CT083 119-20a

FINAL

CONTROL TECHNOLOGY ASSESSMENT
FOR COAL GASIFICATION AND LIQUEFACTION PROCESSES

Solvent Refined Lignite Process Development Unit
University of North Dakota
Grand Forks, North Dakota

Report for the Site Visit of
March 5, 1980

Contract No. 210-78-0084

March 1982

Submitted to:

Phillip A. Froehlich, Project Officer
National Insitute for Occupational
Safety and Health
Division of Physical Sciences and Engineering
Robert A. Taft Laboratories
4676 Columbia Parkway
Cincinnati, Ohio 45226

Submitted by:

Donato R. Telesca, Program Manager
Dynamac Corporation
Enviro Control Division
11140 Rockville Pike
Rockville, Maryland 20852

FOREWORD

On March 5, 1980, the Enviro Control, Inc. (ECI) control technology assessment (CTA) team met with Mr. A. Max Souby to discuss Project Lignite and to conduct a site survey of the Solvent Refined Lignite Process Development Unit (SRL PDU) at the University of North Dakota at Grand Forks. The site visit was adjunct to the site visit of the Slagging Fixed-Bed Gasification Pilot Plant and Liquefaction Facility at the Grand Forks Energy Technology Center at Grand Forks. Although the SRL PDU had been shut down for approximately one and one half years at the time of the visit, the ECI team made a site inspection of the facilities to familiarize themselves with the operating and occupational health and safety equipment.

The ECI CTA team included:

James Evans	Project Manager
Donato Telesca	Principal Investigator
Russell Tanita	Industrial Hygienist

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

I. INTRODUCTION

A. Contract Background

The objective of the "Control Technology Assessment for Coal Gasification and Liquefaction Processes" program is to study the control technology that is available to prevent occupational exposure to hazardous agents in coal conversion plants. Although the Solvent Refined Lignite Process Development Unit (SRL PDU) has not been operating for approximately 1-1/2 years, the CTA team observed the SRL PDU and discussed the program in order to learn what occupational and safety controls had been used to reduce worker exposure to SRL process products.

B. History of the Solvent Refined Lignite Project

The project designated "Project Lignite" was established in 1972 in the Department of Chemical Engineering at the University of North Dakota, Grand Forks for the purpose of determining the technological parameters for the conversion of Northern Great Plains Lignite to premium solid, liquid and gaseous fuels. The ultimate goal of the work was to develop data and expertise so that recommendations could be made for a refinery based on lignite as feedstock.

In keeping with the objective, laboratory and bench scale work were carried out and a continuous process development unit (PDU) was constructed and operated to demonstrate the feasibility of the continuous liquefaction of lignite.

Project Lignite was originally undertaken in a contract with the Office of Coal Research later changed to ERDA for the period of March 28, 1972 through March 27, 1977. A supplemental agreement under the ERDA contract, was made to provide for the continuation of Project Lignite for the period June 16, 1977 through June 15, 1978. The PDU has been down since June 1978.

C. Process Description

The Process Development Unit (PDU), except for the solid-liquid separation, was completed in early 1975. The continuous unit has a nominal design capacity of 50 lb/hr of lignite feed to produce approximately 15 lb/hr of solvent refined lignite (SRL).

Lignite with full "as-received" moisture content is pulverized and slurried with solvent. The slurry is pressurized, preheated and reacted at selected process conditions of temperature and pressure in a reducing atmosphere. Liquefaction products are separated as gases, liquids and SRL from the uncoverted lignite and mineral matter. A block diagram is presented in Figure 1, while a more detailed description of the process follows.

The feed lignite is brought from the mine by rail or truck. The lignite is initially ground in a jaw crusher, tramp iron is magnetically removed and the lignite is fed through storage bins to a pulverizer. Pulverization reduces the lignite to 100 percent minus 60 mesh and 90 percent minus 200 mesh. The pulverized lignite is collected in 55 gallon drums.

The pulverized lignite is charged through an Acrison volumetric feeder into a Marion Mixer at a constant feed rate. Weigh cells are used to measure and control the weight rate of feeding. The solvent is fed through a flow-controller-recorder to give the desired lignite-solvent ratio. Discharge from the mixer is controlled by the slurry level in the mixer tank. A pump circulates the slurry around a mixer loop from which the slurry is fed to the suction side of the high pressure pumps at 50 psi.

The pressurized lignite-solvent slurry is combined with a gas composed of cleaned recycle gaseous reactants combined with make-up hydrogen and carbon monoxide. The slurry mixture is heated to approximately 750 F (400 C) and fed into the bottom of the dissolver-reactor. Outlets along the length of the dissolver allow a four-

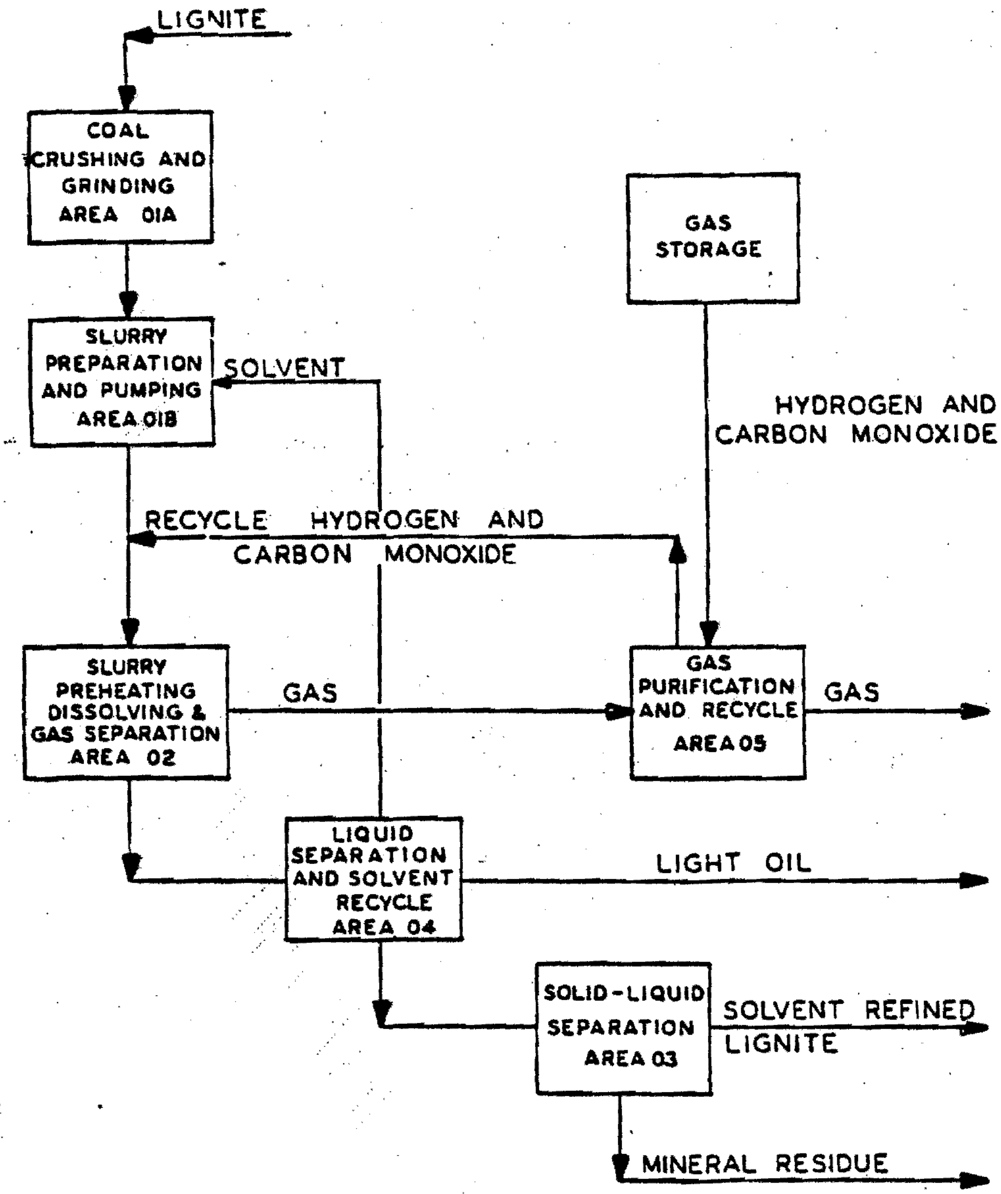

FIGURE 1 - Simplified Block - Flow Diagram[a] of the Solvent Refined Lignite Process Development Unit (SRL PDU), University of North Dakota, Grand Forks

[a]Adapted from Reference 2

fold variation in the residence time of the slurry without a change in feed rate. The mixture is cooled to about 500 F (260 C) and fed to the high pressure separator. The gas from the separator contains unreacted carbon monoxide and hydrogen, carbon dioxide, hydrogen sulfide and light hydrocarbons. After a pressure control valve, the gas enters the gas recovery and recompression area where it is cleaned and recycled.

The slurry products from the high pressure separator pass through a pressure release system to an intermediate pressure separator. Light hydrocarbons and solvent are vaporized, cooled, and combined with liquids from the recycle condensate separator to a low pressure flash separator. The organics are fed to a light-ends-column which separates the light oil from the recycle liquefaction solvent. The light oil is collected, essentially as a stabilized solvent, and the recycle solvent is circulated to the slurry mix tank.

A second captive volume pressure letdown system discharges the slurry from the intermediate pressure flash separator to the partial flash separator. The overhead is collected and fed to the light-ends-column. The bottoms are sent to the solid-liquids separation system.

The separation system is designed to extract solvent refined lignite and solvent from mineral matter and unconverted lignite using toluene as the deashing solvent. The flow diagram is shown in Figure 2.

The bottoms from the vacuum flash are mixed with the slurry and toluene in an in-line mixer. The slurry is heated to 350-400 F (177-204 C) and fed to the solids precipitation tower. The design is such that the terminal velocity of the settling particles is greater than the velocity (controlled) of the upward moving toluene-SRL solution. A second stream of toluene entering the base of the tower dissolves any SRL adhering to ash or uncoverted lignite.

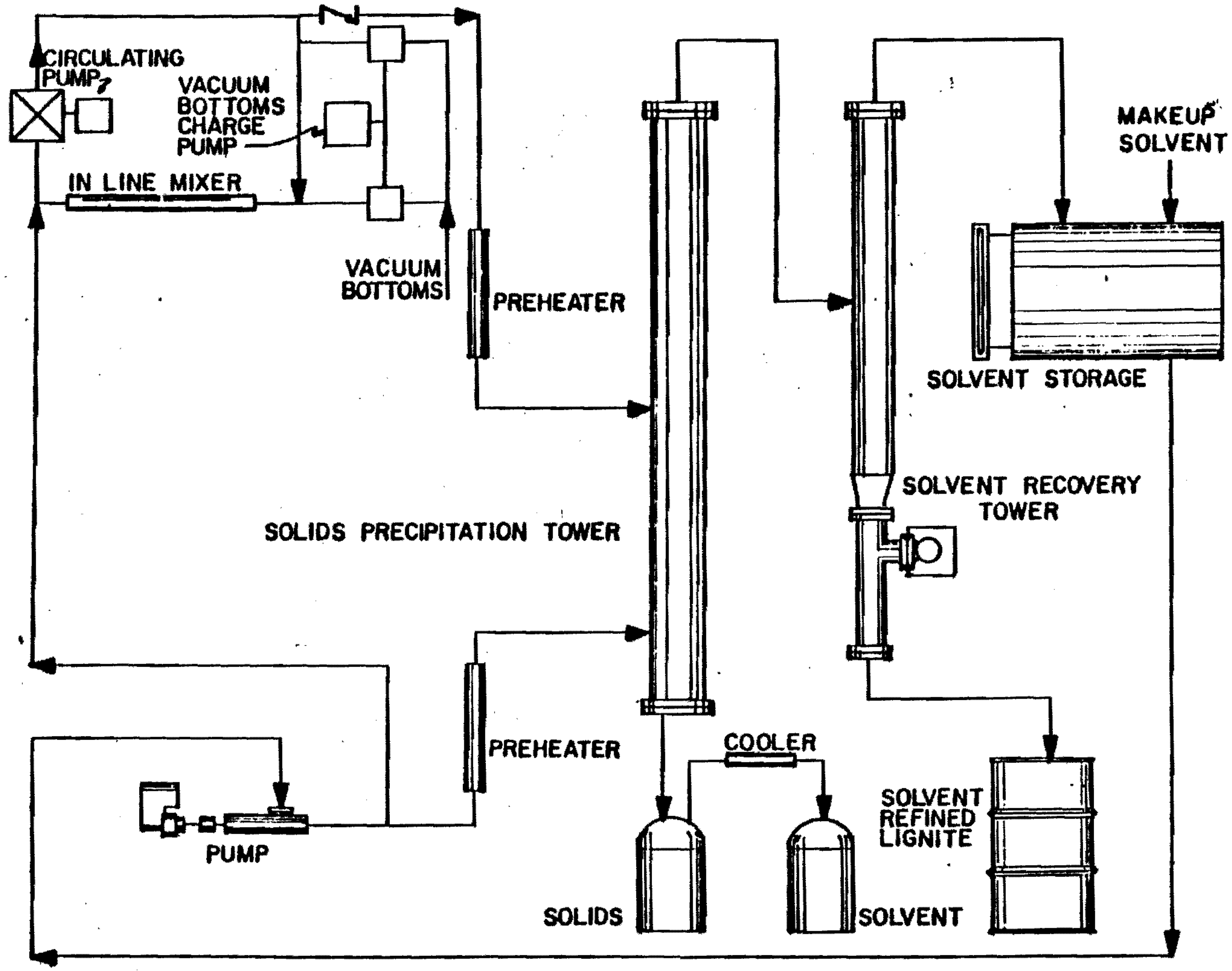

FIGURE 2 - Diagram of the Solid-liquid Separation Unit[a]
50 lb/hr Solvent Refined Coal Process Development Unit
(SRL PDU), University of North Dakota, Grand Forks

[a]Adapted from Reference 1

The solids are collected through a modified lockhopper. The toluene is flashed off the solids and collected.

The liquid overhead from the solids precipitation tower, consisting of dissolved SRL, liquefaction solvent and toluene is depressurized, cooled and fed to a toluene recovery tower (not shown). The toluene flashes, leaving the SRL-solvent mixture behind, which is fed through a preheater to the solvent recovery tower. Here the solvent is flashed for collection in the solvent storage tank, and the SRL is collected in drums.

The melting point of the crude SRL product before deashing is usually in the temperature range of 300 to 400 F (149 to 204 C). Deashed SRL after solvent recovery has lower melting points, sometimes as low as 200 F (93 C). Additionally, smaller quantities of lighter liquids and gases are produced.(1)

D. Potential Hazards

The SRL PDU had not been in operation since June, 1978. Identification of potential hazards was based on information obtained at a meeting with the former manager of the facility and from observation of the facility. These potential hazards are presented by unit operation in Table 1.

II. ENGINEERING CONTROL TECHNOLOGY

The principle concern at the SRL PDU was safety oriented and involved the fire and explosive potential of the high pressure, high temperature, combustible material handled in the SRL process.

General dilution ventilation was installed to minimize the accumulation of combustible vapors/gases. The system has the added benefit of controlling inhalation exposures to polynuclear aromatic hydrocarbon (PNA) material handled in the ~~in the~~ process.

Table 1

Potential Hazard by Unit Operation
Solvent Refined Lignite

Process	Potential Hazard
Coal Crushing and Pulverizing	Coal Dust Noise
Slurry Preparation	Coal Dust Polynuclear Aromatics Noise Toluene Fire Explosion
Preheating and Dissolving	Polynuclear Aromatics Aromatic Amines Carbon Monoxide Toluene Fire Explosion
Cleanup system	Polynuclear Aromatics Aromatic Amines Toluene Fire Explosion

During its operation, University of North Dakota (UND) personnel noted stress corrosion cracking in the preheater coil and the dissolver-reactor, which was made from 316 stainless steel. The 316 stainless steel coil was originally installed because the Wilsonville, Alabama, Solvent Refined Coal (SRC) Pilot Plant (operated by Southern Company Services, Inc.) was using 316 stainless steel; furthermore, the preferred Incoloy 800 did not arrive on schedule. The installation of Incoloy 800 eliminated this stress corrosion problem. The opinion of UND personnel is that the failure was due to chloride stress corrosion; but that the chlorides are not from the coal. The facility had been using trichloroethane (TCE) for cleaning and the TCE may have been the source of chlorides. Use of the chlorinated solvent for cleaning was discontinued.[1]

The plant experienced erosion of small thermocouple wells from the slurry. This problem has also occurred at other plants where thermocouple wells are in slurry or slagging ash streams. To date, the problem has not been resolved.

The SRL PDU had experienced acid corrosion downstream from the reactor at the dew points of the process stream. There was pinpoint corrosion in the 304 SS, which is probably due to sulfur. The corroded parts were replaced with 316 SS and since the change, the PDU experienced no further problems.

Hydrogenation experiments were run at the SRL PDU and some definite difference between SRC and SRL products were observed. For example, the SRL without ash melts around 200 F (93 C) compared to SRC which melts around 300 F (149 C). In addition, chemical characteristics determined for SRL and SRC indicated definite differences in chemical composition. Conclusions concerning an average molecule of SRL are: the average molecule contains several aromatic rings, some condensed; an average of 0.8 acidic groups; mainly as phenols; and an average of 0.4 basic nitrogen atoms. Fixed in the non-acidic or non-basic structures are 1.8 atoms of oxygen and 0.37 atom of nitrogen per average molecule as determined by elemental analyses and titration data.[1].

At the SRL PDU, most of the leaks were attributed to erosion. Tungsten carbide is used as trim for the valves. In addition, the SRL PDU did not use valves for throttling; instead they used a system of sequential opening and closing with the proper volume between valves. This arrangement resulted in uncontrolled pressure surges through the system which hampered steady-state operation of the PDU.

Hills-McCanna reciprocating pumps with ceramic ball checks were successfully used to move the slurry. Circulating pumps with closed impellers were unsatisfactory because of slugging. When the pumps were changed to Lobe pumps,[2] the plant had no problems. Moyno pumps gave poor results.

In attempts to reduce corrosion, linings were tried; Kel Var (a Du Pont elastomer) worked well, but Viton[2] failed. The Viton swells, softens, and extrudes on the benzene side of the operation.

The PDU experienced difficulty in removing vacuum bottoms. The equipment consisted of two valves on the discharge line with means for evacuating the volume between the valves. The valves were closed and the volume between the valves was evacuated. The top valve was opened and the space filled. The top valve was closed, the bottom valve opened, and the bottoms dumped. The bottom valve was closed and the procedure repeated. The problem may be due to bridging in the section between the valves.

One of the improvements made at the PDU was the installation of a throttling valve in the process line between the vacuum-bottoms charge pump and the solids-precipitation-tower preheater.

The packing used in the plant equipment seemed to work satisfactorily. It was John Crane No. 177 GF 1/2" square packing (braided asbestos yarn reinforced with inconel wire). There were two seals on the pump, with 3 rings of packing per seal.

Accumulation of solids in the SRL reactors was a major unresolved problem in maintaining long-term continuous operation of the PDU. This accumulation of solids resulted in a variety of operational problems which resulted in reactor and downstream plugging as well as mechanical failure of pumps and control equipment.[1,2] Of importance to worker health and safety would be the increased amount of maintenance and housekeeping required to continually clean process areas, lines, and equipment plugged with the high melting point material and the subsequent handling of off-spec material and solvent.

It was found that simple flushing of the reactors with solvent left solids in the reactor that coked in subsequent operations. This coked material had to be manually drilled out. It was found that if, immediately following coal-feed termination, reactor shutdown involved circulation of solvent during reactor cooling followed by a violent blow-down of the reactor into receivers, very little, if any, residue would remain in the reactor.[2] Redesign of the reactor or process modifications to prevent the solids accumulation appears to be the only effective method of reducing maintenance-related exposures.

Attempts to dry the lignite down to 5% in a rotating drum dryer ended with an unsatisfactory feed, unless hydrogen was used in the drying drum. Fire and explosive hazards associated with the use of hydrogen in this operation should be evaluated.[3]

The PDU used a blow-out wall to protect against explosion. A heavy steel mat was hung outside this blow-out wall to reduce the area which would be affected by the explosion and debris.

The following are some differences in handling lignite versus bituminous coal: dry lignite is not as reactive with hydrogen; lignite is more difficult to grind; and lignite has a high angle of repose. These factors will increase the potential hazards because more hydrogen will be handled and more maintenance will be required at the grinders and storage lines. Lignite is low in sulfur (0.3 to 0.5%) with all sulfur being organic. This will result in the formation of less hydrogen sulfide (H_2S) thereby reducing worker exposure to H_2S.

III. OTHER CONTROL TECHNOLOGY

The ECI survey team was unable to identify any information sources that could provide a description of the health and safety program at the Solvent Refined Lignite Process Demonstration Unit. However, information obtained at a meeting with the former manager of the facility leads us to believe that the facility did not have a comprehensive health program. Concern appeared to be safety oriented particularly with regards to the potential fire and explosion hazards associated with the process.

Some general inferences made on the basis of the information obtained at the meeting and from observations of the facility are included here. Work practices, which include the required use of safety hats and safety shoes, were also safety-oriented. The presence of a ventilation system did have the potential added benefit of controlling vapor buildup in the facility.

The ECI team observed three carbon monoxide monitors located at the operating panel which took samples at different locations in the PDU. There were no hydrogen sulfide (H_2S) monitors.

Coal tar residue was noticed throughout the process area. Because workers were not required to shower, skin contact may have been a major PNA exposure problem.

IV. CONCLUSIONS AND RECOMMENDATIONS

- The use of blow-out wall or panels should be included in the design of any completely enclosed facility housing a liquefaction unit. The use of heavy metal mats such as those used at the SRL PDU, or a blast directing hill, also should be included in such a design.

- Greater emphasis should be placed on housekeeping. It should be the responsibility of the individual operators and/or the assigned maintenance personnel to keep the various areas clean. It is recognized that this is an R&D plant, with constant changes being made in the equipment in order to run tests; as a result spills will probably be more frequent than in a normally operating demonstration or commercial plant.

- The ECI team recognizes that the SRL PDU was installed in an existing building and that existing vessel spacing was specified to reduce line heat losses and chances of line plugging while providing space around vessels for maintenance work. However, future installations and/or design should provide more space around equipment which requires frequent maintenance. When accessibility for maintenance around equipment, especially pumps, is limited, maintenance personnel often work for longer periods of time and thereby increase their exposure for each job. Also, new plants should have sufficient provisions for cleaning pump areas to prevent process material from going to the waste treatment plants. Working space around the pumps should be sufficient to facilitate maintenance.

- Future programs should be designed to include industrial hygiene/occupational health programs in addition to only safety oriented programs. Included in such a program should be an established respiratory protection program. There are a variety of toxic substances found in coal conversion unit processes. During the normal operations, such as housekeeping, preparation of vessels for maintenance, etc. workers may be exposed to the various hazardous substances by inhalation and dermal exposure. Skin exposure may be controlled by appropriate equipment such as gloves, coveralls, shields, and special work clothing. However, there are no adequate recommendations for providing worker respiratory protection. NIOSH suggests that respirators be provided for three functions. First, escape type respirators should be conveniently stationed around the coal conversion facility for workers to wear during emergencies or process upsets. Second, half-mask respirators with disposable particulate filters/cartridges should be used by workers in the coal preparation area. Finally, and most importantly workers involved in cleaning and maintaining process vessels should wear a combination self-contained breathing apparatus and air-supplied respirator with full-face piece, operated in pressure demand or other positive pressure mode.

- A central control area for thermostat, junction boxes, etc. is recommended in the design of demonstration and commercial plants, so that the thermostat controls for the heat tracing of the pipelines would not be in areas where fouling occurs and in which excessive maintenance is required.

- In measuring tank levels, it suggested that the indicators, both liquid and direct-reading, be purged. Liquid and direct-reading indicators give better results than any other equipment.

- There should be an established procedure for handing down changes in procedure from shift to shift.

- In commercial plants, there should be catch basins with cleanouts in all areas in order to facilitate cleaning and to avoid plugging of drains. This will allow the water to go to the waste treatment plant and prevent solids from getting past the catch basin into the drain system. Periodically, special plant crews should clean the various spills which may occur.

REFERENCES

1 Severson, D.E., Project Lignite, Final Report, March 28, 1972 to June 15, 1977, U.S. Department of Energy, Fossil Energy, FE-1224-80.

2 Severson, D.E., Solvent Refined Lignite Process Development, Final Technical Report, June 16, 1977 to June 15, 1978, January 5, 1979, U.S. Department of Energy, Fossil Energy, FE-4189-15.

3 Hord, J., Is Hydrogen Safe?, NBS Technical Note 690 October 1976. National Bureau of Standards.

REFERENCES

1 Severson, D.E., Project Lignite, Final Report, March 28, 1972 to June 15, 1977, U.S. Department of Energy, Fossil Energy, FE-1224-80.

2 Severson, D.E., Solvent Refined Lignite Process Development, Final Technical Report, June 16, 1977 to June 15, 1978, January 5, 1979, U.S. Department of Energy, Fossil Energy, FE-4189-15.

3 Hord, J., Is Hydrogen Safe?, NBS Technical Note 690 October 1976. National Bureau of Standards.

REFERENCES

1 Severson, D.E., Project Lignite, Final Report, March 28, 1972 to June 15, 1977, U.S. Department of Energy, Fossil Energy, FE-1224-80.

2 Severson, D.E., Solvent Refined Lignite Process Development, Final Technical Report, June 16, 1977 to June 15, 1978, January 5, 1979, U.S. Department of Energy, Fossil Energy, FE-4189-15.

3 Hord, J., Is Hydrogen Safe?, NBS Technical Note 690 October 1976. National Bureau of Standards.