomerate with any other particles it touches with possible growth sufficient to cause plugging of the bed. (Theoretically, the char particle size in the carbonizer should be a function of rate-of-bed turnover, though the operations are not sufficiently instrumented to show this.)

Material from the Consol precipitation unit underflow receiver is pumped to the carbonizer. Feed rate to the carbonizer is determined by level control of the underflow receiver. The underflow material is fed into the bed through a fully open, swirl-type spray nozzle pointing down and centered at, or just above, the bed. Ports exist in the carbonizer to relocate the nozzle within the bed to positions as low as three feet above the grid. (Experiments at the Cresap plant and experiments at the Synthane and  $CO_2$ Acceptor plants have found that location of the feed point within the bed results in a diminution of liquid product.)

The fluidizing gas for the carbonizer bed consists of recycled product gas from the carbonizer with sufficient air added to maintain temperature by oxidation of the char particles. A 6-inch-diameter "lift leg" operated with recycle gas is incorporated within the carbonizer bed to increase the bed circulation rate. The superficial velocity of the primary bed varies between 0.6 to 1.1 ft/sec and "lift leg" velocity is about 10 ft/sec. Bed particle size averages approximately 200 mesh. (There is currently an effort at the Pittsburgh Energy Technology Center (Bruceton) to define the operating parameters of a fluid bed with an internal "lift leg".)

The product gas and vapors pass sequentially through two internal cyclones and are immediately quenched with  $347 \ F$  ( $175 \ C$ ) condensate oil from a downward-directed spray nozzle. (Failure to quench properly will result in condensation of the heavier tars above the spray which will eventually block the product gas line.) The gas is then fed tangentially to the bottom of a quench tower where it is scrubbed through four sequentially located sprays with the  $347 \ F$  ( $175 \ C$ ) condensate. The gas is further cooled to  $120 \ F$  ( $49 \ C$ ) in a water-cooled tube-bundle and then separated from condensed light oil and water. (The light oil-water decanter also contains light oil and water from the residue separation and extraction

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sections.) The water is pumped to process water cleanup and the light oil is stored. A portion of the off-gas, which contains carbon monoxide, carbon dioxide, nitrogen, and some hydrogen sulfide and sulfur dioxide, is purged to the Stretford unit section. The remainder is compressed with a Nash compressor, using a recycle solvent<sup>(1)</sup> as the compressor fluid and stored in a surge tank. It is then fed to the carbonizer.

The condensed oil which contains 5 to 10 percent char fines is  $cooled^{(2)}$  in a single-tube flow-through steam-generating cooler. Excess material is returned to the settler in the residue separation section by means of a centrifugal pump.

Char from the carbonizer is dropped by gravity from a standpipe projecting approximately 1/2-inch above the carbonizer grid plate to a water-filled char quench tank. The char removal rate is based on carbonizer bed level and is controlled by a pneumatically-operated "V"-notched slide valve. The water in the tank is circulated through a centrifugal pump which also pumps the char slurry to the char slurry pond at the north end of the plant. Fresh water feed for the char quench tank is brought in on level control.

(2)Cresap I used a cold-water-cooling unit here. Great difficulty was encountered in maintaining a reasonable cooling rate. The heat transfer coefficient would abruptly decrease by 50 percent. This phenomena was attributed to the laydown of a tar layer. Although the cooler was always clean when opened, the cooler was never opened before first cold flushing it for several hours with fresh solvent.

<sup>(1)</sup>During the operation of Cresap I, recycled water was used as the compressor fluid. However, the water became so acidic (pH = 2.0) that the Nash pump rotors were dissolved in a week's operating time. Use of hydrocarbon solvent has ended this problem, but if the carbonizer bed is fluidized cold with recycle gas for any length of time, the bed and the entire carbonizer system will become solvent-saturated.

## (2) Control Technology Assessment

The carbonization unit is a vessel which is fitted with a grid plate positioned on a 15 degree angle. Several nozzles feed the residue into the carbonization unit. No appreciable difference in the operation of the carbonizer has been noted as a result of the way the grid is drilled. (Originally, the grid plate was drilled so that the holes would be vertical when the grid was in place.)

The carbonizer has a draft tube (or internal lift leg) which assists in circulating the bed and thus controlling the temperature. Normal operating temperature in the carbonization unit is 1100 F, with an instrument capability of 1200 F. In one incident, the unit burned out when the temperature climbed to over 1500 F.

Spray nozzles are positioned in the spray chamber at the top, pointing down; and at the bottom of the unit, pointing up. The nozzle that is located approximately 3 feet from the bottom of the carbonizer has been the most effective. Type 316 stainless steel and Type 416 stainless steel spray nozzles have been used in the carbonizer. These nozzles eroded very quickly. In an effort to reduce nozzle wear, hard-faced NiCarb, silicon carbide, and nickel nozzles have been considered. Boride-faced nozzles with an alumina ceramic insert were installed during the week of April 19, 1979. The opinion of the test engineering department personnel was that these nozzles will probably work best in this service.

The low temperature carbonizer is fed at a 2.5 gallons per minute rate varying somewhere between 2 and 3.5 gallons per minute in, but producing 3 to 3.5 gallons. The difference is probably due to the solvent that is being added to the pumps in order to lubricate the seals. Fresh solvent is fed to adjust the concentration of the streams after the carbonizer. The Cresap facility has improved its extract recovery, and as a result now produces finer char particles. A hole was drilled in the bottom of the unit to drain off condensed water.

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Other difficulties in the low-temperature carbonizer include:

1. If the solvent and char mixture in the low-temperature carbonizer does not ignite, the bottom of the unit floods.

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- Agglomeration above the grid plate causes formation of clinkers, which gradually affects the operation of the fluidized bed. This causes formation of additional clinkers, eventually shutting the unit down.
- 3. Double internal cyclones are used in this unit. Dip legs attached to the solids discharge of the cyclones drop material back into the fluidized bed. Differential pressure taps should be installed on cyclone dip legs to obtain data on dip leg solids density under operating conditions.
- 4. When solids are dropped out of the char cooler, the unit is gradually choked off, and the flow of material through the cooler stops. At this time, the plant must shut down because a hydroblaster must be used to clean the char cooler.

Quenching of the gases occurs in the spray tower where serious nozzle erosion exists. The solids concentration at this point is 33 to 35 percent but has gone as low as 5 to 7 percent. The recycle gas which goes to the Stretford unit for sulfur recovery is sour gas.

A flow meter was installed to measure the amount of gas going to the Stretford unit; however, operation of this meter is not reliable because it was installed improperly.

Solids are let down by the use of a slide valve with a diaphragm-type motor to actuate the valve. Dump of the solids is manual. When the material is drummed out, a manual block valve has to be opened to prevent gas escaping through the conveyor. Because this valve is hard to open, gas does escape. Dust covers are used on the drums in an attempt to prevent the material from getting into the workplace atmosphere.

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Pumps in Area 800 are subject to both erosion and corrosion. For example, the char slurry pump is corroded by carbonic acid which is formed by the carbon dioxide present in this part of the process. In addition, corrosion pits have been found in the seals. The tar quench pump operated at 3600 rpm and is eroded by the solids present in this part of the process. This erosion could be reduced if the pump speed were reduced; however, in order to maintain flow capacities, a number of pumps operating in parallel would be required. This may not reduce the erosion problem enough to justify increased expenses.

#### I. Other Areas

#### (1) Process Description

Process-derived water is pumped to a tank in the utility section where an API separator removes floating oil. The process water is then streamstripped to remove hydrogen sulfide  $(H_2S)$ . The  $H_2S$  is fed to the Stretford unit. The stripped water is passed through a carbon filter to remove solids and some discolored oil, and is then sent to the separator.

Plant runoff water is sent to the external water treatment tank along with the filtered process water. Here the solids are settled in a compartmenttype settler and the oil is skimmed by the API separator. Clean overflow water is pumped into the Ohio River.

Purge gas and entrained gas from the plant (except for purge gas from coal grinding) is fed into the Stretford unit for sulfur removal. After passing through the Stretford unit, the sweetened gas is sent to a thermal oxidizer.

The utility area operations include stored nitrogen; compressed instrument air and plant air; steam with attendent water-treatment facilities and boilers; potable water and cooling water; inert gas generation; an emergency power generator; fire protection pumps; the electrical distribution center, and hydrogen manufacture. The hydrogen is manufactured from natural gas by catalytic reforming using a Girdler skid-mounted catalytic reforming unit and a monoethanolamine gas scrubbing unit for carbon dioxide removal.

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(2) Control Technology Assessment

To date, the Eimco automatic pulse sampler for measuring total flow at the wastewater system has been very reliable.

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Resins, which should operate at a neutral pH, were used at the neutralization basin. However, because of the excursion of hydrogen sulfide into the system, activated carbon has replaced the use of resins.

During heavy rainstorms, oil from the 800 Area gets into the storm sewers and does not go through the API system. In addition, heavy oil plugs the lines in the storage tanks and tank farm area.

Phenols and other off specification material get into the streams. The present Fram wastewater treatment unit can only handle 15 gallons per minute and therefore more capacity is needed at times because of the difference in flow of the chemicals contaminates the storm sewer system.

#### III. WORK PRACTICES

### A. Housekeeping

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In the Cresap plant housekeeping activities were the responsibility of the field operators on an as-time-permits basis. Spills were cleaned-up using V.I.S. hydroblasters. Housekeeping efforts were not very successful as evidenced by the amount of deposition of process solvent in the facility and the time required to clean a spill. The ineffectiveness of the program was due to the understaffing of the facility and the use of contract maintenance. Contract maintenance personnel were not accustomed to cleaning up after themselves, and plant personnel were not inclined to clean-up the trash and spills left behind by the higher paid maintenance workers.

### B. Administrative Controls

Administrative controls are procedures designed to reduce a worker's exposure to potentially hazardous substances by limiting the amount of time the worker spends in process areas or work situations where exposure is most likely to occur. Limiting exposure in this way may be accomplished by regular work scheduling, i.e., planned rotation of workers to different work assignments; and/or by rotation only after a large non-routine exposure, such as from an accidental release.

Extensive cross-training was performed at Cresap so that the field operators would be capable of working in more than one process area. However, because of the extensive down-time and short life of the plant, workers were not often rotated to other assignments. Additionally, because of the small size of the pilot plant it would be difficult to place a worker in a safer environment. In any event, known excursions beyond STEL (Short Term Exposure Limit) values did not occur.

### C. Maintenance Procedures

The use of full personal protective equipment is required during the removal and cleaning of process equipment. Equipment must be decontaminated before it is delivered to the shop for dismantling. These policies are strictly enforced.

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#### **IV. PROTECTIVE EQUIPMENT**

The major emphasis of the program regulations is to provide the necessary protection against skin contact with coal-derived liquids through the use of personal protective clothing and equipment. This effort is supplemented by personal hygiene regulations to prevent prolonged contact with PNA-contaminated clothing.

In-plant protective clothing requirements include hard hats, safety glasses, and coveralls. The use of additional protective equipment is job and area dependent and involves:

- The use of rubber gloves by the field operators in all process areas except coal preparation.
- The use of cotton gloves at all times by the maintenance staff.
- The use of raingear in process stream sampling and in activities which require breaking of the product line.

These regulations, especially the use of gloves, are not strictly enforced so the degree of usage depends upon the worker. This reduces the overall effectiveness of the program.

Respirators are not used to control inhalation exposures to PNAs. Instead, exposure control depends primarily upon engineering controls, especially the closed-system operation, and preventive maintenance as described in the preceding sections. However, full facepiece respirators are available for protection against hydrogen sulfide in duties where exposure to this gas is likely to occur.

#### V. MONITORING PROGRAM

The following plant personnel are likely to be exposed to potentially hazardous process materials: field operators assigned to the extraction, solids separation/carbonizaion, solvent recovery/ fractionation, and the hydrogenation/support operations; the laboratory technicians; and the maintenance staff. All are on shift duty except the maintenance staff who work the day shift only.

The industrial hygiene program provided only for the monitoring of hydrogen sulfide levels using detector tubes. Future plans for the Cresap facility did call for an expanded monitoring program for organics such as PNAs, aromatic amines, benzene, and particulates. For hydrogen sulfide approximately 10 to 15 detector tube readings were taken daily with results indicating airborne concentrations of less than 1 ppm. These results suggest that hydrogen sulfide would not be a health hazard within the facility.

An extensive monitoring program conducted by Enviro during November 1978 and April-May 1979 has indicated that workers were primarily exposed to aromatic contaminants in the parts-per-billion and microgram-per-cubic-meter range (Appendix A). The major contaminant was the PNAs for which no health standards currently exist. The benzene soluble fraction is used as a standard for two specific cases, coke oven emissions and coal tar pitch volatiles, however the related cyclohexane-soluble fraction used in the Enviro study showed no relationship with PNA levels. Therefore, the soluble fraction is an ineffective measure of PNA exposure in the Cresap facility.

The absence of a health standard and the lack of toxicologic data for assessing a safe level of exposure preclude the determination of the risk involved with prolonged exposures to PNAs at these levels. However, the suspected carcinogenicity of these compounds suggests that some increased risk is involved for these workers relative to workers without exposures. Therefore, precautions should be taken to reduce worker exposure to PNAs until the necessary data is available.

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#### VI. HEALTH AND SAFETY PROGRAMS

The Cresap pilot plant maintains a health and safety program under the supervision of a health and safety professional to provide workers with a safe working environment. This program is supplemented by a medical program under the direction of a plant nurse to monitor work related illnesses. The unusual feature of this program is the hazard being dealt with and not the program regulations. The hazard of concern is the polynuclear aromatics (PNAs) which are present in the coal-derived solvents used in the process. The program regulations implemented to control exposures to these compounds represent basic work practices found in other programs dealing with general industrial chemical toxicants. An exception is the strict personal hygiene requirements of the plant. A detailed description of the Cresap industrial hygiene program can be found in Appendix B of the Enviro report to NIOSH titled "A Study of Coal Liquefaction Processes: Industrial Hygiene Comprehensive Survey, Cresap Test Facility, Cresap, West Virginia."

## A. Employee Education and Training

The industrial hygiene education and training program repeatedly emphasized the danger of hydrogen sulfide because of the acute toxicity of this gas. Other hazardous agents including catalysts, ammonia, organic solvents, and PNAs were discussed but did not receive the same emphasis as hydrogen sulfide.

### B. Safety

The CTA team was primarily interested in information concerning the industrial hygiene program. Therefore, information specifically concerned with safety was not gathered.

## C. Industrial Hygiene

The plant personal hygiene requirements have been instituted to provide supplemental protection against the potential skin carcinogenicity of the PNAs. The objective of this program is to minimize worker contact with contaminated clothing. This objective is achieved by the following procedures:

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- Upon reporting to work the employee enters the locker room and changes into a clean set of work clothing. The company provides a sufficient number of uniforms to allow for a daily change of uniform.
- At the end of the shift used clothing is deposited in the appropriate receptacle located in the change room. The worker then uses the foot bath and shower facility before entering the locker room to don street clothes.

Extra uniforms are available in the event a worker is drenched with process solvent. All workers are urged to change uniforms as necessary during their work shift to prevent unnecessary contact with contaminated uniforms.

In order to ensure one hundred percent acceptance of the personal hygiene program, workers who refuse to participate may be fired. In order to encourage voluntary participation, time-and-a-half pay for showering is provided.

#### D. Medical Program

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The possible occurrence of skin abnormalities through PNA exposures is monitored by pre-employment and annual skin examinations. These examinations are performed by a local physician and include a check for lesions and other abnormalities. The physician was provided with information regarding the health effects of PNAs. Quarterly supplemental examinations are provided by the plant nurse. The results of these examinations have shown two cases of mild topical dermatitis which were not believed to be job-related.

The other aspects of the medical program are not directed towards the monitoring of the potential adverse effects of PNA exposure. Instead, the program provides a means of establishing a baseline of the worker's health and a general screening of his health on an annual basis. Prior to the closing of the Cresap facility, a more extensive monitoring program had been approved by the corporate office which would have expanded the parameters being monitored. This expanded baseline would have included additional blood tests, audiometry, and pulmonary function tests, and a change in plant physician. VII. CONCLUSIONS AND RECOMMENDATIONS

In order to prevent plugging of instrument piping with process material, the lines on the differential pressure transmitters should be kept short, located above the process fluid, and/or purged with hydrogen or some other purge fluid.

The CTF instrument department recommends using nuclear instruments for measuring level since they are non-contacting devices and are therefore not subject to plugging.

Because of problems with bridging and freezing of wet coal, the instrument department suggests the use of conductivity probes in the coal feed bins to indicate moisture.

In order to eliminate the problems of leaking shaft seals and solids settling in the extractor, CTF engineers suggested replacing the extractor with a pipeline reactor which would pass through a fired heater. The pipe diameter would be selected so that the slurry velocity would be high enough to prevent solids from settling out in the line.

A study should be made of magnetic flow-meters or ultrasonic differential pressure meters to measure the flow. The problem with the ultrasonic meters is that the wall has an effect and the process stream must be aerated in order to get a reading.

In order to provide the operators with data they need to operate the plant smoothly and efficiently, a method for directly reading (determining) the percent solvent extract in the reactor product is needed.

In order to determine the density of the extract coming off the units, it has been suggested that a Dynatrol in-line probe be used to measure density. This Dynatrol densimeter has a temperature compensator built into the probe so that it can be used in the system over the range of operating parameters.

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Although some of the lines are steam-traced because of the lack of electrical capacity, all lines of commercial plants should be electrically traced because of the higher temperatures and better reliability of electrical tracing.

- In order to improve operations, it has been suggested that a star valve be inserted in the lines to remove the solids from the bottom of the carbonizer unit.
- The carbonizer should be ignited with a pilot light instead of the autoigniter.

It is difficult to measure the level of the fluidized bed because the thermal conductivity of the bed is too low, and it has been suggested that a nuclear device be used to measure the bed level of the unit while the unit is in operation. It has also been suggested that the oxygen be measured with the use of a Westinghouse probe.

Some of the operating problems in Area 800 result when the plant shuts down, at which time char falls through the grid of the ebulating bed. To eliminate the problem, plant personnel have suggested replacing the grid with bubble-cap plates or random-sized ceramic balls plus air diverters.

Erosion probably would be reduced in pumps handling erosive slurries, if the pump speed were lowered to 1750 rpm.

The entire treatment plant should be redesigned in order to recycle wastewater back into the system after treatment.

A safety training program, similar to the one for hydrogen sulfide, emphasizing the potential hazard of PNA exposure should be created to obtain better worker acceptance of the PNA regulations.

Provision of time at the end of the shift and/or the assignment of housecleaning activities to a single worker would be a more effective means of

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keeping the process areas clean than depending on the supervisors to assign clean-up activities on an as-time-permits basis.

There was an established Personal Protective Equipment (protective clothing and respirators) program. During normal operations, such as housekeeping and preparation of vessels for maintenance, workers may be exposed to the various hazardous substances by inhalation and dermal exposure. Skin exposure was controlled by appropriate equipment such as gloves, coveralls, shields, special work clothing, and barrier creams. A number of different types of respirators are available at Cresap to protect workers from inhalation exposure. Escape type respirators, Robert Shaw 5 minute Air Masks, are located throughout the process area for workers to wear during emergencies. Half-mask respirators with disposable MSA type N canisters are available for workers exposed to dilute concentrations of acid gases and organic vapors. MSA 401 and Surviv Air SCBAs with a thirty minute air supply are available for emergency use in the event that a contaminated area must be entered. They are located in the control room and in the process area in sealed cases. Combination supplied-air respirators with self-contained breathing apparatus are not used at Cresap for vessel entry. This is because no employee is allowed to be in a vessel that has not first been purged, flushed, chemically cleaned, and hydroblasted, and whose atmosphere is not being monitored continuously while work is in progress. Since the atmosphere in the vessel is not immediately dangerous to the life and health of the worker, SCBAs are not required protective equipment.

Cresap's tank and vessel entry permit procedures and respirator program were rated above average by OSHA inspectors and may be used as a model for the development of similar programs at future coal conversion plants.

Samples should be taken in enclosed sampled bombs rather than in open containers to reduce worker exposure to process materials, help preserve the sample, and reduce the risk of thermal burns.

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Comprehensive industrial hygiene monitoring should begin before the plant starts operation in order to establish base line measurements, and continue while the plant is in operation until all known and suspected hazards have been characterized.

One shortcoming of the medical program at Cresap was that the plant physician was not experienced in industrial medicine. Actions such as developing a standardized checklist of examinations and diagnostic tests for the physician to complete may help insure that symptoms related to exposure to toxicants found in coal conversion plants are not overlooked.

•••

## REFERENCES

- Enviro Control, Inc., December 1979. <u>Industrial Hygiene Walk-Through</u> <u>survey, Cresap Test Facility - Cresap, West Virginia</u>. Contract No. 210-78-0101. National Institute for Occupational Safety and Health, Morgantown, West Virginia.
- Enviro Control, Inc., May 1980. <u>Industrial Hygiene Comprehensive</u> <u>Survey, Final Report, Cresap Test Facility, Cresap, West Virginia</u>. Contract No. 210-78-0101. National Institute for Occupational Safety and Health, Morgantown, West Virigina.

### APPENDIX A

# Enviro Comprehensive Survey Results for the CSF Process Cresap, West Virginia April and May, 1979

The following tables give the analytical results for the samples taken at Cresap, West Virginia. Tables are arranged by compound groups as represented by the sampling media used. For these tables, an "x" indicates that the compound was not detected in the sample. The less than (<) denotes that the compound was present but at levels below the quantifiable limits of the analytical method. The numerical value following the less than sign represents the quantifiable limit for the given sample volume. The "--" symbol indicates that the compound was not analyzed for.

A description of the sampling and analytical methods used can be found in the Enviro report titled <u>Industrial Hygiene Comprehensive Survey, Final</u> <u>Report, Cresap Test Facility, Cresap, West Virginia</u>.<sup>2</sup>

A-1

## TABLE A-1

# PNA Analytical Results in $\mu g/m^3$ (ppb) for Personal and Area Samples for the CSF Process Cresap, West Virginia, April and May 1979

									S	alid Sepa	iration (	300}/Carl	bontzatti	on (800)	Units		
		t n l unni	t Extrac	(200)						1	300 Area			300 Area			
	Ar	-54 	r	Operator		300	Area	800	Area	М	i i i wright		Pipe- fitter	Pipe- fitter	0	300/60Ni Iperators	
SAMPLE RUMBER	100	118	050	112	096	119	120	121	122	005	012	021	010	002	105	113	113
SAMPLE VOLUME(1)	2187	2257	862	654	620	2499	2487	2310	2539	622	810	311	376	323	538	553	587 0845-
SAMPLE TIME	0826- 2100	0900- 2256	0744- 1455	0328- 1450	0902 - 1455	0903- 2256	0907- 2256	0910- 2255	0915- 2254	0934- 1430	0918- 1603	0837- 1330	0905- 1521	0900- 1335	1541- 2235	0829- 1455	0845- 1515
Naphthalene	8	16.2	0.05	0.05	0.03	14.4	10.7	14.2	12.8	0.01	0.04	0.1	<0.01	×	9.02	0.04	00.04
Quinoline	0.4	1.4	<0.01	<0.01	<0.01	0.6	2.4	1.3	0.2	ж.	0.02	0.01	×	×	0,01	<0.01	<0.01
2-Hethy Insphthalene	42.6	x	0.05	0.1	0.09	28.8	0.09	42.5	21.9	0.03	0.08	0.2	0.02	0.01	0.06	0.1	0.1
1-MethyInaphthalene	10.3	21.2	0.05	0.07	0.05	27.0	0.02	13.2	10.04	0.01	0.03	0.1	<0.01	<0.01	0.03	0.06	0.05
Acenaphthalane	0.3	0.05	x	<0.01	<0.01	0.3	1.0	0,1	0.05	x	ж	я	x	×	<0.01	<0,01	*
Acenaphthene	0.8	1.9	<0.01	<0.01	<0.01	0.7	2.5	0.3	0.2	<0.31	<0.01	<0.01	×		<0.01	<0.01	<0.01
Fluorene	0.5	1.2	<0.01	<0.01	<0.01	0.4	1,6	0.2	0.1	<0.01	<0.01	<0.01	<0.01		<0.01	<0.01	<0.0
Phenanthrene Anthrecene	0.3	0.8	<b>&lt;0.01</b>	X	<0.01	0.3	1.2	0.1	0.00	×	<0.01	<0.01	<0.0I	×	<0.01	<b>∢0.01</b>	-0.0
Acridine	×	0.08	*	×	<b>&lt;0</b> .01	*	×	×	х	ж	ж	x	×	×	×	<0.91	×
Carbazole	x	0.03	я	x		0.02	~0. <b>0</b> 1	<0.01	<0.01	×	×	ж	x	×	×	×	×
Fluoranthene	x	0.01	x	×	x	0.03	0.07	<0.01	x	ж	×	×	•	ж	*	×	×
Pyrene	0.1	0.3	ж	x	<0.01	0.3	0.7	0.07	<0.01	×	×	×	1	×	<0.01	×	1
Benzo(a)fluorene	50.0	0.04	x	х	x	0.04	0.2	0.03	x	×	ж	x	ж	×	×	x	×
Benzo(b)fluorene	×		х	x	x	Χ.	×	X	x	×	×	×	х	×	¥		×
Benz(a)anthracene	×	ĸ	ж	x		x	×	0.02	0.01	×	×	X	×	X	х	×	*
Chrysene Triphenylene	×	×	×	x	x	x	0.02	•	X	×	×	×	×	×	×	X	×
Olmethylbenz(a)anthracene	x		x	х	×	×		×	x	×	*	×	×	×	×	×	×
Benzo(e)pyrene	x	*	x	x	×	X		x	×	×	×	×	×	x	*	x	×
Benzo(#)pyrene	× 1	x	x	×	×	*	x	x	×	×	×	×	x	*	۲	×	
Perylene	x	x	ĸ	x	x	×		×	x	×	х	*	ĸ	×	×	×	×
Dibenz(a,j)acridine	х	ж	×	x	x	×	×	×	×	×	*	×	×	×	*	ж	×
Dibenz(a,1)carbazole	ж	x	x	ж	x	×	- × -	×	×	ĸ	×	×		×	×	×	×
Indeno(1,2,3-cd)pyrene	к	×	x	ж		×	× 1	×	x	×	×	×	×	×	×	×	×
Dibenzanthracene		×	x	ж	×	×		X	×	×	х	x	×	×	×	×	
Benzo(g,h,i)perylene	x	×	×	×	*	* [	*	×	x	×	x	*	х	×	×	*	ľ
Anthanthrene	x	х	x		x I	x	• [	×	x	×	×	×	×	<b>. x</b>	*	×	
Coronene	×	×	х	x	x		×	×		×	×	х	×	×	×	x	[ *
Dibenzpyrene	ж	*	×	ж	x	×	- 1	×	×	x	х	×	ж	×	×	*	×
TOTAL	63.3	43.Z	0.2	0.2	0.2	72.9	20.5	72.0	45.4	0.05	0.2	0.1	0 02	0.01	0.1	0.1	0.3

x - Not detected

A-2

1		Salven	t Recover	ry (500)/	Fraction	Mition(70	(U)	Hydro	ogenation				
[					500/70	)				600/9	00		4045
		500 Area	700 Arei	•	Operato	or.		600 Ai	rea	Opera	itor		HNd
Sample Humber	097	123	124	098	074	094	131	078	099	016	077	133	021
Sample Volume(1)	2812	2 3 2 0	2382	2870	640	602	741	2790	2685	1120	571	631	595
Samp]e_1ime	0813-2320	0927- 2247	0934- 2248	0816- 2318	0735-	0804-	0717-	0755- 2325	0823- 2318	0735- 1508	+19413- 1522	0724- 1455	0921-
laphthal ene	22.9	11.09	7.4	19.6	0.02	0.08	0.05	11.5	16.8	-0.01	0.02	-0.01	0.03
luinotine	3.5	0.5	0.7	1.4	0.01	0.01	-0.01	0.2	2.4	-0.01	-0.01	-0.01	<0.01
-Nethylnaphthalene	35.6	42.4	19.01	12.2	0.06	0.1	x	21.02	0.08	0.02	0,06	0.08	0.05
-NethyInaphthalene	x	14.0	9.0	31.5	0.03	Ŭ.07	x	28.7	0.02	<0.01	0.03	0.2	0.02
cenaphthalene	1.0	0.1	0.06	0.2	x	+0.01	x	0.1	0.9	X	+0.01	-0.01	×
Icenaphthene	2.02	0.4	0.2	0.6	<0.01	<0.01	-0.01	0.4	2.5	<0.01	<0.01	+0.01	<0.01
luorene	1.03	0.2	0.3	0,4	/0.01	<0.01	-0.01	0.2	1.7	<0.01	<0.01	-0.01	-0.01
Phenanthrene Anthracene	0.6	0.2	0.2	0.4	<0.01	<0.01	<0.01	0.2	1.0	×	<0.01 	+0.01	-0.01
Acridine	x	x	×	0.6	X	X	X	X	0.04	×	-0.01	×	X
Carbazole	0.01	<0.01	0.2	×	X	x	<0.01	x	X	x	×	x	X
Fluoranthene	0.07	1	0.03	0.09	X	x	-0.01	<0.0I	0.2	x	x	+0.01	X
Pyrene	0.5	0.04	0.Z	0.6	<0.01	<0.01	<0.01	0.06	0.9	X	<0.01	- 0 <b>01</b>	X
Benzolalfluorene	0.02	x	0.03	0.2	x	x	+0.01	X	0.3	x	x	x	×
Benzo(b)fluorene			1	1		1				1		1	1
Benzo(a)anthracene	x	x	X	0.05	x	x	X	0.04	x	X	X	x	X
Chrysene Triphnylene	X .	x	x	×	×	×	x	x	0.06	×	×	x	X
Dimethylbenz(a)anthracene	x	X	×	X	X	X	<0.01	. X	<0.01	X	X	X	X
Renzole)pyrene	x	X	X	X	×	x	X	X	<0.01	X	×	X	×
Benzo(a)pyrene	x	X	X	X	×	X	X	X	<0.01	X	X	X	X
Perylene	x	X	X	×	X	X	×	X	×	X	X	X	X
Oibenz(a,j)acridine	х	X	X	X	×	X	X	X	X	X	×.	X	X
Dibenz(a,i)carbazole	x	x	x	x	X	×	X	*	X	X	X	X	X
Indeno(1,2,3-cd)pyrene	х	[ x ]	x	X	X	X	×	X	X	X	×	x	X
Dibenzanthracene	x	x	X.	x	X	X	X	X	X	X	X	X	×
Benzo(g,h,i)perylene	x	x	X	x	×	X	X	X	×	X	X	×	X
Anthanthrene	x	x	x	x	×	×	x	X	X	X	I X	×	X
Cornnene	x	x	X	×	X	X	X	X	X	X	X	×	X
Dibenzpyrene	x	x	X	x	X	X	X	X	x	×	X	X	X

# TABLE A-1 (Continued)

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\*Millwright

x = Not detected

TABLE A-1 (Continued)

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	J		Laborato				Sh	1A				_	
	1	Chewis		Lab	lechnic	an	Super	visor			Blani	ده <sup>د</sup> 	<b>4</b>
Sample Number	008	052	116	009	019	051	095	106	036	039	046	089	094
Sample Volume(1)	621	494	603	255	533	587	627	769					
Sample Time	0806- 1500	0806- 1535	0830- 1541	0755- 1500	0811- 1501	0801- 1500	0756- 1454	1534- 2241					
Naphthalene	<0.01	0,05	<0.01	0.1	-0.01	0.03	0.04	0.01	x	x	x	X	X
Quinoline	x	<0.01	<0.01	X	<0.0i	<0.01	<0.01	<0.01	×	x	X	x	X
2-Methylnaphthalene	0.02	0.06	0,01	0.1	0.02	0.04	0.09	0.05	X	x	. <b>X</b>	X	X
1-Nethy Inaphtha Tene	-0.01	0.04	<0.01	0.08	-0.01	0.02	0.04	0.02	I X	369	133	X	1177
Acenaphthalene	X	X	X	X	X	X	<0.01	<0.01	x	x	×	X	X
Acenaphthene	<0.01	<0.01	<0.01	<0.01	X	<0.01	<0.01	<0.01	x	X	X	X	X
fluorene	+0.01	<0.01	<0.01	×0.01	X	<0.01	-0.01	<0.01	x	x	X	X	X
Phenanthrene Anthracene	X	<0.01 X	××	<0.01 X	XX	<0.01 X	<0.0) X	<0.01 X	X	××	××	×	XX
Acridine	x	x	· x	x	x	x	X	x	x	x	X	x	X
Carbazole	x	x	x	x	x	x	x	x	x	x	X	X	x
Fluoranthene	x	x	×	x	x	x	x	x	17	x	X	X	X
Pyrene	x	x	x	l x	x	x	x	x	22	x	x	x	x
Benzo(a)fluorene	X	X	X	X	X	X	X	X	X	x	X	×	X
Benzo(b)fluorene	Х	X	X	X	X	X	X	X	2	x	x	X	X
Benzo(a)anthracene	X	X	X	X	X	X	X	X	X	x	x	×	X
Chrysene Triphenylene	x	×	×	×	X	×	X	×	×	x	×	X	×
Dimethylbenz(a)anthracen	e X	х	x	X	X	x	X	X	x	x	X	x	X
Benzo(e)pyrene	×	X	X	X	X	x	X	X	x	×	X	X	X
Benzo(a)pyrene	X	X	X	×	x	x	X	×	×	x	X	x	X
Perylene	X	x	x	X	×	x	×	X	x	X	X	X	×
Dibenz(a,j)acridine	×	x	×	x	X	×	x	X	x	x	x	X	X
Dibenz(a,t)carbazole	X	x	×	x	×	X	X	X	x	x	x	X	X
Indeno(1,2,3-cd)pyrene	X	x	. X	×	x	×	x	×	x	x	x	x	x
Dibenzantbracene	X	x	×	X	×	×	×	x	×	x	x	x	x
Benzo(g,h,1)perylene	x	X	x	x	×	[ X	X	x	x	X	×	X	X
Anthanthrene	×	x	×	x	x	X	x	x	x	x	X	X	X
Coronene	X	x	X	X	x	X	×	X	x	x	X	X	X
Dibenzpyrene	X	x	x	×	X	×	×	×	x	x	x	x	×
lotal	0.02	0.2	0.01	0.3	50.0	0.09	0.2	0.08					

x = Not detected

<sup>C</sup>In nanograms per samp}e

## TABLE A-2

# Analytical Results (In PPM) for Silica Gel Samples for the CSF Process Cresap, West Virginia, April and May 1979

			Solveni	Extract	ion (200	))		Solid Separation (300)/Carbonization (800)									
	Area	Insi	lator	Hill- wright		Operator		300	Area	800	Area	Piper	jiter		Operator		
SAMPLE HEMBER	135	021	072	026	027	073	134	054	100	053	099	024	025	045	.046	057	
SAMPLE VOLUME(1)	43	40 .	37	48	42	91	44	92	79	46	. 11 .	. 75	- 62	79	. 89 _	61	
SAMPLE TIME	0730- 1500	0903- 1530	0910- 1530	0937- 1535	1622- 2303	0733- 1458	0733-	0819- 1603	0830- 1530	0821- 1601	0834- 1530	0915 1543	11916- 1543	0742-	0743- 1502	1528- 2357	
Antline		x			×	<0.05		<0.05	< 9.05	×		<0.05	×	<0.05	<0.05	×	
N,N-Dimethylaniline		x		x	0.1	<0.05	×	<0.05	<0.05	*	•	×0.05	×	<0.05	<11.05	×	
2,4-Dimethylaniline	1	×	×	x	,	<0.05	*	<0.05	<0.05		×	<b>*0.0</b> 5	×	-0.05	<0,05	1	
p-Nitroaniline	×	[ x ]		x	×	<0.05	x	<0.05	<0.05	x	×	<1).05		<0.05	<0.05	×	
- o-Toluidine		x	] .	x	L x	<0.05	х	<0.05	<0.05		×	<0.05	×	<0.05	+0.05	×	
o-Anisidine	x	x	x I			×	x	×	X	x	x	x	x	x	×	×	
p-Anisidine		×		x	×	x		×	я		x	×	1	×	×	×	
Phenol				*					••	••	x	••					
o-Ethylphenol				x				••	•-							•	
p-Ethylphenol				x				•-									
o-Cresol				x			••			•**	× 1				•.		
m-Cresol				L L						•	×						
p-Cresol				×	· · ·						×						
2.3-Xylenol				×											•-		
2,5-Xylenol				×							×					•-	
3,5-Xylenol		l									[ × ]						

x \* Not detected

-- = Hot analyzed

# TABLE A-2 (Continued)

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I.

	Solve	ent Reco	very (SO	)/Fracti	onation	(700)	Hydri	igenation	(600)/0	tilities	(900)		Tank Farm (1190)/Environmental (1200)						
	500	Area	700 Area		Operator		600	Area		600/900	Operator		1100 Area	1200 Area		100/1200	) Operato	)r	
SAMPLE NUMBER	108	117	055	103	114	138	014	082	006	044	101	109	015	056	031	034	047	104	
SAMPLE VOLUME(1)	47	60	44	84	38	75	45	45	88	82	91	57	44	46	48	40	45	41	
SAMPLE TIME	1600- 2319	0920- 1545	0825- 1604	1542- 2248	0817- 1448	1610- 2258	0853- 1620	1538- 2326	0740- 1506	0740- 1530	1523- 2300	1603- 2318	0900- 1626	0829- 1630	1620- 2306	1625-	0750- 1448	1547 2252	
Antline				<0.05	×	×	x	x	<0.05	<0.05	<0.05			x			×	Ι,	
1,H-Dimethylanlline	×	1 ×	x	<0.05	x	x	x		<0.05	<0.05	<0.05			x	×		I K	1,	
2,4-Dimethylaniline			×	<0.05	x	×	x	×	<0.05	<0.05	0.05		x				0.01	×	
p-Nitroaniiine	*	<b>.</b>	x	<0.05			×	× 1	<0.05	<0.05	<0.05			×	×		x	×	
o-Taluidine	*	×	x	<0.05				×	<0.05	<0.05	<0.05			×			×	,	
o-Anisidine	1 x		x		×	×	×	×					× 1				×	1 .	
Pheno I								1				×		×		×			
o-Ethylphenol												×		×		1			
p-Ethylphenol		[				I								×		× 1		-	
o-Cresol												×	[			×		1 -	
n-Eresol												x	·	×				-	
p-Cr <b>esol</b>												,		×		ĸ			
2,J-Xylenot												x		×		×	•-		
2,5-Xytenol												×		x				l .	
),5-Xylenol	••		1				1			I	1	1.	l	1.				- I	

x = Not detected

-- > Not analyzed

TABLE A-2 (Continued)

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				aborato	ry			<u> </u>	iuperviso	H	I				
	1 a	b Technic	lan		1	Chemis	t	ot	Sh	ift			Blai	14 5	
SAMPLE NUMBER	017	018	049	062	075	098 .	136	011	140	061	040	087	144	145	1 146
SAMPLE VOLUMETT)	83	80		- 76 -	- 82 -	- ()	- 42 -	88	1 11	84					1
SAMPLE TIME	0800- 1500	0809- 1501	0804- 1504	1546- 2300	0810- 1559	0847- 1547	0900- 1543	0925- 1510	0750- 1555	1554- 2359					
latt ine	«0.05	<0.05	×		x	×		×	x	<0.05	×	,	x	×	
N,N-Dimethylanfline	<0.05	<0.05	x		×	x		×	×	<0.05	×	×	×	×	
2,4-Olmethylaniline	<0.05	<0.05	×		×	×	×	×	0.06	0.06	×	×	×		×
p-Hitroan/line	<0.05	<0.05	×			ж	x I	×	x	<0.05		× 1		•	1 *
o-Tatuldine	<0.05	<0.05	ĸ		×	x	×	×	×	<0.05	x	*		×	1
o-Anisidine	R	x	×		x	x	×	×	×	x	×	×	×	•	
Phenol										×				* I	1
o-Ethylphenol				×	•••					×	*		· · ·	× 1	
p-Ethylphenol				x	**	•				×	t x		**	×	1
o-Cresol				×						x	x	*-		×	
n-Cresol				x						×	- x			×	
p-Cresol				*		•••				×	×			×	
2,3-Kylenol				×							×			1	
2,5-Kylenol				×	••	••				x	×			x	
3,5-Iylenol				x					1	x	x			x	

x = Not detected

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-- - Not analyzed

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# TABLE A-3

# Analytical Results (in PPM) for Charcoal Tube Samples for the CSF Process Cresap, West Virginia April and May, 1979

						Compound	
Sample Number	Sample Volume(L)	Sample Time	Unit Operation	Sample Type	Benzene	Toluene	Xylene
013	42	0852- 1620	Hydrogenation	Area	<0.04	0.06	<0.01
084	47	1547- 2327	Fractionation	Area	<0.04	0.03	<0.01
064	57	1550- 2300	Laboratory	Personal Lab Tech	<0.03	0.02	<0.01
076	85	0740- 1448	Laboratory	Personal Lab Tech	<0.02	0.08	<0.01
077	46	0742- 1447	Laboratory	Personal Lab Tech	<0.04	0.03	<0.01
041		1		Blank	x	x	×
088				Blank	×	x	×

x = Not detected

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