

January 1981



# Research and Development

ENVIRONMENTAL ASSESSMENT:  
SOURCE TEST AND EVALUATION REPORT  
KOPPERS-TOTZEK PROCESS

## Prepared for

EFFLUENT GUIDELINES DIVISION  
OFFICE OF SOLID WASTE  
OFFICE OF AIR QUALITY PLANNING AND  
STANDARDS

## Prepared by

Industrial Environmental Research  
Laboratory  
Research Triangle Park NC 27711

## DISCLAIMER

This report has been reviewed by the Industrial Environmental Research Laboratory (RTP), U.S. Environmental Protection Agency, and approved for publication. Approval does not signify that the contents necessarily reflect the views and policies of the Agency, nor does mention of trade names or commercial products constitute endorsement or recommendation for use.



U.S. ENVIRONMENTAL PROTECTION AGENCY

Research Triangle Park, North Carolina 27711

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3/11/81

The attached report presents data that were obtained from data acquisition performed at the AECI facility at Modderfontein in the Republic of South Africa. This facility utilizes Koppers-Totzek gasifiers which have potential use in this country for indirect coal liquefaction and other synthetic fuels from coal systems.

W. J. Rhodes

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United States  
Environmental Protection  
Agency

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ENVIRONMENTAL ASSESSMENT:  
SOURCE TEST AND EVALUATION  
REPORT KOPPERS-TOTZEK PROCESS

January, 1981

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## ABSTRACT

A source test program was conducted at a Koppers-Totzek (K-T) coal gasification facility operated by AECI Limited at Modderfontein, Republic of South Africa. The EPA's interest in the K-T process stems from the fact that the process economics and demonstrated commercial reliability make it a viable prospect for U.S. applications. The responsibilities for sampling, analysis, and engineering descriptions of the Modderfontein plant were shared between TRW and Krupp-Koppers GmbH of Essen, Federal Republic of Germany. EPA's phased approach for environmental assessments was followed. Level 1 and Level 2 data were collected along with priority pollutant screening data. Much of the effort was focused on wastewater streams. The wastewater treatment, consisting of a clarifier and settling pond, was adequate to produce a final discharge that had lower pollutant levels than the fresh input waters supplied to the plant. The complete data are presented in this report along with descriptions of the K-T process and the Modderfontein plant. The purpose of the Source Test Evaluation (STE) was intended as an initial effort and was somewhat limited in scope. Thus recommendations for future STE programs are also provided.

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## 1. INTRODUCTION AND SUMMARY

TRW, under contract EPA 68-02-2635 to the Environmental Protection Agency (EPA), is performing a comprehensive environmental assessment of high-Btu gasification and indirect liquefaction technologies. A major portion of this environmental assessment project is to obtain data on operating facilities through Source Test and Evaluation (STE) programs. The objective of each STE program is to obtain the data necessary to:

- 1) evaluate environmental (ecological and health) effects of waste streams or streams that may potentially be discharged in plants designed for U.S. sites, and
- 2) allow subsequent evaluation of the equipment available or required for controlling these streams.

An STE program was conducted by TRW on a Koppers-Totzek (K-T) coal gasifier. The EPA's interest in the K-T process stems from two principal factors: first, in the national drive to supplement liquid and gaseous fossil fuels through coal conversion, process economics dictate that the more viable conversion products will be those having the highest unit retail value. The K-T process represents one of the prime candidates for converting raw coal into the intermediate synthesis gas needed to produce these high-value products. Secondly, the K-T process has a lengthy history of successful application to a variety of foreign coals and promises to be equally adaptable over the range of American coals. This factor is particularly important in view of the contrasting lack of demonstrated commercial reliability on the part of the developmental U.S. gasifiers, and is viewed in a very positive light by both conversion project financiers and program managers.

The K-T process operates on an entrained bed principle. It utilizes a high temperature ( $1400^{\circ} - 1600^{\circ}\text{C}$ ), atmospheric pressure reaction fueled by a continuous co-current input stream of coal, oxygen and steam. The licensor-developer of the Koppers-Totzek gasification process is Krupp-Koppers GmbH (K-K) of Essen, Federal Republic of Germany. As of 1978, there were 54 K-T gasification modules operating in the world of which 47 were

using coal as a feed stock. All of the K-T gasifiers in operation as of 1978 were used to make synthesis gas as an input stream for the production of ammonia. The facility selected for testing was the Number 4 Ammonia Plant at Modderfontein, Republic of South Africa. The plant is owned and operated by AECI Limited and has a design production rate of 1000 tonnes per day of ammonia. The plant was commissioned in 1974.

## 1.1 PROGRAM SUMMARY

The Source Test Evaluation (STE) program was carried out as a joint effort between TRW and K-K. TRW's initial review of the Modderfontein plant, shown schematically in Figure 1, resulted in the selection of 25 streams as necessary to the comprehensive STE goals. Of these 25 streams, as summarized in Table 1, nine were actually tested (i.e., Streams 7, 15, 16, 32, 33, 38, 40, 46, and 50). The selection of streams for testing resulted from discussions between K-K and TRW in which streams considered proprietary, not applicable to STE goals, or otherwise restricted were eliminated from the list. The STE thus became limited in scope and focused on the remaining available streams. Later developments indicated that several of the 25 streams were not considered feasible.

The on-site sampling and analysis were performed by K-K. Samples were taken according to the schedule shown in Table 2. Their overall effort spanned a three-week period in November, 1979. The gas samples were analyzed for the species  $H_2O$ ,  $H_2$ ,  $CO$ ,  $CO_2$ ,  $N_2$ ,  $CH_4$ ,  $H_2S$ ,  $COS$ ,  $CS_2$ ,  $R-SH$ ,  $SO_2$ ,  $NH_3$ ,  $HCN$ , and  $NO_x$ . Aqueous samples were analyzed by K-K for the standard wastewater tests (e.g., pH, alkalinity, conductivity, BOD, COD, anions, etc.) with a few supplemental wastewater tests also being performed by a local commercial laboratory, McLachlan & Lazar (pty) LTD.

Wastewater samples were shipped to TRW for comprehensive organic and inorganic analyses per the EPA procedures for Level 1, Level 2, and Priority Pollutants (references 1, 2, 3). The Level 1 methods provide a broad semi-quantitative survey from which constituents found to be present at levels of potential concern are selected for further quantitative examination (Level 2). The Priority Pollutant screening consists of analyses for a specific list of 129 pollutants of concern to the EPA.

Figure 1. Schematic of Modderfontein Koppers-Totzek Coal Gasification Facility

Table 1. PROCESS STREAMS REQUESTED FOR STE PROGRAM

Stream Number*	Stream Description
6	Coal Bin Purge Gas
7	Coal Feed to Gasifier (tested)
9	Gasifier Slag
12	Gasifier Poke Hole Gas
15	Raw Product Gas (tested)
16	Input Water, Cooling Water (tested)
17	Compressed Raw Gas
19	Sulfur-Free Raw Gas
20	Compressed Sulfur-Free Gas
21	Shifted Product Gas
22	CO <sub>2</sub> -Free Product Gas
23	Synthesis Gas
24	Compressed Synthesis Gas
25	Recycled Gas from Synthesis
26	Spent Shift Catalyst
28	Nitrogen Wash Tail Gas
32	Diluted Rectisol Condensate (tested)
33	CO <sub>2</sub> Absorber Tail Gas (tested)
34	CO <sub>2</sub> Rich By-Product Gas
35	H <sub>2</sub> S Rich By-Product Gas
38	H <sub>2</sub> S Absorber Tail Gas (tested)
40	Compressors Condensate (tested)
46	Input Water Purified Sewage Effluent (tested)
48	Cooling Tower Recycle Wash Water
50	Settling Pond Discharge (tested)

\* Stream Numbers correspond to those shown on Figure 1.

Table 2. SCHEDULE FOR SAMPLE ACQUISITION

Streams Sampled/Stream Numbers*	November 1979												
	11	12	13	14	15	16	17	18	19	20	21	22	23
Gas Streams:													
Raw Gas after Raw Gas Blower/15													X
Tail Gas from H <sub>2</sub> S Absorber/38						X							
Tail Gas from CO <sub>2</sub> Absorber/33						X							
Aqueous Streams:													
Input Water-Treated Sewage/46		X											
Process Water-Cooling Water/16									X				
Compressor Condensates/40		X							X				
Settling Pond Effluent/50		X							X				
Diluted Rectisol Condensate/32		X							X				
Solid Streams:													
Sized Coal Feed/7									X				X

\* Stream numbers correspond to those shown on Figure 1.



All of the data obtained from this STE were used in the EPA's Source Analysis Model/IA which compares the measured concentrations on the constituents analyzed to the EPA's Discharge Multimedia Environmental Goals (references 4, 5). This model calculates discharge severities based on the constituent concentrations alone and on the concentrations combined with the stream flowrate (weighted discharge severity). This approach provides a consistent basis for evaluating STE data.

The results of utilizing the SAM/IA approach with the data from the Modderfontein Koppers-Totzek facility are summarized in Figure 2. The two tail gas streams are direct emissions at Modderfontein. The discharge water is the settling pond effluent. Results from the input waters (purified sewage effluent and cooling water) supplied to the gasification facility are also provided for comparison. The data from Modderfontein indicate that the streams tested do not appear to be of particular concern. The discharge severity values obtained are similar to or lower than those obtained on similar streams from other gasifiers (references 6, 7). The discharge severities presented should not be construed as the result of optimised control of pollutants from this unit. Depending upon design of each plant and auxiliary processes, the number and location of effluent streams could vary widely. A more conclusive determination of health and ecological effects or lack thereof requires complementary biological tests. Such tests were not included in this STE.

It should be noted that the magnitude of the discharge severity values result from relatively few constituents. The TDS and WDS values for the two tail gas streams are due primarily to the CO and NH<sub>3</sub> concentrations. The TDS and WDS for the aqueous streams are due mainly to P and Mn and to a lesser extent the metals Fe, Cd, Cu, Ni, Pb, and Zn. The lowest DMEG value in the phosphorous class of compounds was used because individual phosphorous species were not determined. The reduction in both TDS and WDS values for the discharge versus the input waters appears to be due to a decrease in concentrations of P, Cu, Pb, and Zn. These appear to be lost to the settling pond sludge.

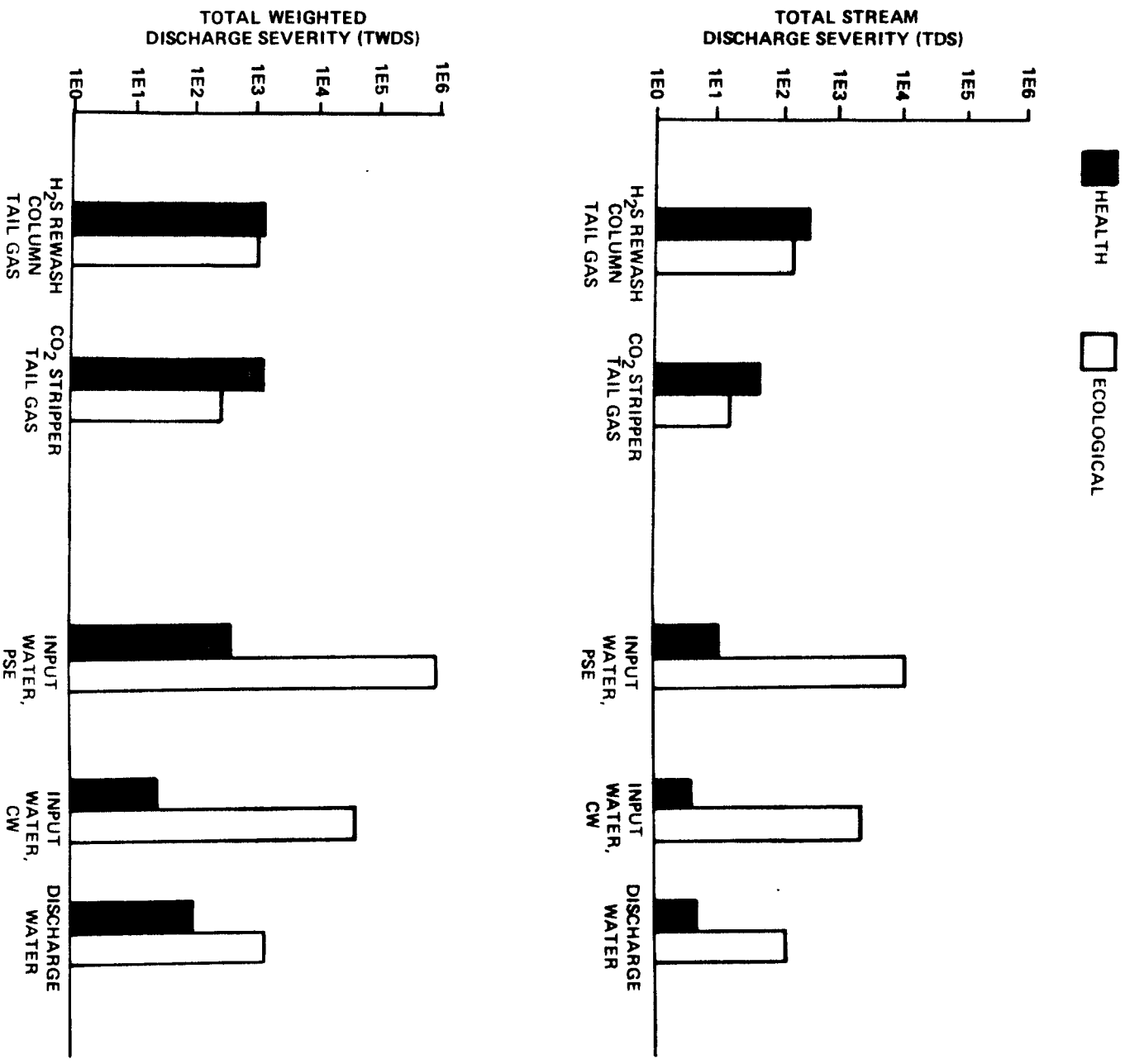


Figure 2. Summary of SAM/IA Results for Koppers-Totzek Facility

## 1.2 CONCLUSIONS AND RECOMMENDATIONS

The limited source test program conducted at the Modderfontein facility has provided some of the key data needed for the environmental assessment of Koppers-Totzek based synthetic fuels plants which may be built in the United States. The data obtained do not indicate that any special problems should be encountered in controlling the process effluents to environmentally acceptable levels for plants built in the U.S. Relatively steady state conditions were realized during the test period thus most of the samples taken were generally representative of typical plant operation. This in turn indicates that the data can reliably be used as intended. One exception was the Rectisol unit which apparently was not operating properly at the time and hence data on Rectisol tail gas characteristics are not believed to be typical.

Except for the Rectisol tail gases, additional sampling of the streams which were the subject of the initial test program is not expected to yield information other than of a confirmatory nature. Hence only limited additional sampling of these streams is suggested in conjunction with aqueous stream sampling as outlined below. In the case of the Rectisol tail gases, no additional gas stream sampling is recommended since the specific Rectisol design for ammonia production featuring "cold" shift between  $\text{H}_2\text{S}$  and  $\text{CO}_2$  removal would not be employed for synfuels production and data on this type of design would not be especially useful for evaluation of synfuels discharge streams.

Several aqueous and solid waste streams were not subject to testing in the initial program, however, data relating to the characteristics of these streams would be helpful in the evaluation of pollution control needs for U.S. facilities. Table 3 identifies these streams along with the type of data of interest for each stream. As indicated in the table, data on the characteristics of aqueous streams resulting from raw gas cooling and particulate removal, from slag quenching and from the cold water wash unit (HCN removal) are needed. Of major concern are constituents in the aqueous wastes (e.g.,  $\text{NH}_3$ , HCN,  $\text{H}_2\text{S}$ ) which may become volatilized in the clarifier or cooling tower systems resulting in atmospheric discharges. In addition, characteristics of the gas cooling/washing wastewaters would provide an indication of some of the original constituents in the crude

Table 3. ADDITIONAL DATA NEEDS FOR KOPPERS-TOTZEK PROCESS

Stream Name	Stream Number	Constituents/Parameters of Interest	Uses of/Justification for Additional Data
Coal Feed to Gasifier	7	Proximate/Ultimate, Trace elemental survey.	To corroborate data collected from initial STE.
Input Water (Purified Sewage Effluent)	45	Standard wastewater tests*, Trace element survey, Organic compounds survey, priority pollutants, Level 2 as needed (POM's).	To corroborate data collected from initial STE and to provide background comparisons for the aqueous process streams.+
Input Water (Cooling Water)	16	Standard wastewater tests*, Trace element survey, Organic compounds survey, priority pollutants, Level 2 as needed (POM's).	To corroborate data collected from initial STE and to provide background comparisons for the aqueous process streams.+
Washer Cooler Blowdown	44	Standard wastewater tests*, Trace element survey, Organic compounds survey, Level 2 as needed (POM's).	To indicate those constituents of crude K-T gas which are likely to be removed/condensed with water in this or alternate quench designs.+
Disintegrator Blowdown	43	Standard wastewater tests*, Trace element survey, Organic compounds survey, Level 2 as needed (POM's).	Same as for Washer Cooler Blowdown.+
ESP Wash Water	41	Standard wastewater tests*, Trace element survey, Organic compounds survey, Level 2 as needed (POM's).	Same as for Washer Cooler Blowdown.+
Raw Gas Compressors Condensate	40	Standard wastewater tests*, Trace element survey, Organic compounds survey, Level 2 as needed (POM's).	To corroborate data collected from initial STE and to allow constituent material balances around gasification operations.+
HCN Removal Wash	39	Standard wastewater tests*, Trace element survey, Organic compounds survey, Level 2 as needed (POM's).	To allow constituent material balances around gasification operations.+
Slag Quench Blowdown	11	Standard wastewater tests*, Trace element survey, Organic compounds survey, Level 2 as needed (POM's).	To indicate solids buildup and consequent blowdown requirements in the slag cooling circuit and to allow constituent material balances around gasification operations.+
Clarifier Influent	45	Standard wastewater tests*, Trace element survey, Organic compounds survey, priority pollutants, Level 2 as needed (POM's).	To allow constituent material balances around gasification operations.+
Clarifier Effluent	45	Standard wastewater tests*, Trace element survey, Organic compounds survey, priority pollutants, Level 2 as needed (POM's).	To compare to clarifier influent in order to indicate degree of removal of both dissolved and suspended materials expected during clarification and the possible atmospheric emissions of volatile substances.+
Cooling Tower Recycle Water	48	Standard wastewater tests*, Trace element survey, Organic compound survey, priority pollutants, Level 2 as needed (POM's).	To indicate possible atmospheric emissions of volatile substances in clarifier effluent and to allow constituent material balances.
Quenched Gasifier Slag	10	RCRA leach test for soluble elements/substances which may be potentially toxic (POM's).	To provide an indication of the likely disposal requirements for K-T solid wastes for facilities constructed in the U.S. and to be able to relate data to U.S. coals.+
Settled Clarifier Solids/Clarifier Underflow	49	RCRA leach test for soluble elements/substances which may be potentially toxic (POM's).	To provide an indication of the likely disposal requirements for K-T solid wastes for facilities constructed in the U.S.+
Raw Gas after Blower	15	Flow rate, temperature, H <sub>2</sub> , CO, CO <sub>2</sub> , H <sub>2</sub> S, COS, CS <sub>2</sub> , mercaptans, NH <sub>3</sub> , HCN, methane, higher hydrocarbons, POM's, particulate matter, H <sub>2</sub> O.	To corroborate initial STE data and to allow constituent material balances around gasification operations.
Raw Gas prior to Acid Gas Cleanup and Shift	18	Flow rate, temperature, H <sub>2</sub> , CO, CO <sub>2</sub> , H <sub>2</sub> S, COS, CS <sub>2</sub> , mercaptans, NH <sub>3</sub> , HCN methane, higher hydrocarbons, POM's, particulate matter, H <sub>2</sub> O.	To allow constituent material balances around gasification operations.

\* Standard wastewater tests include: Flow rate, temperature, hardness, conductivity, dissolved oxygen, pH, alkalinity, total suspended solids, total dissolved solids, BOD, COD, TOC, NH<sub>3</sub>, SCN<sup>-</sup>, CN<sup>-</sup>, Cl<sup>-</sup>, sulfur species, phosphorus species.

+ Bioassay Tests

The future data base may have to include bioassay data to fully determine the requirements for meeting U.S. environmental standards. Such tests would focus on final discharges such as Stream 10 above and any final aqueous effluents. However bioassay tests on selected in-process streams would have value because the resultant larger data base would aid in correlating biological toxicity with chemical composition.

gas, which would be helpful in evaluation of potential wastes generated by K-T designs featuring other gas cooling/particulate removal systems. In order to complete constituent mass balances around the gasifier/gas cooling systems, repeat sampling of the raw gas (after blower) would be desirable so that a consistent set of data is available.

Also indicated in Table 3 are solid wastes/sludges generated by the slag quenching operating and by the clarifier unit. The primary concern with these wastes is the leachability of specific trace elements and other potentially toxic substances. Such data are specific to each coal. Samples can be generated in a test gasifier. The leach test referred to in the table is that specified in regulations promulgated by the EPA under the Resource Conservation and Recovery Act of 1976 (RCRA). This type of data would be used as an indication of disposal requirements/methods for solid wastes generated by facilities built in the U.S. Conceivably it could also become pertinent to perform bioassay tests in conjunction with future STE efforts if these data should also be necessary to understanding the requirements of U.S. facilities.

It should be commented that additional sampling/testing activities at the Modderfontein facility would have as the primary goal that of providing basic characterization data on K-T generated wastes so that control technology requirements for facilities built in the U.S. can be identified early in the planning stages. It is not intended that any data resulting from tests of a commercial operating facility at Modderfontein be used for the purpose of either promoting or criticizing specific process designs or operating practices of that facility. The Modderfontein plant was designed in 1972 to meet the specific environmental requirements in force at that time.

## 2. PLANT DESCRIPTION

The testing of a Koppers-Totzek (K-T) coal gasification facility was conducted at the Number 4 Ammonia Plant at Modderfontein, Republic of South Africa. The plant is owned and operated by AECI Limited and has a design production rate of 1000 tonnes per day of ammonia. The plant was commissioned in 1974 and operated in 1978 with an on-stream time of 81%. This plant utilizes six K-T two-burner coal gasification reactors. A process schematic for the Number 4 Ammonia Plant showing the various process modules is presented in Figure 1.

Descriptions of the K-T process in general, operating conditions specific to the Modderfontein plant, and sampling point locations are provided in this section. Further detailed discussions of the Modderfontein plant can be found in the appended K-K report.

### 2.1 PROCESS DESCRIPTION

The licensor and developer of the Koppers-Totzek (K-T) gasification process is K-K of Essen, Federal Republic of Germany. As of 1978, there were 47 K-T coal gasification modules operating in fifteen plants throughout the world. All of the K-T gasifiers in operation as of 1978 are used to make synthesis gas as an input stream for the production of ammonia.

The K-T process operates on an entrained bed principle. It utilizes a high temperature, atmospheric pressure reaction fueled by a continuous co-current input stream of coal, oxygen, and steam. The gasification reactor vessel is a horizontal, ellipsoidal, double-walled steel chamber with a refractory lining. Two gasifier designs are available. The two-burner gasifier design has a burner head located on each end of the ellipsoid. The four-burner gasifier resembles two of the two-burner gasifiers which intersect one another at a  $90^{\circ}$  angle. A burner head is located at each of the ends of the two intersecting ellipsoids. Figure 3 schematically depicts a two-burner gasifier design of the type employed at

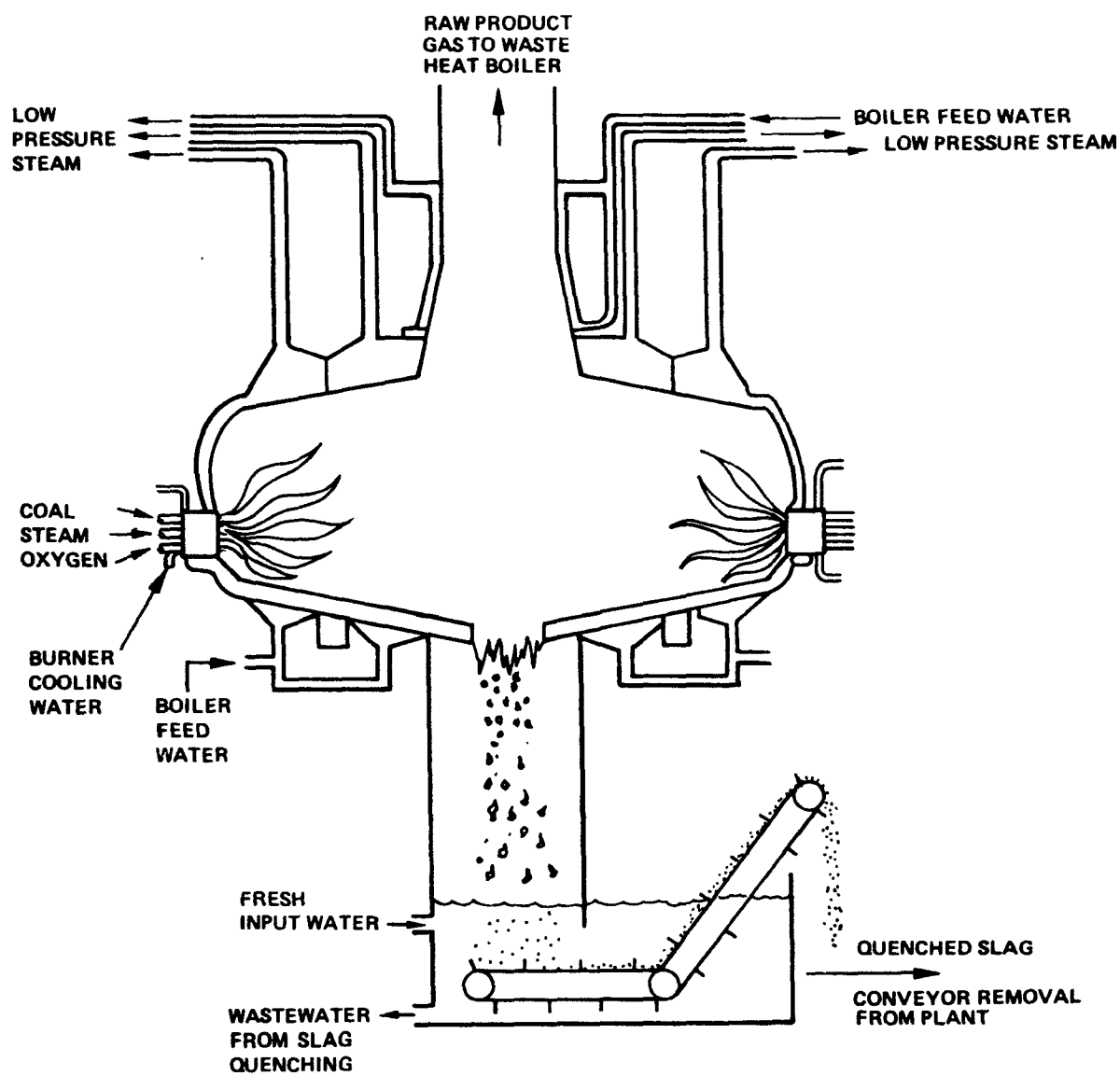


Figure 3. Koppers-Totzek Gasifier

Modderfontein. The reaction gases exit the gasifier vertically from a port located on top of the gasifier in the center of the ellipsoid.

The process reactants are fed to the gasifier in the following manner: a continuous screw conveyor feeds the pulverized coal to mixing nozzles which are located at the ends of the gasifier but which are not part of the burner head. The coal is then entrained in a premixed stream of steam and oxygen. The mixture is then injected into the gasifier through sets of two adjacent nozzles comprising each burner head.

Coal ash residue from the gasification process is removed from the reactor by two mechanisms. Approximately 50 percent of the ash flows down the gasifier walls as a molten slag and drains into a slag quench tank where circulating water causes it to shatter into a granular form. A conveyor lifts the granules out of the quench tank and transports them out of the plant area. The remainder of the coal ash leaves the gasifier as a "soot" entrained in the raw product gas. The entrained soot is largely removed from the gas stream in a water spray tower.

The gasifier operates with a flame temperature of  $2000^{\circ}\text{C}$  ( $3650^{\circ}\text{F}$ ) or more, and a gas outlet temperature of  $1400^{\circ}$  to  $1600^{\circ}\text{C}$  ( $2550^{\circ}$  to  $2900^{\circ}\text{F}$ ). The pressure inside the gasifier is essentially one atmosphere. The coal is gasified within about 1 second. Opposing burner heads in the reactor provide for high turbulence and efficient mixing of reactants. The heterogeneous reactions between carbon, oxygen and steam in the input stream are generally characteristic of coal gasification. The major constituents of the gasifier output stream are carbon monoxide and hydrogen.

Most of the organic and inorganic sulfur contained in the coal is converted to  $\text{H}_2\text{S}$  and  $\text{COS}$  at a ratio of about 9 to 1. Smaller amounts of  $\text{CS}_2$  and  $\text{SO}_2$  are also formed. A portion of the feed coal sulfur is retained with the ash, with the retention ranging from 5% to over 30% depending on the coal. Organic nitrogen contained in the coal is converted mainly to elemental nitrogen, although small amounts of  $\text{NH}_3$ ,  $\text{HCN}$  and  $\text{NO}$  are also generated.

Other auxiliary processes and operations of the K-T process include coal preparation, oxygen production, particulate removal from the raw gas, and treatment (and recycle) of process water used for gas cooling and



cleaning and slag quenching. These auxiliary processes operate more or less independently of the actual gasification process and do not represent specialized development or adaption to the K-T process. Consequently they are not described in detail in this report.

## 2.2 PLANT OPERATION DURING THE TEST PERIOD

The joint sampling and analysis activities involving K-K, AECI, and TRW were conducted during the period November 7, through November 29, 1979. During this period one of the six gasifiers in the plant was not operating. However, nearly full design capacity was obtained throughout this period with the remaining 5 gasifiers. All collection of samples and associated operating data occurred at production rates of between 102,000 and 104,000 normal cubic meters per hour ( $\text{Nm}^3/\text{h}$ ) of dry raw gas. K-K personnel reported that during the test period the gasification plant operated in a very stable manner with no process upsets. However, problems were encountered with the operation of the downstream Rectisol unit for  $\text{H}_2\text{S}$  removal which prevented the collection of sulfur species data on the tail gas stream.

## 2.3 SUMMARY OF SAMPLE POINT LOCATIONS

Sampling locations for each of the nine streams tested are provided below. The stream numbers given in parentheses correspond to those shown in Figure 1. The coal dust sample (7) was taken at the exit of the coal dust bunker in the coal preparation operation. The raw gas (15) was sampled from the common duct leading to the raw gas holder. Thus the raw gas sample represents the average gas composition from all five operating gasifiers. The Rectisol tail gases (33, 38) were sampled from tap lines fitted to the respective vent lines. The purified treated sewage input water (45) was sampled from the main line entering the plant. The cooling water input (16) was sampled from the pressure line entering the plant. The cooling water input (16) was sampled from the pressure line entering the plant. Both of these input water streams originate from facilities in the Modderfontein complex other than the coal gasification facility. Condensate from the raw gas compressor was taken from the line leading to the wash water system which collects the various wastewaters and conveys them to the clarifier. The hot condensate effluent from the methanol/water separation column of the Rectisol unit is diluted with

cooling water. This diluted condensate (32) was sampled from the line leading to the wash water system. The settling pond effluent (49) was sampled at the exit of the channel which collects the overflow from numerous drain pipes in the pond.



### 3. SAMPLING AND ANALYSIS METHODS

The sampling and analysis responsibilities for the K-T facility test were divided between TRW and K-K. K-K performed all of the sampling and most of the on-site analyses. TRW arranged to have the remaining time-critical analyses performed by a local South African laboratory and to have portions of the coal feed and aqueous process stream samples shipped back to TRW for analysis. Table 4 summarizes responsibilities of the participants and the following sections describe the methods used by K-K and TRW for their respective activities.

#### 3.1 K-K METHODS

K-K's responsibilities were for:

- All sampling,
- All gas stream analysis, and
- Much of the coal and aqueous stream analyses.

The methods used in each of these areas are described in the following paragraphs.

##### 3.1.1 Coal Feed Sampling and Analysis

The coal feed dust was sampled over half-hour periods on November 19 and 23. The November 19 sample was supplied to TRW and the November 23 sample was analyzed by K-K using Deutsche Industrie Norm (DIN) procedures for the following parameters:

- Particle Size Distribution - DIN 51 704
- Moisture Content - DIN 51 718
- Ash Content - DIN 51 719
- C and H Content - DIN 51 221
- N Content - DIN 51 722
- Total and Combustible S - DIN 51 724
- O Content - by difference
- Ash Composition - DIN 51 729.

### 3.1.2 Gas Stream Sampling and Analysis

The sampling procedure for all three gas streams consisted of flowing the gas through a manifold to the various absorption trains over a two-hour period. Each of these trains contained two to three wash bottles in a series and a gas meter. In the case of the Draeger tubes used for  $\text{CS}_2$  and  $\text{NO}_x$ , these were placed in the stream of  $\text{H}_2\text{S}$  free gas eluting from the cadmium acetate gas scrubber bottles. Each of the gas streams was sampled only once. The Rectisol tail gases were sampled and analyzed on November 16 and the raw gas was sampled and analyzed on November 23.

A summary of the gas analysis methods is given in Table 5. Generally these methods are of acceptable specificity and accuracy for source evaluations of this type except for the Draeger tube measurements which can be subject to interferences from other species present, and the Orsat method for hydrocarbons which failed to provide adequate detection limits. Gas chromatography (GC) techniques are preferable for hydrocarbons, however, problems with the GC equipment available on-site prevented its use in this source test.

### 3.1.3 Aqueous Stream Sampling and Analysis

The sampling procedure for all five aqueous streams consisted of collecting and preserving six samples within a one-hour period on each of two days a week apart, November 12 and 19. These samples were used for the determination of suspended solids, one sample was acidified immediately for  $\text{NH}_3$  analysis, one sample was filtered into an alkali/cadmium carbonate solution for the analysis of  $\text{H}_2\text{S}$  and other acidic species, and one sample was filtered and used for the remaining analyses.

A summary of the analytical methods used by K-K is given in Table 6. These methods are essentially equivalent to standard test procedures used in the U.S. and are acceptable for this type of source evaluation.

Table 4. DISTRIBUTION OF SAMPLING AND ANALYSIS RESPONSIBILITIES

	K-K	TRW
SAMPLING	All	—
ANALYSIS		
<u>Level 1</u>		
Coal Feed	—	Trace Element Survey (SSMS*) Proximate/Ultimate*
Gases	Fixed Gases (CO, CO <sub>2</sub> , O <sub>2</sub> , N <sub>2</sub> , H <sub>2</sub> ) Sulfur Species (H <sub>2</sub> S, COS, CS <sub>2</sub> , Mercaptans) Hydrocarbons (C <sub>1</sub> to C <sub>7</sub> )	
Liquids	Wastewater Tests (pH, TSS, TDS, hardness, alkalinity, conductivity, COD, NH <sub>3</sub> , CN <sup>-</sup> , SCN <sup>-</sup> , H <sub>2</sub> S, S <sub>2</sub> O <sub>3</sub> <sup>=</sup> , SO <sub>3</sub> <sup>=</sup> , SO <sub>4</sub> <sup>=</sup> , PO <sub>4</sub> <sup>-3</sup> , Cl <sup>-</sup> , methanol, dissolved oxygen)	Wastewater Tests (Nitrates, CN <sup>-†</sup> , SCN <sup>-†</sup> , BOD <sup>†</sup> , COD <sup>†</sup> , TOC, total phenols, S <sup>=</sup> )
<u>Priority Pollutants</u>		
Liquids		Organic Screening (volatiles and base/neutral and acid non-volatiles) Inorganic Screening (Ag, As, Be, Cd, Cr, Cu, Hg, Pb, Ni, Sb, Se, Tl, Zn)
<u>Level 2</u>		
Liquids	—	PAH compounds Additional Inorganic quantitation of Fe and Mn

\* Subcontracted to Commercial Test and Engineering, Inc.

† Subcontracted to McLachlan and Lazar (pty) LTD

Table 5. SUMMARY OF K-K GAS ANALYSIS METHODS

Parameter(s)	Method
H <sub>2</sub> , CO, CO <sub>2</sub> , N <sub>2</sub> , hydrocarbons	Orsat analyzer
H <sub>2</sub> O	Dew point hygrometer
H <sub>2</sub> S	Absorbed in cadmium acetate solution. CdS precipitate is acidified in presence of iodine and determined iodometrically.
CS <sub>2</sub> , SO <sub>2</sub> , COS	H <sub>2</sub> S-free gas is first obtained using copper acetate. CS <sub>2</sub> is then determined by Draeger tube. SO <sub>2</sub> iodometrically after absorption in iodine solution. COS by difference after determining total non-H <sub>2</sub> S sulfur compounds by absorption in KOH, oxidation with H <sub>2</sub> O <sub>2</sub> , and precipitation with BaCl <sub>2</sub> .
NO <sub>x</sub>	Measured with Draeger tubes in H <sub>2</sub> S-free gas obtained as above.
Mercaptans	GC, Tracor 270HA Sulfur Analyzer using Tracor "Special" silica gel column.
HCN	Absorbed in KOH. The KCN is reacted with Br <sub>2</sub> to yield CNBr. The CNBr is determined iodometrically. (Ref. Ruhrgas. A.G.)
NH <sub>3</sub>	Absorbed in H <sub>2</sub> SO <sub>4</sub> and determined per DEV Standard Method. (1)

(1) Deutsch Einheit Vorschriften (a compilation of standard methods)

Table 6. SUMMARY OF K-K WATER ANALYSIS METHODS

Parameter	K-K Method	Comparable American Method
pH	DEV STD. Method (1)	APHA 424 (3)
Conductance	DEV STD. Method	APHA 205
Dissolved and Suspended solids	DEV Method H1 and H2	APHA 208-B or C and APHA 208-D
Hardness	DEV STD. Method	APHA 309-B
Acidity/Alkalinity	DEV STD. Method	APHA 402/APHA 403
Chloride	DEV Method D1, No. 2	APHA 408
Sulfide	Precipitation with $\text{CdCO}_3$ . $\text{CdS}$ precipitate is determined iodometrically.	APHA 427
Sulfite and Thiosulfate	Iodometric titration of filtrate from $\text{CdS}$ separation determines the total. $\text{SO}_3^{2-}$ is complexed with formaldehyde and the $\text{S}_2\text{O}_3^{2-}$ is titrated with iodine. $\text{SO}_3^{2-}$ is determined by difference.	APHA 429
Total Phosphate	DEV D11-1B (Molybdenum Blue)	APHA 425
Ammonia in water	DEV STD. Method (Make water sample alkaline. Sparge into std. $\text{H}_2\text{SO}_4$ , and back titrate excess $\text{H}_2\text{SO}_4$ .)	APHA 418
Sulfate	DEV Method D5 (Barium precipitation).	APHA 427
Cyanide and Thiocyanate	Boyer Method (2) Purge into $\text{KOH}$ and titrate with $\text{AgNO}_3$ . $\text{SCN}^-$ remaining in solution is titrated by Bromocyanide method. (DEV method for $\text{HCN}$ in gases.)	APHA 413
Methanol	GC/Thermal conductivity or GC plus hydrogenation to methane and FID.	No routine standard method
Ammonium Ion	Analyze as $\text{NH}_3$ (DEV STD. Method)	APHA 418
COD	DEV Method H4-1A or 1B (Chemische Sauerstoff Bedarf)	APHA 508

(1) Deutsch Einheit Vorschriften (a compilation of standard methods)

(2) This is not a standard method. The procedure is adapted from Gas und Wasserfach. Vol. 105, Heft. 13, p. 334ff.

(3) American Public Health Association, Standard Methods for the Examination of Water and Wastewater, 14th Edition, 1976.



### 3.2 TRW ANALYSIS METHODS

TRW's responsibilities were for

- Any Level 1 analyses not included in Krupp-Koppers effort,
- Priority pollutant screening, and
- Level 2 analyses.

The methods used in each of these areas are described in the following paragraphs.

#### 3.2.1 Level 1 Analysis

Most of the Level 1 analyses that are time critical were performed by K-K (i.e., all gas analyses and most wastewater quality tests). The only wastewater quality tests remaining were nitrates and BOD, which were then handled by a local commercial laboratory in Johannesburg. Replicate analysis of a few of the species measured by K-K were also performed by the local laboratory. The methods used by the commercial laboratory were comparable to U.S. methods and were acceptable for source evaluations. The analysis of organic materials and trace metals was performed by TRW on preserved aliquots of the eight aqueous stream samples that were shipped back to the U.S. The methods used for the Level 1 analyses were taken from the EPA-IERL/RTP procedures manual (reference 1).

#### 3.2.2 Priority Pollutant Screening Analysis

The analyses for organic priority pollutants were done in three phases. Volatile, acid extractable non-volatile and base-neutral extractable non-volatile organics were tested in accordance with the EPA procedures manual (reference 2). The samples were analyzed by a gas chromatography-mass spectrometry (GC/MS) system equipped with an INCOS data system. A computer program was used to screen the data and the final reports were manually examined and if necessary, modified. The specific parameters utilized in each of the three phases of the organic priority pollutant screening are delineated in Table 7. The compounds analyzed for are specifically mandated by the EPA procedures and are listed in Tables 8, 9, and 10.

Table 7. PRIORITY POLLUTANT SCREENING ANALYTICAL PARAMETERS

	VOLATILE SPECIES	SEMI-VOLATILE ACIDS	SEMI-VOLATILE BASE/NEUTRALS
METHOD	PURGE AND TRAP	DIRECT INJECTION OF CONCENTRATED ORGANIC EXTRACT	
Organic Extract Sample Size	5 mL	1 µL	1 µL
GC Conditions:			
Column	8'-0.2% Carbowax 1500	6'-1% SP12400A	6'-3% SP2250
Temperature Program	60°C - hold 4 min 60°C → 170°C at 8°/min 170°C - hold 12 min	30°C → 190°C at 8°/min	50°C - hold 4 min 50°C → 260°C at 8°/min
Injector	75°C	190°C	275°C
Jet separator	295°C	250°C	275°C
Ion source	240°C	220°C	250°C
Helium flow	30 mL/min	30 mL/min	30 mL/min
Mass Spec Conditions:			
Mass range	40 - 540 AMU	40 - 450 AMU	40 - 450 AMU
Scan up	1.90 sec	1.90 sec	1.90 sec
Scan down	0.00 sec	0.00 sec	0.00 sec
Hold top	0.00 sec	0.00 sec	0.00 sec
Hold bottom	0.10 sec	0.10 sec	0.10 sec
Scan time	2.00 sec	2.00 sec	2.00 sec
Internal Standard (I.S.)	Bromochloromethane 1,4-Dichlorobutane	D <sub>10</sub> -Anthracene	D <sub>10</sub> -Anthracene
I.S. Amount	20.0 µg/L	10.0 µg/mL Extract	10.0 µg/mL Extract

Table 8. VOLATILE ORGANIC COMPOUNDS ANALYZED

Compounds	Mass used to quantitate
Bromochloromethane (internal standard)	128
Chloromethane	50
Dichlorodifluoromethane	101
Bromomethane	94
Vinyl Chloride	62
Chloroethane	64
Methylene Chloride	88
Trichlorofluoromethane	101
1,1-Dichloroethylene	96
1,1-Dichloroethane	63
Trans-1,2-Dichloroethylene	61
Chloroform	83
1,2-Dichloroethane	98
1,1,1-Trichloroethane	97
Carbon Tetrachloride	117
Bromodichloromethane	127
1,4-Dichlorobutane (internal standard)	55
1,2-Dichloropropane	112
Trans-1,3-Dichloropropene	75
Trichloroethylene	130
Benzene	79
Cis-1,3-Dichloropropene	75
1,1,2-Trichloroethane	97
Dibromochloromethane	127
Bromoform	173
Tetrachloroethylene	164
1,1,2,2-Tetrachloroethane	83
Toluene	92
Chlorobenzene	112
Ethylbenzene	106

Table 9. ACIDIC SEMI-VOLATILE COMPOUNDS ANALYZED

Compound	Mass used to quantitate
D <sub>10</sub> -Anthracene (internal standard)	188
2-Chlorophenol	128
2-Nitrophenol	139
Phenol	94
2,4-Dimethylphenol	107
2,4-Dichlorophenol	162
2,4,6-Trichlorophenol	196
4-Chloro-m-cresol	142
2,4-Dinitrophenol	184
4,6-Dinitro-o-cresol	198
Pentachlorophenol	266
4-Nitrophenol	65

Table 10. BASE/NEUTRAL SEMI-VOLATILE COMPOUNDS ANALYZED

Compound	Mass used to quantitate
D <sub>10</sub> -Anthracene (internal standard)	188
1,3-Dichlorobenzene	146
1,4-Dichlorobenzene	146
Bis(2-Chloroethyl)ether	93
1,2-Dichlorobenzene	146
Hexachloroethane	117
Bis(2-Chloroisopropyl)Ether	77
N-Nitrosodi-n-Propylamine	70
Nitrobenzene	123
1,2,4-Trichlorobenzene	180
Hexachlorobutadiene	225
Naphthalene	128
Bis(2-Chloroethoxy)Methane	93
Isophorone	82
Hexachlorocyclopentadiene	237
2-Chloronaphthylene	162
Acenaphthylene	152
Acenaphthene	154
Dimethylphthalate	163
2,6-Dinitrotoluene	63
Fluorene	166
4-Chlorophenylphenylether	204
2,4-Dinitrotoluene	89
1,2-Diphenylhydrazine	77
Diethylphthalate	149
N-Nitrosodiphenylamine	169
Hexachlorobenzene	284
4-Bromophenoxybenzene	248
Anthracene/Phenanthrene	178
Di-n-Butylphthalate	149
Fluoranthene	202
Pyrene	202
Benzidine	184
Butylbenzylphthalate	149
Bis(2-ethylhexyl)Phthalate	167
Benzo(a)Anthracene	228
Chrysene	228
3,3'-Dichlorobenzidine	252
Di-N-Octylphthalate	149
Benzo(b)Fluoranthene	252
Benzo(k)Fluoranthene	252
Benzo(a)Pyrene	252
Dibenzo(a,h)Anthracene	278
Indeno-1,2,3-(c,d)-Pyrene	276
Benzo(g,h,i)Perylene	276

The components eluting from the GC column are monitored by a continuously scanning mass spectrometer. The mass spectra are then stored on computer disk to be examined at a later date. A computer program which mimics the manual procedure for qualitative and quantitative analysis of samples for priority pollutants is used as a first pass analysis of the data. Before any analyses are attempted, a standard or series of standards are run by GC/MS. This operation provides the program with three pieces of information: a reference mass spectrum, a relative retention time and a relative response factor for each compound. Once these factors are generated, the samples are analyzed. The standards are also run on a routine basis during the sample analysis to allow for adjustment of the relative retention times and relative response factors.

The program tests for each compound in sequence until the list of compounds is exhausted. The computer outputs the results which are then manually checked for consistency, completeness and correctness. The internal standard results are manually examined to assure that the retention time and peak area are within acceptable limits. The chromatogram is examined to assure that all components are identified. That is, if a chromatographic peak is present but is not identified as a priority pollutant, its spectrum is manually examined to assure that it is not a pollutant. And finally, a general comparison of the program results and the GC/MS data is made to assure that no inconsistencies exist.

The analysis for the required 13 priority pollutant metals (i.e., Ag, As, Be, Cd, Cr, Cu, Hg, Pb, Mn, Sb, Se, Tl, and Zn) were also performed in accordance with the EPA procedures manual (reference 2).

### 3.2.3 Level 2 Analysis

The level 2 analysis of the Modderfontein samples consisted of atomic absorption techniques (AAS) for Fe and Mn, and a high performance liquid chromatography (HPLC) technique for polynuclear aromatic hydrocarbon (PAH) compounds. These two metals and the PAH compounds were selected on the basis of comparing the Level 1 data to the EPA's discharge multimedia environmental goal (DMEG) values, thus determining the constituents of potential environmental concern which warrant further investigation. The AAS techniques were standard methods (reference 8). The HPLC technique was developed by TRW and is described here briefly.

The HPLC technique utilizes a reverse phase, quaternary solvent system for separation of three-ring and larger PAH compounds. Both UV and fluorescence detectors are used in tandem in order to yield corroborative data for the identification and quantitation of the compounds present. A synopsis of the HPLC parameters is given below.

Apparatus: A DuPont model 850 high pressure liquid chromatograph equipped with the DuPont variable wavelength UV spectrophotometer and a fluorescence detector in tandem was used.

Reagents: PAH standards were purchased from several sources. The sources used included Aldrich, Inc. (San Leandro, CA 94577); Analabs, Inc. (80 Republic Drive, North Haven, CT 06473), and Chemicals Procurement Laboratories, Inc. (18-17 130th St., College Point, NY 11356). Chromatographic solvents: methanol, acetonitrile and tetrahydrofuran; were purchased from Burdick and Jackson (Muskegon, Mich.). Water was J.T. Baker brand HPLC water.

Instrument Parameters:

HPLC Columns	2 DuPont Zorbax <sup>®</sup> ODS, 4.6 mm ID x 25 cm (total column length was 50 cm)
Mobile Phase	Solvent A - 15% water/85% methanol Solvent B - Tetrahydrofuran/70% Acetonitrile
Gradient	Hold at 5% B for 90 minutes then a linear gradient to 100% in 20 minutes
Temperature	45°C
Flow	1.0 mL/min.

A number of PAH compounds have been obtained which are used to: 1) spike the samples in order to determine compounds present by retention time and relative response to the two detectors, and 2) prepare standard mixtures for quantitation of the PAH compounds. A list of the compounds used to identify and quantitate PAHs in the Modderfontein samples is given in Table 11. Calibration mixtures of the compounds identified in the samples were prepared and run at four different quantitative levels in order to bracket the sample concentrations and provide accurate quantitation.

In addition to using the relative response data from the two detectors, further qualitative data were obtained by collection fractions off the HPLC and analyzing these by GC/MS. The eluent from the HPLC column was collected over five minute intervals, which resulted in eleven fractions

for each sample. These fractions were evaporated to near dryness at ambient conditions under a steady flow of argon. Once the concentration step was completed, the samples were analyzed by GC/MS. A 6 ft (1.9m) 3% Dexsil 300 GC column programmed at 4°C from 100°C to 300°C was used to separate the components in the collected fractions. These data were then used to confirm identifications and the selection of compounds for the calibration mixture.

Table 11. PAH COMPOUNDS USED AS STANDARDS

Phenanthrene	Dibenzo(a,c)anthracene
Fluoranthene	Dibenzo(a,h)anthracene
Pyrene	Picene
Chrysene	o-Phenylenepyrene
9-Phenylanthracene	Benzo(g,h,i)perylene
Benzo(b)fluorene	9,10-Diphenylanthracene
Benzo(k)fluoranthene	Dibenzo(a,i)pyrene
1,2-Benzofluorene	5,6,11,12-Tetraphenylnaphthacene
1,2-Benzanthracene	Coronene
Perylene	Decacyclene
Benzo(a)pyrene	

#### 4. RESULTS

The data presented in this section are the combined results from the efforts of both K-K and TRW. The methods used were described in Section 3, and the division of responsibilities was summarized in Table 4.

##### 4.1 COAL FEED STREAM

The coal dust sample may be characterized as Bituminous, High Volatile B coal based on the results of proximate and ultimate analyses, as shown in Table 12. The coal is very high in ash content and low in sulfur compared to most U.S. coals. A trace element survey was also performed on the coal sample, yielding the results shown in Table 13. More precise determination of the major minerals in the ash, along with particle size distributions and other measurements, were performed by K-K and can be found in their report (Appendix A). The differences between the South African coal and American coals and the effect this has on the composition of the product, by-product, and waste streams must be kept in mind by anyone trying to use the data in this STER to assess the characteristics of K-T facilities that might be built in the U.S.

Table 12. PROXIMATE AND ULTIMATE RESULTS  
FROM COAL ANALYSIS

PROXIMATE ANALYSIS			ULTIMATE ANALYSIS		
	As Rec'd	Dry Basis		As Rec'd.	Dry Basis
% Moisture	1.49	xxxxx	% Moisture	1.49	xxxxx
% Ash	19.60	19.90	% Carbon	64.41	65.38
% Volatile	27.52	27.94	% Hydrogen	3.72	3.78
% Fixed Carbon	51.39	52.16	% Nitrogen	1.12	1.14
	100.00	100.00	% Chlorine	0.01	0.01
			% Sulfur	0.99	1.01
Btu/lb.(kcal/kg)	10853(6028)	11017(6119)	% Ash	19.60	19.90
% Sulfur	0.99	1.01	% Oxygen (diff)	8.66	8.78
				100.00	100.00



Table 13. SSMS RESULTS FROM COAL ANALYSIS

Element	Concentration (ppm)	Element	Concentration (ppm)
Lithium	71	Bromine	0.8
Sodium	>500	Iodine	0.8
Potassium	>500	Scandium	1
Rubidium	4	Yttrium	26
Cesium	3	Titanium	>500
Beryllium	0.8	Zirconium	100
Magnesium	>500	Vanadium	15
Calcium	>500	Niobium	10
Strontium	320	Tantalum	ND
Barium	>500	Chromium	51
Boron	3	Molybdenum	2
Aluminum	>500	Tungsten	ND
Gallium	17	Manganese	26
Silicon	>500	Iron	>500
Germanium	0.5	Cobalt	2
Tin	2	Nickel	3
Lead	7	Copper	5
Phosphorus	>500	Silver	ND
Arsenic	4	Zinc	1
Antimony	0.4	Cadmium	ND
Bismuth	ND	Lanthanum	21
Sulfur	>500	Cerium	52
Selenium	1	Praseodymium	5
Tellurium	0.3	Neodymium	10
Fluorine	310	Thorium	9
Chlorine	15	Uranium	6

ND = Not detected (average detection limit is 0.2ppm)

## 4.2 GAS STREAMS

The three gas streams studied were sampled per the schedule shown in Table 1. The engineering data obtained were as follows:

<u>Stream/Stream Number (from Figure 1)</u>	<u>Flow Rate</u>	<u>Temperature</u>
Raw gas after raw gas blower/15	28.3 Nm <sup>3</sup> /sec	46°C
Tail gas from H <sub>2</sub> S absorber/38	3.8 Nm <sup>3</sup> /sec	27°C
Tail gas from CO <sub>2</sub> absorber/33	13.6 Nm <sup>3</sup> /sec	29°C

### 4.2.1 Analysis Results

All gas analyses were performed by K-K and the data obtained are shown in Table 14. The raw gas results reflect the average composition from all five operating gasifiers (the stream was sampled at a common line leading to the gas holder) after the gas has been water-washed for particulate removal. A description of the reactions that take place in the raw gas washing stages is as follows:

- NH<sub>3</sub>, HCN, SO<sub>2</sub>, and to a small degree H<sub>2</sub>S and CO<sub>2</sub>, are dissolved in the wash water.
- H<sub>2</sub>S is eventually converted to S<sub>2</sub>O<sub>3</sub><sup>=</sup>, SO<sub>4</sub><sup>=</sup>, and insoluble iron sulfides due to the pH, temperature and flyash content of the water.
- HCN reacts with the sulfur compounds to form SCN<sup>-</sup> and with the iron content of the flyash to form insoluble complexes.
- Additional oxidation reactions occur which are catalyzed by the flyash involving NH<sub>3</sub>, SO<sub>3</sub><sup>=</sup>, S<sub>2</sub>O<sub>3</sub><sup>=</sup>, CN<sup>-</sup>, and SCN<sup>-</sup>.

The main components in the water-washed gas are then H<sub>2</sub>O, CO, CO<sub>2</sub>, H<sub>2</sub>, and N<sub>2</sub>. Data on hydrocarbons contained in the raw gas stream were not obtained, but low concentrations would be expected due to the high temperature of the K-T gasification reaction. Hydrocarbon data from previous tests of the Modderfontein plant under comparable conditions are provided by K-K in Appendix A.

The two tail gas streams from the Rectisol module consist primarily of CO<sub>2</sub> and the nitrogen used for stripping along with some CO and H<sub>2</sub>O, and traces of NH<sub>3</sub> and HCN. During the test period the H<sub>2</sub>S absorber was not operating properly and thus sulfur species data on this tail gas stream were not made available. A design value of <2 ppm total sulfur is quoted by K-K but this cannot be confirmed.

Table 14. RESULTS FROM GAS ANALYSES

Species	Raw Gas after Raw Gas Blower mg/Nm <sup>3</sup>	Tail Gas from H <sub>2</sub> S Absorber mg/Nm <sup>3</sup>	Tail Gas from CO <sub>2</sub> Absorber mg/Nm <sup>3</sup>
H <sub>2</sub> O	5.4 X 10 <sup>4</sup>	5.0 X 10 <sup>3</sup>	5.0 X 10 <sup>3</sup>
H <sub>2</sub> , (dry)	2.3 X 10 <sup>4</sup>	<10	<10
CO, (dry)	6.9 X 10 <sup>5</sup>	2.2 X 10 <sup>4</sup>	3.0 X 10 <sup>3</sup>
CO <sub>2</sub> , (dry)	2.0 X 10 <sup>5</sup>	9.6 X 10 <sup>5</sup>	1.6 X 10 <sup>6</sup>
N <sub>2</sub> /Ar*, (dry)	2.1 X 10 <sup>4</sup>	5.3 X 10 <sup>5</sup>	1.8 X 10 <sup>5</sup>
CH <sub>4</sub> , (dry)	<7 X 10 <sup>2</sup>	<7 X 10 <sup>2</sup>	<7 X 10 <sup>2</sup>
H <sub>2</sub> S, (dry)	6.3 X 10 <sup>3</sup>	+	<1
COS, (dry)	7.4 X 10 <sup>2</sup>	+	<3
CS <sub>2</sub> , (dry)	4.5 X 10 <sup>2</sup>	+	<10
Mercaptans, (dry)	<1	<1	<1
SO <sub>2</sub> , (dry)	14	<3	<3
NH <sub>3</sub> , (dry)	57	39 39	3.0
HCN, (dry)	76	62	8.0
NO <sub>x</sub> (as NO <sub>2</sub> ), (dry)	28	<1	<1

\* By difference

+ Not determined

#### 4.2.2 Source Analysis Model Results

The analytical data were used to perform Source Analysis Model/IA (SAM/IA) calculations (4). This model, developed by the EPA as part of their standardized methodology for interpreting STE results, assesses the potential health and ecological effects of discharge streams based on chemical constituents. In performing SAM/IA evaluation, different evaluation indices may be calculated:

- Discharge Severity (DS)
- Total Discharge Severity (TDS)
- Weighted Discharge Severity (WDS)
- Total Weighted Discharge Severity (TWDS)

The DS is calculated by dividing the measured concentration of a compound or class of compounds by the Discharge Multimedia Environmental Goal (DMEG) values (5). There are two DMEG values for each compound or class. One is based on health effects while the second is based upon ecological effects. When a concentration is known only for a class of compounds, then the evaluation is made using the lowest DMEG value in the class. A DS value greater than one indicates a level of potential concern, while a value less than one indicates little or no potential concern. A total stream discharge severity (TDS) is calculated by summing the DS's for all constituents found in the stream sample. A Total Weighted Discharge Severity is then calculated by multiplying the TDS by the stream flow rate. Because TWDS's incorporate stream flow rate data, they are useful indices for ranking the waste stream from a facility in terms of their potential environmental concern.

The results of calculating DS values for the two waste gas streams, are summarized in Table 15. In the tail gas stream from the H<sub>2</sub>S absorber, CO, HCN and NH<sub>3</sub> are present at levels of potential concern; and in the tail gas from the CO<sub>2</sub> absorber, CO and NH<sub>3</sub> are of potential concern. The TDS values for the H<sub>2</sub>S absorber and CO<sub>2</sub> absorber tail gases are listed in Table 16 along with TWDS values. Unfortunately the lack of sulfur species data for the H<sub>2</sub>S absorber tail gas and the lack of stated detection limits for many other species limits the usefulness of these calculations.

Table 15. SUMMARY OF SAM/IA DS RESULTS  
FOR GAS STREAMS

Gas Species	Discharge Severity (DS)			
	Health-Based		Ecology-Based	
	Tail Gas from H <sub>2</sub> S Removal	Tail Gas from CO <sub>2</sub> Removal	Tail Gas from H <sub>2</sub> S Removal	Tail Gas from CO <sub>2</sub> Removal
CH <sub>4</sub>	NM	NM	NM	NM
CO	5.5 E + 02	7.5 E + 01	1.8 E + 02	2.5 E + 01
COS	NM	ND	NA	NA
CS <sub>2</sub>	NM	ND	NA	NA
RSH	ND	ND	NA	NA
H <sub>2</sub> S	NM	ND	NA	NA
SO <sub>2</sub>	ND	ND	NA	NA
HCN	5.6 E + 00	7.3 E - 01	1.8 E + 00	2.5 E - 01
NH <sub>3</sub>	2.2 E + 00	1.7 E - 01	1.1 E + 02	8.6 E + 00
NO <sub>x</sub>	ND	ND	NA	NA

NM - Not Measured, no data was collected on these species

ND - Not Detected, no measurable amount of this species was found

NA - Not Applicable, no ecology DMEG value for this species.

Table 16. SUMMARY OF SAM/IA TDS AND TWDS RESULTS FOR GAS STREAMS

TDS and TWDS Values	Tail Gas from H <sub>2</sub> S Removal	Tail Gas from CO <sub>2</sub> Removal
Total Discharge Severity (TDS)		
Health-Based	5.6 E + 01	7.6 E + 01
Ecology-Based	2.9 E + 02	3.4 E + 01
Total Weighted Discharge Severity (TWDS)		
Health-Based	2.1 E + 03	1.3 E + 03
Ecology-Based	1.1 E + 03	4.6 E + 02

There are several assumptions implicit in the use of the SAM/IA evaluation technique. The major assumptions include:

- Transport of the components in the waste stream to the external environment occurs without chemical or physical transformation of those components.
- Actual dispersion of a pollutant from a source to a receptor will be equal to, or greater than, the safety factors normally applied.
- The DMEG values developed for each substance are adequate for estimating acute toxicity.
- No synergistic effects occur among the waste stream components.

Because of the uncertainties introduced by these assumptions, the SAM/IA results should be used only as a very qualitative assessment. To more fully determine the potential concern of any stream requires that biological tests as well as chemical tests be evaluated.

#### 4.3 AQUEOUS STREAMS

The five aqueous streams studied were sampled per the schedule shown in Table 1. The engineering data obtained were as follows:

<u>Stream/Stream Number (from Figure 1)</u>	<u>Flowrate</u>	<u>Temperature</u>
Input Water (PSE)/46	215 m <sup>3</sup> /hr	23°C
Input Water (CW)/16	54 m <sup>3</sup> /hr	30°C
Compressor Condensates/40	9.2 m <sup>3</sup> /hr	33°C
Settling Pond Effluent/50	230 m <sup>3</sup> /hr	23°C
Condensate from Rectisol Unit/32	3.9 m <sup>3</sup> /hr	52°C

##### 4.3.1 Analysis Results

The analyses performed on the aqueous process streams can be categorized as follows:

- Level 1
  - Wastewater Tests
  - Organic Survey
  - Inorganic Survey
- Level 2
  - Detailed Organic Characterization
  - Detailed Inorganic Characterization

- Priority Pollutant Screening
  - Volatile Organics
  - Base/Neutral and Acid Organics
  - Trace Elements

Results from each of these categories are presented in the following paragraphs.

#### 4.3.1.1 Level 1 Analysis Results

The wastewater analysis results from K-K, TRW, and McLachlan & Lazar (M&L) are summarized in Tables 17 and 18. The relationship of these streams to each other is best illustrated in Figure 1. The compressor condensate and Rectisol unit water streams are process streams, they are in effect diluted with fresh (PSE) input water before being recycled through the cooling tower. The only waste stream discharged from the plant is the settling pond effluent which appears from the data to be quite similar in composition to the input waters (purified treated sewage and cooling water).

Table 17. RESULTS FROM K-K WASTEWATER ANALYSES

Wastewater Tests/Units of Measure	11/11 Input Water (PSL)	11/19 Input Water (CW)	11/12 Compressor Condensate	11/19 Compressor Condensate	11/12 Settling Pond	11/19 Settling Pond	11/12 Rectisol Unit	11/19 Rectisol Unit
pH	6.8	8.5	8.2	8.0	8.0	9.4	9.1	8.1
Total suspended solids, mg/L	<1	8	<1	12	<1	<1	70	20
Total dissolved solids, mg/L	1580	1460	260	170	1580	1530	1390	1640
Hardness, mg/L as CaCO <sub>3</sub>	452	621	60	46	420	664	691	554
Alkalinity:p-Value, mg/L as CaCO <sub>3</sub>	0	0	0	0	0	44	.0	0
m-Value, mg/L as CaCO <sub>3</sub>	26	167	2990	2690	126	79	78	144
Conductivity, umhos/cm	2300	1900	6000	5500	2100	2100	1800	2000
COD, mg/L	38	118	644	569	353	43	28	1000
NH <sub>3</sub> , mg/L	73	2.4	973	900	38	28	26	49
CN <sup>-</sup> , mg/L	0.2	1.2	7.3	10.5	0.2	0.2	2.8	ND
SCN <sup>-</sup> , mg/L	2.1	2.1	10.9	17.1	1.3	2.2	110	137
H <sub>2</sub> S, mg/L	<1	traces	43.9	53.5	<1	<1	1.1	4.5
S <sub>2</sub> O <sub>3</sub> <sup>2-</sup> , mg/L	<1	<1	4.8	7.8	<1	<1	18.5	16.4
SO <sub>3</sub> <sup>2-</sup> , mg/L	<1	<1	<1	<1	3.6	<1	<1	<1
SO <sub>4</sub> <sup>2-</sup> , mg/L	584	853	56	49	752	706	461	541
PO <sub>4</sub> <sup>3-</sup> , mg/L	10	2.4	2	3	5	0.4	0.1	2.8
Cl <sup>-</sup> , mg/L	185	172	23	13	145	163	153	158
Methanol, mg/L	ND	ND	ND	ND	ND	ND	<0.1	<0.1
Dissolved oxygen, mg/L	ND	ND	ND	ND	ND	ND	ND	ND

ND = Not Determined

Table 18. RESULTS FROM TRW AND M&amp;L WASTEWATER ANALYSES

Sampling Day	Stream Description/Stream Number*	BOD** (mg/L)	COD** (mg/L)	Phenols (mg/L)	TOC (mg/L)	Sulfide as S (mg/L)	Thiocyanates as SCN** (mg/L)	Cyanide as CN** (mg/L)	Nitrates as N** (mg/L)
Nov. 12	Input Water--Purified Treated Sewage/46	5	16	0.005	31	<1.0	9.7	0.16	70
Nov. 19	Input Water--Cooling Water/16	4	24	0.020	16	<1.0	0.8	0.02	2.3
Nov. 12	Combined Condensates from #1--4 Compressors/40	620	670	0.006	130	38	9.5	2.6	<0.1
Nov. 19	Combined Condensates from #1--4 Compressors/40	480	540	0.012	140	46	15.9	5.5	<0.1
Nov. 12	Settling Pond Effluent/50	5	4	<0.001	5.2	<1.0	0.36	0.42	30
Nov. 19	Settling Pond Effluent/50	3	4	0.014	5.4	<1.0	0.8	0.10	37
Nov. 12	Condensate from Rectisol Unit/32	120	350	0.010	15	1.9	145	2.8	0.1
Nov. 19	Condensate from Rectisol Unit/32	1490	2830	0.034	600	5.3	140	1.00	0.6

\* Stream numbers correspond to those shown on Figure 1.

\*\* Results from McLachlan & Lazar

This would indicate that any pollutants contributed by the gasification process are essentially removed in the settling pond.

The wastewater analysis data from testing the same streams a week apart show generally constant conditions with the exception of the Rectisol unit samples. The high BOD, COD, and TOC values for the November 19 Rectisol unit sample correspond to the presence of high molecular weight hydrocarbons. In subsequent organic analyses these were found to be oils/lubricants which were most likely contributed by pumps, valves, and other process equipment.

The level 1 organic survey consists of separate quantitative analyses for volatile (those organics with boiling points in the range of 30°C to 100°C) and nonvolatile (boiling points >100°C) materials. As the data in Table 19 show, the total organic loading (volatiles plus nonvolatiles) is low, and what is there is primarily nonvolatile. Examination of the nonvolatile material by infrared (IR) spectroscopy indicates that the classes of compounds present in all of the samples are primarily saturated hydrocarbons along with some esters. There is also some IR evidence of low levels of aromatic hydrocarbons present in the compressor condensate and Rectisol unit samples.

Examination of the nonvolatile portion of the samples by mass spectroscopy yielded additional information regarding the types of compounds present. The intensity of the mass spectra peaks were used to assign relative concentration levels (100 - major, 10 - minor, 1 - trace) to the compound classes identified. These concentration factors were then applied to the total



Table 19. RESULTS FROM LEVEL 1 ORGANIC SURVEY

Sampling Day	Stream Description/Stream Number*	Volatiles (mg/L)	Non-Volatiles (mg/L)	Total Organics (mg/L)
Nov. 12	Input Water--Purified Treated Sewage/46	0.04	0.68	0.72
Nov. 19	Input Water--Cooling Water/16	<0.01	0.88	0.88
Nov. 12	Combined Condensates from #1--4 Compressors/40	<0.01	4.15	4.15
Nov. 19	Combined Condensates from #1--4 Compressors/40	0.01	3.51	3.52
Nov. 12	Settling Pond Effluent/50	0.03	0.12	0.15
Nov. 19	Settling Pond Effluent/50	0.06	<0.01	0.06
Nov. 12	Condensate from Rectisol Unit/32	0.69	11.5	12.2
Nov. 19	Condensate from Rectisol Unit/32	0.29	55.3	55.6

\* Stream numbers correspond to those shown on Figure 1.

organic loading values in order to arrive at approximate concentrations. The results of these calculations are presented in Table 20. The mass spectra data confirm the IR data indicating the presence of aliphatic hydrocarbons, esters, and traces of aromatics. Traces of phenols, cresols, and alcohols also appear in many of the samples. Significant levels of elemental sulfur ( $S_8$ ) are also seen because of its appreciable solubility in the solvent used for these extractions (methylene chloride). Table 20 also includes a list of the MEG categories that correspond to the classes of compounds identified (reference 5). Comparison of the DMEG values for these categories with the approximate concentrations, revealed that the phenol, cresol, chlorinated phenol, chlorinated cresol, and phthalate ester concentrations consistently exceeded their DMEG values. In the Rectisol unit samples, the aromatic hydrocarbons also exceeded their DMEG values. These compound classes thus became subject to further investigation as is described in the section discussing Level 2 analyses (Section 4.3.1.2).

The Level 1 inorganic survey consisted of a spark source mass spectroscopy (SSMS) analysis of all eight samples. These results are given in Table 21. Only those elements found in at least one sample are reported in this table. There are twenty other elements included in the SSMS scan that were not found in any of the samples (i.e., Tl, Au, Pt, Ir, Os, Re, Hf, Lu, Yb, Tm, Er, Ho, Dy, Tb, Gd, Eu, Sm, Pd, Rh, and Ru). The average detection limit for these elements is 1  $\mu$ g/L. As was also noticed in the

Table 20. DISTRIBUTION OF COMPOUND CLASSES IN AQUEOUS SAMPLES

Sampling Day	Stream Description/Stream Number	Compound Category	MEG Category	Approximate Concentration, $\mu\text{g/L}$
Nov. 12	Input Water--Purified Treated Sewage/46	Primary Alcohols	5A	60
		Esters (phthalates)	8D	560
		Nitro Aromatic Hydrocarbons	17A	60
Nov. 19	Input Water--Cooling Water /16	Esters (phthalates)	8D	880
Nov. 12	Compressor Condensates/40	Chlorinated Cresols	19B	20
		Esters (phthalates)	8D	190
		Fused Polycyclic Hydrocarbons, 2-3 Rings	21A	20
		Esthers	3A	1,960
		Sulfur ( $\text{S}_8$ )	--	1,960
Nov. 19	Compressor Condensates/40	Phenols	18A	30
		Chlorinated Phenols	19A	30
		Esters (phthalates)	8D	310
		Fused Polycyclic Hydrocarbons, 2-3 Rings	21A	30
		Carboxylic Acids	8A	30
		Sulfur ( $\text{S}_8$ )	--	3,080
Nov. 12	Settling Pond Effluent/50	Primary Alcohols	5A	55
		Aliphatic Hydrocarbons, Alkynes	1C	5
		Unsaturated Alkyl Halides	2B	0.5
		Secondary Alcohols	5B	55
		Esters (phthalates)	8D	5
Nov. 19	Settling Pond Effluent/50	Aliphatic Hydrocarbons, Alkenes	1B	0.09
		Unsaturated Alkyl Halides	2B	0.9
		Secondary Alcohols	5B	9
		Ketones	7B	0.09
		Esters (phthalates)	8D	0.09
Nov. 12	Condensate from Rectisol Unit/32	Aliphatic Hydrocarbons, Alkanes	1A	10,190
		Fused Polycyclic Hydrocarbons, 2-3 Rings	21A	100
		Fused Polycyclic Hydrocarbons, 4 Rings	21B	100
		Sulfur ( $\text{S}_8$ )	--	1,020
Nov. 19	Condensate from Rectisol Unit/32	Aliphatic Hydrocarbons, Alkanes	1A	48,130
		Phenols	18A	480
		Fused Polycyclic Hydrocarbons, 2-3 Rings	21A	480
		Fused Polycyclic Hydrocarbons, 4 Rings	21B	480
		Fused Non-Alternate Polycyclics	22B	480
		Esters (phthalates)	8D	480
		Sulfur ( $\text{S}_8$ )	--	4,810

wastewater analyses results, the settling pond effluent is quite similar to the input water. The only trace elements that shown an increase in concentration are Cs, Sr, Ba, Ga, and Mo. Other elements (i.e., Al, Fe and Mn) actually show a significant decrease in the pond effluent versus the input waters.

Table 21. RESULTS FROM LEVEL 1 SSMS ANALYSIS

	Concentration in Process Water Samples (ug/L)							
	11/12 Input Water (PSE)	11/19 Input Water (CW)	11/12 Compressor Condensates	11/19 Compressor Condensates	11/12 Settling Pond	11/19 Settling Pond	11/12 Rectisol Unit	11/19 Rectisol Unit
Lithium	100	6	3	10	< 1	< 1	6	3
Sodium	>2,000	1,000	2,000	>1,000	>1,000	>1,000	>1,000	>2,000
Potassium	>9,000	>6,000	>10,000	>10,000	>6,000	>6,000	>5,000	>10,000
Rubidium	30	40	8	3	100	40	9	6
Cesium	1	ND	<1	ND	6	7	<1	ND
Beryllium	<1	ND	ND	ND	ND	ND	ND	ND
Magnesium	>10,000	>10,000	1,000	2,000	>10,000	>10,000	>10,000	>10,000
Calcium	>10,000	>10,000	>10,000	>10,000	>10,000	>10,000	>10,000	>10,000
Strontium	1,000	200	70	30	8,000	7,000	300	500
Barium	80	400	100	40	200	200	200	200
Boron	30	<1	<1	<1	2	<1	<1	ND
Aluminum	>700	500	5	9	20	200	100	100
Gallium	6	2	<1	3	40	40	<1	<1
Silicon	6,000	2,000	300	100	400	2,000	1,000	2,000
Germanium	7	3	5	7	1	<1	<1	2
Tin	<2	ND	2	<1	1	1	4	ND
Lead	30	400	20	30	4	2	20	10
Phosphorus	8,000	2,000	70	70	80	80	700	1,000
Arsenic	70	9	2	4	8	9	20	10
Antimony	10	ND	<2	10	4	2	<1	ND
Bismuth	ND	ND	2	ND	ND	ND	ND	ND
Sulfur	>4,000	>3,000	>6,000	>2,000	>3,000	>3,000	>2,000	>4,000
Selenium	20	3	500	1,000	2	6	50	40
Tellurium	ND	ND	3	3	ND	8	ND	ND
Fluorine	>10,000	≈700	≈30	≈3,000	≈700	≈2,000	≈400	≈3,000
Chlorine	300	300	100	60	70	300	200	300
Bromine	100	40	80	300	100	300	30	60
Iodine	30	9	4	8	20	20	8	9
Scandium	<1	<1	<1	<1	<1	<1	<1	<1
Yttrium	6	<3	<1	1	<1	<1	<1	<1
Titanium	400	100	30	200	60	500	100	200
Zirconium	5	1	3	10	1	1	<1	<1
Vanadium	5	20	2	<1	9	10	3	5
Niobium	1	2	2	5	6	1	2	<1
Tantalum	ND	ND	2	ND	ND	ND	ND	ND
Chromium	7	5	5	5	<1	2	3	7
Molybdenum	30	5	30	20	50	100	40	20
Tungsten	ND	ND	ND	ND	10	10	ND	ND
Manganese	900	20	10	9	200	200	50	80
Iron	200	1,000	500	1,000	50	100	>10,000	2,000
Cobalt	20	<1	3	<1	4	3	1	<1
Nickel	100	8	4	7	8	20	200	100
Copper	100	300	10	10	7	6	50	100
Silver	ND	ND	<2	<1	ND	ND	ND	ND
Zinc	2	*	600	1,000	30	30	6,000	5,000
Cadmium	ND	ND	3	7	8	8	1	ND
Lanthanum	8	1	ND	1	ND	ND	ND	ND
Cerium	20	<1	ND	1	ND	<1	<1	ND
Praseodymium	2	ND	ND	ND	ND	ND	ND	ND
Neodymium	2	ND	ND	ND	ND	ND	ND	ND
Thorium	<6	<4	<8	<3	<4	<4	<3	<6
Uranium	<5	20	<7	<3	<3	<3	6	6

\* Zinc could not be quantitated in this sample due to a heterogeneity problem with the electrodes.

ND = Not detected (average detection limit is 1ug/L)

As with the organic data, the data from the inorganic survey were compared to the DMEG values for each species. This resulted in finding that Cd, Cu, Fe, Mn, Ni, P (as  $\text{PO}_4^{=}$ ), Pb, S, Se, and Zn exceeded their DMEG values in most of the samples. These elements thus became subject to further investigation as is described in the section discussing Level 2 analysis (Section 4.3.1.2).

#### 4.3.1.2 Level 2 Analysis Results

As is mentioned in the preceeding paragraphs, the Level 1 data were compared to the EPA's Discharge Multimedia Environmental Goals (DMEGs) in order to determine which species were present at potential levels of concern and were thus candidates for further investigation. From the organic survey, phenols, cresols, chlorinated phenols and cresols, phthalate esters, and aromatic hydrocarbons were determined to be of concern (i.e., present at concentrations greater than their DMEG values). The Level 2 analytical needs for these materials were thus to identify the specific compounds present and more accurately quantify the concentrations. From the inorganic survey the elements Cd, Cu, Fe, Mn, Ni, P, Pb, S, Se and Zn were determined to be present at levels of concern. The Level 2 needs for trace elements required more accurate quantitation, and for major constituents such as S and P included speciation of the various anions. The best approach to satisfying these additional investigation needs, within the overall constraints of the project, was evaluated with the following results:

- The identification and quantitation of phenols, cresols, chlorinated phenols and cresols, and phthalate esters would be accomplished as part of the priority pollutant screening.
- The identification and quantitation of aromatic hydrocarbons would be addressed as a separate, specific analysis.
- The quantitation of Cd, Cu, Hg, Ni, Pb, Se and Zn would be accomplished as part of the priority pollutant screening.
- The quantitation of Fe and Mn would be addressed as a separate, specific analysis.
- Sulfur speciation (i.e.,  $\text{SCN}^-$ ,  $\text{H}_2\text{S}$ ,  $\text{S}_2\text{O}_3^{=}$ ,  $\text{SO}_4^{=}$ ) had been adequately addressed as part of the wastewater analysis.
- Phosphorus speciation could not be addressed because adequate samples had not been collected and stabilized for that purpose.

Thus, of the Level 2 data needs identified, most are addressed and reported in the priority pollutant screening results (Section 4.3.1.3) and wastewater analysis results (Section 4.3.1.1). Aromatic hydrocarbons, iron, and manganese required additional specific analyses, the results of which are reported in the following paragraphs. Determination of phosphorus species could not be accomplished due to the lack of appropriately stabilized samples. Hopefully the need for phosphorus species data can be addressed in a future source test effort.

The high performance liquid chromatograph (HPLC) is a very useful analytical tool for polynuclear aromatic hydrocarbon (PAH) compounds. The technique separates by functionality thus allowing the aromatics to be separated from the large quantities of aliphatics present. HPLC also is not limited, as gas chromatography is, by the volatility of the compounds to be analyzed. Even large PAH compounds such as decacyclene (MW 450) can be determined.

The two Rectisol unit water samples were analyzed by HPLC, yielding the chromatograms shown in Figures 4 and 5. The composition of the two samples, even though they were obtained a week apart, is essentially the same. The trace at the bottom of each figure is the response to a fluorescence detector (which is very specific for PAHs) and the trace at the top is the response to an ultra-violet detector. The ratio of the response to the two detectors along with the retention times was the means for determining the identity of the compounds present. Those compounds which were positively identified are indicated in the legends of Figures 4 and 5. The unknowns did not correspond to any of the standards available (see Table 11) and thus could not be positively identified. In order to obtain some indication of what these compounds might be, the HPLC column eluent was collected and analyzed by gas chromatography/mass spectroscopy (GC/MS). The GC/MS cannot identify different isomers but did indicate that the following types of compounds are possible present:

- Compound "F" - a methylbenzofluorene
- Compounds "G" and "H" - methylbenzanthracenes
- Compounds "J" and "K" - unknown.

It should be noted that the very toxic compound, benzo(a)pyrene, is one of the standards used thus compounds "J" and "K" are clearly some other isomer.

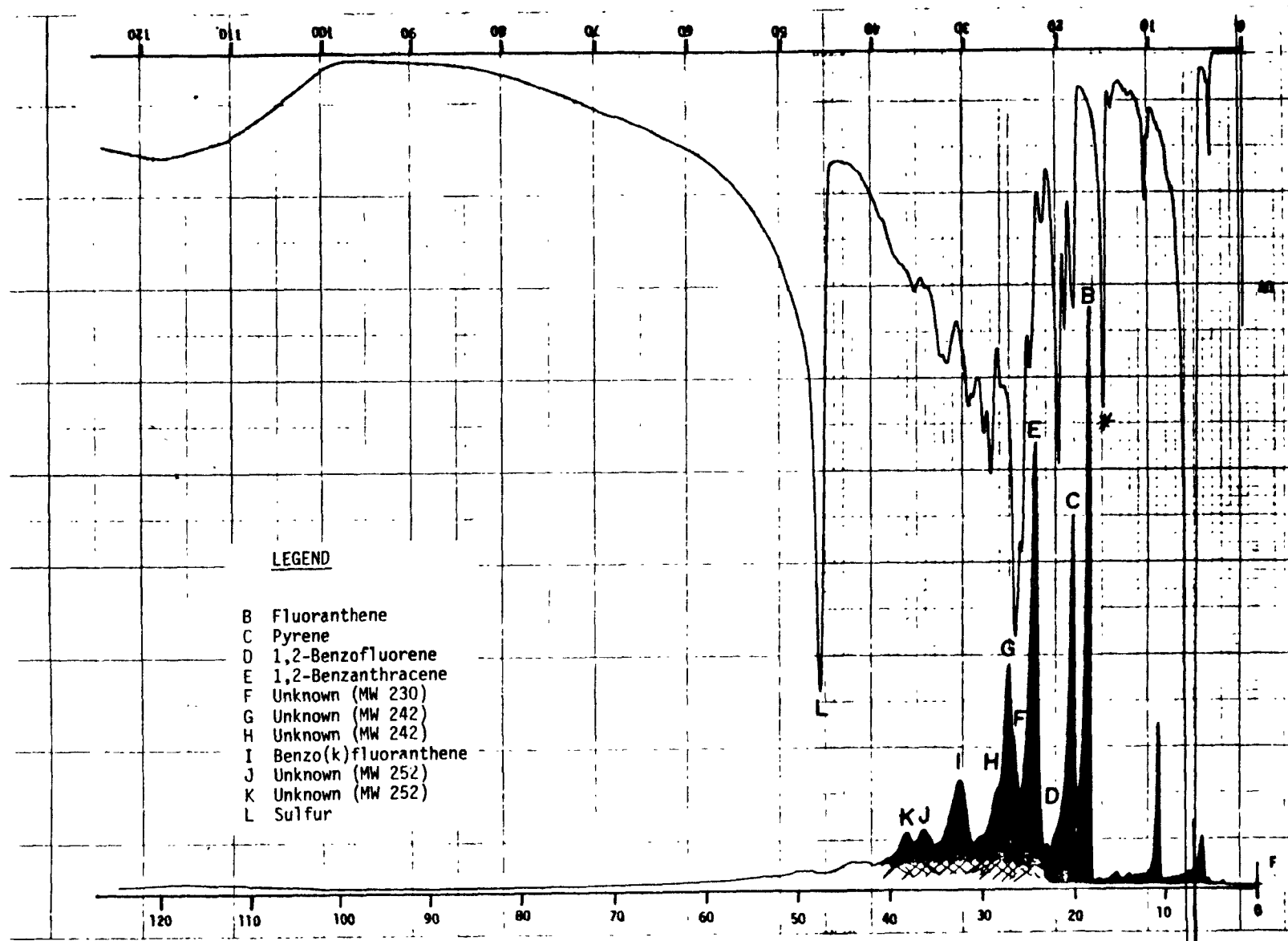


Figure 4. HPLC Chromatogram of November 12, 1979 Rectisol Unit Sample

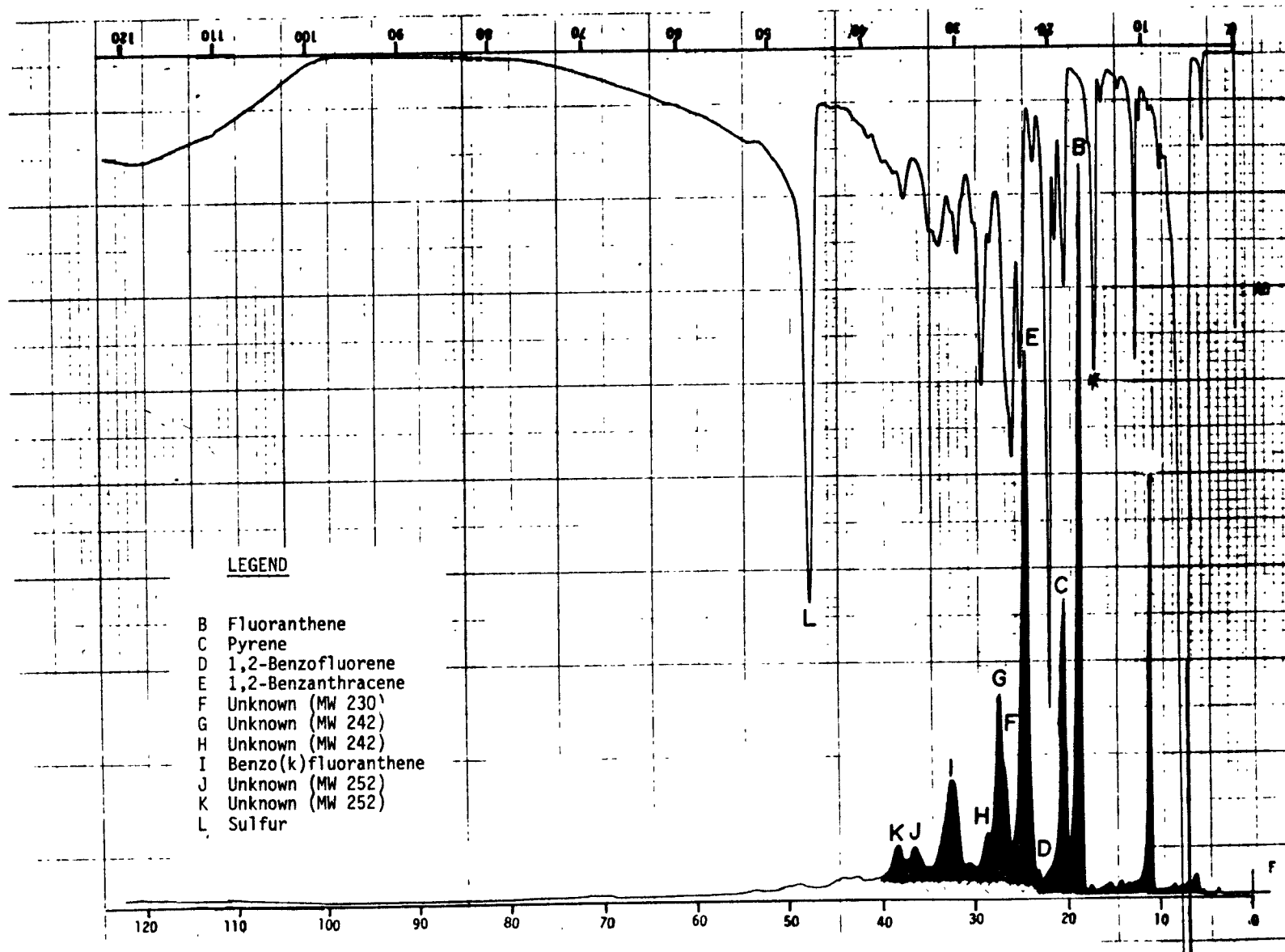


Figure 5. HPLC Chromatogram of November 19, 1979 Rectisol Unit Sample

The compounds that were positively identified were quantitated, yielding the results shown in Table 22. Those compounds which overlap with the priority pollutant screening (i.e., fluoranthene and pyrene) are more accurately quantitated by the HPLC technique. The priority pollutant screening also identified a four-ringed compound as chrysene which in the HPLC analysis was determined to be 1,2-benzanthracene (also four-ringed).

The DMEGs for the compounds identified range from 670 µg/L to 24,000 µg/L for the health-based values and an ecology-based value of 100 µg/L. Thus the levels measured would not be considered potentially toxic. However, direct biological tests could be performed to confirm this.

Table 22. RESULTS FROM LEVEL 2 ANALYSIS OF AROMATIC HYDROCARBONS

Compounds Identified	Nov. 12, 1979 Rectisol Unit Sample	Nov. 19, 1979 Rectisol Unit Sample
Fluoranthene	24 µg/L	17 µg/L
Pyrene	32 µg/L	25 µg/L
1,2-Benzofluorene	15 µg/L	15 µg/L
1,2-Benzanthracene	23 µg/L	16 µg/L
Benzo(k)fluoranthene	2 µg/L	2 µg/L

The Level 2 inorganic analyses to quantitate the elements Fe and Mn were quite straightforward compared to the HPLC analysis. Routine atomic adsorption techniques were used, yielding the results shown in Table 23. These data, along with the priority pollutant screening data for Ag, Tl, Sb, As, Se, Zn, Pb, Cd, Ni, Be, Cu, Cr and Hg; were used instead of the less accurate Level 1 SSMS survey data in computing the SAM/IA results discussed in Section 4.3.2.



Table 23. RESULTS FROM LEVEL 2 QUANTITATION OF Fe AND Mn

Sampling Day	Stream Description/Stream Number*	Concentrations, ppb	
		Fe	Mn
Nov. 12	Input Water - Purified Sewage Effluent/46	<100	1250
Nov. 19	Input Water - Cooling Water/16	700	<50
Nov. 12	Combined Condensates from #1 - #4 Compressors/40	500	<25
Nov. 19	Combined Condensates from #1 - #4 Compressors/40	1800	<25
Nov. 12	Settling Pond Effluent/50	175	850
Nov. 19	Settling Pond Effluent/50	100	580
Nov. 12	Condensate from Rectisol Unit/32	4600	50
Nov. 19	Condensate from Rectisol Unit/32	3400	50

\* Stream numbers correspond to those shown in Figure 1.

#### 4.3.1.3 Priority Pollutant Screening Analysis

Very few of the 116 organic priority pollutant compounds were found, as shown in Table 24. Those that were present were mostly at very low concentrations. The level of concern determined by the EPA's Effluent Guidelines is 10  $\mu\text{g/L}$ . The fact that few compounds were detected and that those which are present are mostly below this level of concern is evidence of the relatively acceptable composition of the streams tested, particularly the settling pond effluent. The quality of the priority pollutant screening data is believed to be quite satisfactory. The only exceptions to this are the two Rectisol Unit samples. These samples both contain large amounts of normal and branched saturated hydrocarbons in a molecular weight distribution ranging from  $\text{C}_{14}$  up through and exceeding  $\text{C}_{30}$ . These compounds have been quantified as part of the Level 1 analysis previously described. However, because of the very large amounts of aliphatic hydrocarbons compared to the total organic content of the Rectisol unit samples, it is possible that other nonvolatile priority pollutants may be present at low  $\mu\text{g/L}$  levels in these two samples but are completely masked.

Table 24. RESULTS FROM ORGANIC PRIORITY POLLUTANT SCREENING

Sampling Day	Stream Description/Stream Number*	Priority Pollutant Compounds Found					
		Base/Neutral Fraction		Acid Fraction		Volatiles	
		Compound	µg/L	Compound	µg/L	Compound	µg/L
Nov. 12	Input Water - Purified Sewage Effluent/46	Nitrobenzene	T	None Detected		None Detected	
		1,2,4-Trichlorobenzene	T				
		Isophorone	T				
		Bis (2-Ethylhexyl)phthalate	T				
		Di-n-octylphthalate	T				
Nov. 19	Input Water - Cooling Water/16	Butylbenzylphthalate	T	None Detected		Chloroform	T
Nov. 12	Combined Condensates from #1 - 4 Compressors/40	Naphthalene	T	4-Chloro-m-Cresol	2.3	None Detected	
Nov. 19	Combined Condensates from #1 - 4 Compressors/40	Naphthalene	T	Phenol	T	Chloromethane	7.8
		Diethylphthalate	T				
		Di-n-butylphthalate	6.0				
		Butylbenzylphthalate	T				
Nov. 12	Settling Pond Effluent/50	None Detected		None Detected		None Detected	
Nov. 19	Settling Pond Effluent/50	Butylbenzylphthalate	T	None Detected		Chloroform	T
Nov. 12	Condensate from Rectisol Unit/32	Naphthalene	T	None Detected		Chloroform	T
		Fluorene	T				
		Anthracene plus phenanthrene	T				
		Fluoranthene	6.3				
		Pyrene	25				
		Butylbenzylphthalate	T				
Nov. 19	Condensate from Rectisol Unit/32	Acenaphthalene	T	Phenol	T	Chloroform	T
		Dimethylphthalate	T	2,4-Dimethylphenol	T		
		Fluorene	1.0				
		Diethylphthalate	T				
		Anthracene plus phenanthrene	4.6				
		Fluoranthene	19				
		Pyrene	97				
		Chrysene	34				

T = Trace (&lt;1µg/L)

\* Stream numbers correspond to those shown in Figure 1.

The priority pollutant metals screening involves the analysis of 13 elements each of which has its own level of concern. These elements and the corresponding levels of concern which have been defined by the EPA are: Ag - 5 ppb, Tl - 50 ppb, Sb - 100 ppb, As - 25 ppb, Se - 10 ppb, Zn - 1000 ppb, Pb - 25 ppb, Cd - 5 ppb, Ni - 500 ppb, Be - 50 ppb, Cu - 20 ppb, Cr - 25 ppb, and Hg - 1 ppb. The results obtained from atomic adsorption and emission spectroscopy analyses for these 13 elements are presented in Table 25. The data show that the process waters (compressor condensate and Rectisol unit samples) frequently exceed the levels of concern particularly for Se, Zn, Cu and Hg. However, as was noticed in the Level 1 SSMS inorganic survey, the discharged stream (settling pond effluent) is relatively clean compared to both the process streams and the input water (purified sewage effluent).

#### 4.3.2 Source Analysis Model Results

The analytical data were used to perform Source Analysis Model/IA (reference 4) calculations. This model, developed by the EPA as part of their standardized methodology for interpreting STE results, assesses the potential health and ecological effects of discharge streams. It uses concentrations of chemical constituents to calculate a Discharge Severity (DS), Total Discharge Severity (TDS), and Total Weighted Discharge Severity (TWDS). The method for calculating these indices and the assumptions contained in the model are described in Section 4.2.2 and will not be repeated here.

The results of calculating TDS and TWDS values for the aqueous streams are summarized in Table 26. It should be noted that the only true discharge stream is the settling pond effluent. The input water is provided as a background value. The process streams (compressor condensate and diluted Rectisol condensate) were also evaluated as an indication of the relative potential concern of the streams produced.

The fact that the health based values for the aqueous input and discharge streams reflect a potential concern is due mainly to Mn and Fe and to a lesser extent P. The ecology-based values are almost entirely due to P. The ecology DMEG value for P and its various anions as a class of compounds is extremely low (0.5 µg/L) and thus easily becomes the most significant value obtained in SAM/IA calculations. However ecology-based

Table 25. RESULTS FROM INORGANIC PRIORITY POLLUTANT SCREENING

Samples	Trace Element Concentrations, in parts per billion												
	Silver	Thallium	Antimony	Arsenic	Selenium	Zinc	Lead	Cadmium	Nickel	Beryllium	Copper	Chromium	Mercury
11/12 Input Water (PSE)	<1	<5	10	33	<2	660	50	1.3	180	0.6	78	<5	0.5
11/19 Input Water (CW)	<1	<5	<3	<5	<2	3500	28	<0.5	<10	<0.5	43	7	<0.2
11/12 Compressor Condensates	<1	<5	<3	<5	3500	310	32	0.6	<10	<0.5	10	5	360
11/19 Compressor Condensates	<1	<5	<3	<5	3500	230	5	<0.5	<10	<0.5	52	6	140
11/12 Settling Pond Effluent	<1	<5	5	12	2	<100	<5	<0.5	20	3.4	<5	7	<0.2
11/19 Settling Pond Effluent	<1	<5	<3	6	3	<100	<5	2.2	<10	<0.5	10	<5	<0.2
11/12 Rectisol Unit Condensate	2	<5	4	10	15	2400	19	<0.5	220	<0.5	110	7	33
11/19 Rectisol Unit Condensate	<1	<5	<3	12	36	2700	7	<0.5	160	<0.5	71	6	13

Table 26. SUMMARY OF SAM/IA TDS AND TWDS RESULTS FOR AQUEOUS STREAMS

Sampling Day	Stream Description/Stream Number *	Total Discharge Severity (TDS)		Total Weighted Discharge Severity (TWDS)	
		Health-Based	Ecology-Based	Health-Based	Ecology-Based
Nov. 12	Input Water - Purified Sewage Effluent <sup>+</sup> /46	9.8 E + 00	1.6 E + 04	5.9 E + 02	9.6 E + 05
Nov. 19	Input Water - Cooling Water <sup>+</sup> /16	6.7 E + 00	4.2 E + 03	1.0 E + 02	6.3 E + 04
Nov. 12	Combined Condensates from #1 - 4 Compressors/40	1.1 E + 02	4.3 E + 02	3.7 E + 02	1.1 E + 03
Nov. 19	Combined Condensates from #1 - 4 Compressors/40	6.1 E + 01	5.1 E + 02	7.0 E + 01	5.2 E + 02
Nov. 12	Settling Pond Effluent/50	6.9 E + 00	1.9 E + 02	4.4 E + 02	1.2 E + 04
Nov. 19	Settling Pond Effluent/50	5.2 E + 00	1.8 E + 02	3.3 E + 02	1.2 E + 04
Nov. 12	Condensate from Rectisol Unit/32	3.6 E + 01	1.6 E + 03	4.0 E + 01	1.8 E + 03
Nov. 19	Condensate from Rectisol Unit/32	3.6 E + 01	2.4 E + 03	3.6 E + 01	2.4 E + 03

\* Stream numbers correspond to those shown in Figure 1.

+ These streams are included as background values for comparison to the other streams.

Discharge Severity values >1 were also obtained for Cd, Cu, Mn, Ni, Pb, S, Zn and phthalate esters in the input water streams and Cd, Mn, Ni, and S in the settling pond discharge stream. The reduction in both TDS and TWDS values for the effluent versus the input water appears to be due to a decrease in the concentrations of the phthalate esters, P, Cu, Pb, and Zn. These and other constituents as well appear to be lost to the settling pond sludge.

For the other streams evaluated, their TDS values resulted primarily from the following constituents:

- Compressor Condensates - phthalate esters, phenols, cresols, Cd, Fe, Hg, P, S, Se, and Zn.
- Rectisol Unit Water - phthalate esters, phenols, aromatic hydrocarbons, Cd, Cu, Fe, Hg, Ni, P, S, Se, and Zn.

The TWDS values for the compressor condensates and Rectisol unit samples turn out to be relatively low because of the small flow rates for these two streams, approximately 9 and 4 m<sup>3</sup>/hr, respectively. Whereas the flow rates for the input and effluent waters is over 200 m<sup>3</sup>/hr.



## 5. REFERENCES

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## APPENDIX A

### KRUPP-KOPPERS REPORT

# KRUPP-KOPPERS

Environmental Assessment  
of the Koppers-Totzek Process

for

Defense and Space Systems Group  
of TRW Inc.,  
Redondo Beach, California, USA

Essen, February 1980

Subcontract No. : J 01440 DE 9-M  
Project No. : 4540

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Environmental Assessment of the  
Koppers-Totzek Process

Investigations Performed for TRW Inc. at the  
Coal-Based Ammonia Plant of AECI Limited,  
Modderfontein, South Africa

Client: Defense and Space Systems Group of TRW Inc.,  
Redondo Beach, California, USA  
TRW Subcontract No. J 01440 DE9 - M  
KK Project No. 4540  
Project Manager: Mr. Kress  
Reported by Dr. B. Firnhaber

Contents:

13 pages  
3 tables  
5 figures

DEP.: FE-I	NAME Dr. Firnhaber	DATE Febr. 1980
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Summary

According to the Professional Services Agreement of October 2, 1979 between TRW Inc. and Krupp-Koppers GmbH, Krupp-Koppers carried out investigations, comprising measurements and analytical work on waste and by-product streams, on the coal-based ammonia plant of AECI Limited in Modderfontein, South Africa. The plant incorporates a Koppers-Totzek coal gasification plant and a Rectisol gas purification unit. The aim of the investigations was the assessment of the environmental impact of the KT process.

The investigations on the plant site were carried out in the period of November 7 to 29, 1979. The plant operated during the measurements at almost 100 % design capacity of about 103 000 m<sup>3</sup>/h dry raw synthesis gas.

The analyses of the waste streams document the low environmental impact of the KT process. Alternate processing feasibilities for further reduction of environmental pollution are discussed.

Note

The data and information reported hereafter shall only be used in accordance with the terms and conditions of the Professional Services Agreement of October 2, 1979 between TRW Inc. and Krupp-Koppers GmbH and the Letter Secrecy Agreement referred to in Clause 16 of aforementioned agreement.

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<p>Contents</p> <p>-----</p> <ol style="list-style-type: none"> <li>1. Introduction</li> <li>2. The Koppers-Totzek Process</li> <li>3. The Ammonia Plant of AECI in Modderfontein</li> <li>4. Experimental Procedure</li> <li>5. Results</li> <li>6. Discussion of the Results</li> </ol> <p>Table 1: Feed Coal Analysis</p> <p>Table 2: Gas Analyses</p> <p>Table 3: Water Analyses</p> <p>Fig. 1: No. 4 Ammonia Plant - Process Scheme</p> <p>Fig. 2: Coal Preparation</p> <p>Fig. 3: Koppers-Totzek Gasification</p> <p>Fig. 4: Gas Treatment</p> <p>Fig. 5: Wash Water System - Flow Scheme</p> <p>Fig. 6: View of the Gasification Plant</p> <p>Fig. 7: View of the Gas Treatment Unit</p>			
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1. Introduction

In 1977 the Environmental Engineering Division of TRW Inc. signed an agreement with the United States Environmental Protection Agency, EPA, (Contract No. 68-o2-2635) concerning the environmental assessment of high BTU coal gasification. It is the aim of this investigation program to quantify effluent streams obtained in the operation of commercial-scale coal gasification plants, to identify possible treatment or control technology, and to assess the environmental impact on future construction sites to be evaluated for large-scale application of the coal gasification technology.

The Koppers-Totzek process is one of nine coal gasification processes to be investigated in this program.

In Subcontract No. T 01 440 DE9-M signed on Oct. 2, 1979, it was agreed between TRW Defense and Space Systems Group of TRW Inc., Redondo Beach, California, USA, and Krupp-Koppers GmbH, Essen, Germany, that Krupp-Koppers carried out measurements in the coal gasification plant in Modderfontein, South Africa, which were to supply data for assessing the environmental impact of a commercial-scale Koppers-Totzek plant.

The investigations to be performed in the program have been carried out by Krupp-Koppers personnel in the No. 4 Ammonia Plant of AECI Limited in Modderfontein, South Africa in the period of November 7 to 29, 1979. An employee of TRW Inc. was present in South Africa during the investigation period to receive the agreed on coal and water samples and for the necessary liaison between the partners.

After a description of the Koppers-Totzek technology in general and the ammonia plant Modderfontein in particular,

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the investigations and their results are reported herein. Finally, discussion of alternate processing steps and their effect on the environment is added.

2. The Koppers-Totzek Process

In 1936/1942 Friedrich Totzek and his coworkers at Heinrich Koppers GmbH in Germany, now Krupp-Koppers GmbH, developed a new coal gasification principle where pulverized coal is gasified in an entrained-bed reactor, using oxygen and steam as gasification media. The target of the development was a process with virtually no restrictions to coal properties, a resulting synthesis gas with CO and H<sub>2</sub> as main components, and practically no environmental pollution.

The principle of entrained bed gasification according to the Koppers-Totzek process operates autothermally, i.e. without supplying outside heat. The reactants, coal, oxygen, and steam, enter the reactor in certain proportions via opposite burners located at the heads of the gasifiers cones.

The coal dust has a particle size, that is predominantly smaller than 0.1 mm. The permitted portion of larger particles in case of bituminous coal amounts to about 10 %, in case of lignite to 15 % to 20 %. In a preceding coal preparation unit the moisture content depending on the type of coal is reduced to approximately 1 to 2 % in the case of bituminous coal and 8 to 10 % in the case of lignite. Generally, oxygen has a purity of about 90 %. Coal and gasification agents enter the gasifier in cocurrent flow. The coal is gasified within about 1 second. The temperatures in the core of the flame amounts to approximately 2000 °C. Under these conditions the heterogenous reactions between carbon, oxygen and steam occur which are characteristic of coal gasification.

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<p>The raw gas leaves the gasifier with a composition which is determined by the homogeneous water gas equilibrium. Reactions as the formation of methane are of minor importance because of the high reactor temperatures of 1400 to 1600 °C.</p> <p>The sulfur contained in the coal as organic and inorganic compounds for the most part is converted to H<sub>2</sub>S and COS, at a ratio of about 9/1, and appears as such in the raw gas. Other reactions are possible which result in trace amounts of CS<sub>2</sub> and SO<sub>2</sub>.</p> <p>The nitrogen contained in the coal and in the oxygen used as gasification medium will react under the prevailing conditions to form traces of NH<sub>3</sub>, HCN and NO.</p> <p>The raw gas of the KT process does not contain higher molecular weight organic compounds. They are gasified completely under the prevailing thermal conditions.</p> <p>The raw gas produced from different solid fuels is characterized by 80 to 88 Vol. % of CO and H<sub>2</sub> and a CO/H<sub>2</sub>-ratio of 2/1 to 2.5/1. Carbon gasification degrees exceeding 98 % have been reached, depending on the typ of coal used in the process. The KT raw gas has a calorific value between 10.8 MJ/m<sub>n</sub><sup>3</sup> and 11,8 MJ/m<sub>n</sub><sup>3</sup> and based on the heating value is between blast furnace and coke oven gas.</p> <p>The high temperatures prevailing in the gasification reactor requires suitable refractory lining to protect the reactor walls, since at these temperatures the coal ash is liquified. The wall structure must be designed in a way that the liquid slag does not attack the lining. The liquid slag running down the gasifier walls is cooled in a water bath and granulated.</p> <p>KT plants are built with gasifiers with 2 or 4 burner heads, their capacities amount up to 25 000 and 50 000 m<sub>n</sub><sup>3</sup>/h raw gas, respectively. A further increase in the output is basically possible.</p>			
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<p>Independently of the eventual utilisation of the produced raw gas, the KT process includes the stages of coal preparation and mechanical cleaning of the raw gas, the treatment of the wash water used for gas cooling and cleaning, as well as package units for the production of pure oxygen by air fractionation.</p> <p>The KT process operating under normal pressure has shown its efficiency in numerous large-scale plants totalling more than 50 gasifiers.</p> <p>3. <u>The Ammonia Plant of AECI in Modderfontein</u></p> <p>In 1972 AECI Limited, South Africa, ordered a grass-roots ammonia plant, based on the Koppers-Totzek coal gasification process, to produce 1000 t/day ammonia. The plant was commissioned in 1974.</p> <p>The highest daily production achieved so far was 1060 metric tons ammonia. The plant operated in 1978 with an on-stream time of 81 % and is expected to reach the figure of 86 % - quoted for typical gas and naphtha based ammonia plants - in the following years.</p> <p>The basic plant layout is described with the help of the block diagram shown in Fig. 1.<sup>x)</sup> A single stream air separation plant supplies oxygen at 98 % purity to six two-headed Koppers-Totzek gasifiers. Twin ring-and-ball type mills are used to pulverise the sub-bituminous coal feed to a nominal size of 90 % less than 90 µm. The oxygen is pre-mixed with steam and the mixture entrains coal dust from screw feeders into the gasifiers. The gasifiers operate essentially at atmospheric pressure and a gas outlet temperature of about 1600°C. A major part of the coal ash is entrained in the gas leaving the gasifiers, and is subsequently removed by scrubbing with water and passing through electrostatic precipitators.</p> <p>x) Ref.: A.D. Engelbrecht, L.J. Partridge (AECI Limited), Paper presented at the Ammonia from Coal Symposium, May 8-10, 1979 Muscle Shoals, USA</p>			
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<p>The gas is compressed to 30 bar in twin-stream raw gas compressors and desulphurised (to less than 1 ppm H<sub>2</sub>S and COS) in a methanol scrubbing column at about -38°C. A final stage of compression raises the gas pressure to 50 bar after which it is subjected to a water-gas shift reaction in a converter unit with a conventional promoted iron oxide catalyst. Steam for the shift reaction is supplied from waste-heat-boilers on the gasifiers. The carbon monoxide and steam are converted to carbon dioxide and hydrogen and the residual (dry basis) CO content is about 3 Vol. %.</p> <p>Carbon dioxide is removed (to less than 10 ppm) from the gas by absorption in methanol at about -58°C. CO<sub>2</sub> is recovered from the methanol in a stripping column and a proportion thereof is used in urea manufacture in another plant. Sulphur compounds absorbed from the gas (H<sub>2</sub>S and COS) are removed from the circulating methanol stream in a stripping column and produce a byproduct stream containing about 60 % H<sub>2</sub>S and COS. The gas purification process using methanol is termed the Rectisol process.</p> <p>The final traces of CO<sub>2</sub> are removed by adsorption on molecular sieves and the gas then passes to a column for scrubbing with liquid nitrogen at -190°C. The gas purification process results in a synthesis gas of high purity, such that no voluntary purge of the synthesis loop is required to avoid buildup of inerts.</p> <p>A conventional ammonia synthesis loop, operating at 220 bar, is employed.</p> <p>The synthesis gas compressor, refrigeration compressor and nitrogen compressor are single-stream centrifugal units while there are two each centrifugal air compressors and raw gas compressors in parallel. All the major machines are driven by steam turbines (except one motor-driven air compressor) and the motive steam is supplied from two large spreader-stoker fired boilers.</p>				
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<p>The individual process stages identifying the plant effluent streams are shown diagrammatically in Figs. 2-5.</p> <p>In the coal preparation unit, Fig. 2, raw coal is milled and simultaneously dried using flue gas from the steam boilers. The coal dust is conveyed pneumatically to the feed bunkers of the gasification unit using impure nitrogen. Both, the flue gas used for coal drying and the coal conveying gas returned to the coal preparation unit are dedusted in an electrostatic precipitator before venting via a chimney.</p> <p>The gasification unit, Fig. 3, comprises the gasifiers proper as well as raw gas cooling and dedusting. Part of the coal ash leaves the gasifier after quenching in water as granulated slag which can be deposited or used as a road construction material. Unconverted coal and a major part of the coal ash is entrained with the raw gas which passes after partial quench through a waste heat boiler to final cooling and dedusting. Cooling and coarse dedusting is reached in the cooling washer. The following disintegrator brings the dust content of the raw gas down to a level which allows the use of blowers for conveying the gas to the gas holder. Compressor-grade dedusting is obtained in electrostatic precipitators.</p> <p>Fig. 3 also shows the recycle of the wash water via settling tank (clarifier) and cooling tower. The water purge containing the flyash (slurry) is pumped to a settling pond.</p> <p>The gas treatment for the production of ammonia synthesis gas is shown in Fig. 4. The cooled and dedusted raw gas is compressed to 30 bar, water-washed for HCN removal, desulfurized, and compressed in a final stage to 50 bar. After CO shift conversion, CO<sub>2</sub> is removed in the second Rectisol stage. Final purification of the hydrogen and admixture of the stoichiometric amount of nitrogen is obtained in the liquid nitrogen wash.</p>			
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<p>The water streams, compressor condensate, water from the HCN wash, and condensate from the Rectisol unit, not identified in Fig. 4, are fed to the common wash water system of the gasification plant. A water purge from the CO shift conversion is used as quench water in gasification.</p> <p>In the Rectisol unit, a concentrated H<sub>2</sub>S stream for further processing and a pure CO<sub>2</sub> stream are obtained. Tail gases from desulfurization and CO<sub>2</sub> wash are vented. Tail gas from the liquid nitrogen wash is burned in the steam boilers.</p> <p>Fig. 5, finally, shows a block diagram of the entire wash water system. It is fed with approximately 55 m<sup>3</sup>/h of cooling water and 145 m<sup>3</sup>/h of an AECI produced water, called "PSE". An additional 70 m<sup>3</sup>/h PSE-water is used for conveying ash from the boiler houses to the settling pond. The only effluent water stream is the run-off from the settling pond, comprising approximately 230 m<sup>3</sup>/h.</p> <p>4. <u>Experimental Procedure</u></p> <p>To accomplish the agreed investigation program, four Krupp-Koppers employees travelled to South Africa and carried out the necessary data recording, sampling, and analytical work in the time period between November 7 and 29, 1979. They were actively supported by AECI laboratory and operating personnel whose friendly cooperation is greatly appreciated.</p> <p>The specified work agreement necessitated the compilation of complete material balances over gasification and gas purification units the results of which - due to the proprietary nature - cannot be included in this report. For those measurements and analyses which are not performed during normal operation of the plant, additional installations and analytical equipment had to be provided.</p>			
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Although the entire work was performed on several days spread over a two-week period, particular care has been taken that all data compilation, sampling, and analyses were carried out under identical plant performance conditions. Thus, the data obtained on different days for different stages of the plant can be correlated for complete material balance.

During the investigation period one gasifiers was out of commission. Practically full design capacity was obtained with the remaining five gasifiers. All sampling and data collecting was done at production rates of between 102 000 and 104 000 m<sup>3</sup>/h dry raw gas.

The sampling of the process waters for TRW was carried out on Nov. 12 and on Nov. 19, 1979, between 900 and 1400 hours. For each specified water stream, 11 sample containers supplied by TRW were filled and then handed over to Mr. John F. Clausen, the TRW representative present in Johannesburg. Parallel samples were analyzed by Krupp-Koppers personnel.

The following water samples were taken and handed over to TRW:

<u>on Nov. 12, 1979</u>	TRW-Designation
- Fresh Water Input (PSE-water)	- PW -
- Condensate from Raw Gas Compressor (stages 1 to 4)	- C4 -
- Condensate from Rectisol unit (effluent from methanol/water separation diluted with cooling water)	- RU -
- Effluent from the ash settling pond (clear water run-off)	- C5 -

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<p>on Nov. 19, 1979</p> <table border="0"> <tr> <td></td> <td>TRW-Designation</td> </tr> <tr> <td>- Fresh water input (cooling water)</td> <td>- PW -</td> </tr> <tr> <td>- Condensate from raw gas compressor (as above)</td> <td>- C4 -</td> </tr> <tr> <td>- Condensate from Rectisol unit (as above)</td> <td>- RU -</td> </tr> <tr> <td>- Effluent from the ash settling pond (as above)</td> <td>- C5 -</td> </tr> </table> <p>Since two different fresh water inputs to the wash water system were used but only 2 x 4 sets of sample containers were supplied, on one day PSE water on the other cooling water was sampled.</p> <p>In addition, one average sample (2 kg) of pulverized feed coal, taken at the exit of the coal dust bunker in coal preparation, was supplied to TRW.</p> <p>5. <u>Results</u></p> <p>The essential results specified in the agreement between TRW and Krupp-Koppers are summarized in Tables 1 to 3.</p> <p>Table 1 shows the analysis of a feed coal dust sample determined by Krupp-Koppers. It also contains analyses of the average raw coal used in gasification for the weeks ending Nov. 14 and 21, 1979, respectively, as determined by the AECI laboratory. In Table 2 the analyses of the agreed-on gas streams are compiled. The raw gas sample was taken after the raw gas blower in the common line for all gasifier trains leading to the gas holder. Thus, an average sample of the total raw gas production was obtained.</p> <p>Methane and higher hydrocarbon content of the raw gas is extremely low. A previous study by AECI resulted in approximately 140 ppm v/v of CH<sub>4</sub> and 20 to 25 ppm of C<sub>2</sub> plus C<sub>3</sub></p>					TRW-Designation	- Fresh water input (cooling water)	- PW -	- Condensate from raw gas compressor (as above)	- C4 -	- Condensate from Rectisol unit (as above)	- RU -	- Effluent from the ash settling pond (as above)	- C5 -
	TRW-Designation												
- Fresh water input (cooling water)	- PW -												
- Condensate from raw gas compressor (as above)	- C4 -												
- Condensate from Rectisol unit (as above)	- RU -												
- Effluent from the ash settling pond (as above)	- C5 -												
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hydrocarbons in the raw gas before CO shift conversion. Some methanation occurs during CO shift. Mercaptans and aromatics have never been found in KT raw gas.

The tail gases from the Rectisol unit consist primarily of CO<sub>2</sub> and nitrogen used for stripping.

The results of the water analyses are summarized in Table 3. The samples were taken in parallel to those supplied to TRW.

Two different water streams are used as make-up water for the wash water system, PSE water and cooling water. PSE water is a mixture of Johannesburg purified-sewage water and different process waters from the Modderfontein plant, including water effluent from the settling pond of No. 4 ammonia plant. The only effluent from the wash water system is the run-off of the ash settling pond, sampled and analysed on two different days. Also two analyses each are included of the raw gas condensates obtained in the raw gas compressor and in the Rectisol unit.

Tables 2 and 3 list besides the analyses the determined flow rates and temperatures of the process streams.

6. Discussions of the Results

Composition and properties of the feed coal have a strong effect on the gasification results, but also on the side reactions which lead to formation of the trace by-products contained in the raw gas. In addition, in the plant layout many alternatives are feasible for processing the product gas as well as the sidestreams. Therefore, general application of the reported results are limited.

The product raw synthesis gas leaving the gasifier/waste heat system is further cooled and intensely washed for flyash removal in the cooling washer and the disintegrator stages.

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The composition of the water-washed raw gas is listed in Table 2. The main components of the raw gas are CO, H<sub>2</sub>, CO<sub>2</sub> and N<sub>2</sub>. The sulfur contained in the coal feed is present in the gas as H<sub>2</sub>S, COS and CS<sub>2</sub> which are removed from the gas in the following Rectisol unit. Trace components of the raw gas are NH<sub>3</sub>, HCN, SO<sub>2</sub> and NO.

A big advantage of the Koppers-Totzek process with regard to its effect on the environment lies in the fact that the produced raw gas contains no coal distillation products because of their spontaneous gasification at the extremely high temperatures. Aromatics, phenols, and mercaptans have never been detected in KT raw gas.

Waste gas streams in the Modderfontein plant are the tail gases from the Rectisol unit, the tail gas of the liquid nitrogen wash and the combined stream of flue gas used for drying in the coal preparation unit and of the conveying gas for coal dust. The tail gases of the Rectisol unit (Table 2) consist primarily of CO<sub>2</sub> and the nitrogen used for stripping. The tail gas of the liquid nitrogen wash is burned in the boiler station. If flue gas cannot be used for drying the coal because of environmental reasons it can be replaced by hot gas produced by burning the tail gas from the liquid nitrogen wash and/or desulfurized raw gas. In this case the flue gas can be vented through a relatively low stack of 25 m after the dust content has been lowered to less than 100 mg/m<sub>n</sub><sup>3</sup>.

In the raw gas washing stages NH<sub>3</sub>, HCN, SO<sub>2</sub> and to a small degree H<sub>2</sub>S and CO<sub>2</sub> are dissolved in the water. At the pH of the wash water, its temperature level and especially because of its flyash content, there is a rapid conversion of H<sub>2</sub>S to S<sub>2</sub>O<sub>3</sub><sup>2-</sup> and SO<sub>4</sub><sup>2-</sup>. The HCN reacts with the sulfur compounds to form SCN<sup>-</sup> and with the iron content of the flyash to form insoluble complexes.

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<p>All water streams, wash waters and condensates, which have been in contact with the raw gas are fed to the common wash water system (Fig. 5). The water streams are conducted through covered gutters to the clarifier where the solids are removed. Clarified water is recycled via a cooling tower to the main wash stages. The flyash leaves the clarifier as a slurry and is pumped to settling ponds for deposit.</p> <p>The make-up water to the wash water recycle consists of PSE water fed to the clarifier and cooling water which is used in the HCN wash and for dilution of the Rectisol condensate. Both make-up waters have a relatively high salt content. The PSE water contains also a certain amount of <math>\text{NH}_3</math>. A water purge stream from the CO shift conversion unit is used as quench water for partial quenching of the hot raw gas exiting the gasifier.</p> <p>The only water effluent leaving the No. 4 ammonia plant is the run-off of the ash settling pond. As the analyses in Table 3 show, is its composition very similar to that of the make-up water. Its content of toxic substances is very low indeed. This is due to the long residence time in contact with the flyash in the settling pond. In one commercial KT-coal gasification plant this waste water has for years been used to water the fields and as drinking water for animals.</p> <p>If there is not sufficient space for settling ponds the fly-ash slurry can be filtered and the purge water stream which may be significantly smaller than that used in Modderfontein can be cleaned by an oxidative chemical treatment. It is possible to eliminate in a relatively simple way the traces of toxic material virtually completely by oxidation so that the impositions by the authorities, for instance, those applicable in the German Federal Republic, can be met. Should regulations reduce the limits for the ammonia content and the COD value in waste water, a biological treatment after chemical oxidation can ensure further decomposition of detrimental ingredients.</p>			
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<p>While meeting stricter regulations concerning pollution is relatively easy for KT gasification, one must take into account that some measures necessary for extreme environmental protection show up in increased investment costs.</p>		
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KRUPP-KOPPERS		Table 1	
Feed Coal Analysis		SEITE	VON
		AUSGABE	
Sample	Coal Dust	Raw Coal	
Date	23-11-79	14-11-79	21-11-79
Determination by	KK	AECI	AECI
Particle Size    wt %			
>100 µm	26,9		
> 61	15,1		
> 29	20,5		
> 19	7,2		
> 11	9,3		
> 6,6	4,1		
< 6,6	16,9		
Moisture            wt %	1,5	3,7	3,6
Elemental Composition        wt % (mf)			
H	3,7	3,5	3,5
C	65,1	62,6	69,7
Scombustible	0,5		
N	1,6	1,7	1,6
Ash	20,7	19,1	18,3
O	8,4		
Ash Composition    wt %			
Fe <sub>2</sub> O <sub>3</sub>	5,0		
SiO <sub>2</sub>	44,8		
Al <sub>2</sub> O <sub>3</sub>	27,8		
CaO	9,6		
MgO	2,2		
Na <sub>2</sub> O	0,2		
K <sub>2</sub> O	0,6		
TiO <sub>2</sub>	1,4		
P <sub>2</sub> O <sub>5</sub>	1,3		
SO <sub>3</sub>	6,6		
Total Sulfur        wt % (mf)	1.1	1.2	1.0
ABT.: FE-I    NAME Dr. Firnhaber    DATUM 12.2.80		Krupp-Koppers GmbH, D-4300 Essen 1	

KRUPP-KOPPERS				Table 2																			
		Gas Analyses		PAGE	OF																		
				ISSUE																			
Sample		Raw Gas after Raw Gas Blower	Tail Gas from H <sub>2</sub> S absorber	Tail Gas from CO <sub>2</sub> absorber																			
Date		23-11-79	16-11-79	16-11-79																			
Flow Rate	Nm <sup>3</sup> /h	103 600	13 700	48 800																			
Temperature	°C	46	27	29																			
Composition																							
H <sub>2</sub> O	g/Nm <sup>3</sup>	54	5	5																			
H <sub>2</sub>	Vol.%(dry)	28.2	nil	nil																			
CO	"	59.1	1.9	0.3																			
CO <sub>2</sub>	"	10.9	52.6	84.3																			
N <sub>2</sub> /Ar	"	1.8	45.5	15.4																			
CH <sub>4</sub>	"	< 0.1 <sup>1)</sup>	< 0.1	< 0.1																			
H <sub>2</sub> S	mg/Nm <sup>3</sup> (dry)	6333	2)	nil																			
COS	"	740	2)	nil																			
CS <sub>2</sub>	"	450	2)	nil																			
SO <sub>2</sub>	"	14	nil	nil																			
NH <sub>3</sub>	"	57	39	3																			
HCN	"	76	62	8																			
NO <sub>x</sub>	ppm v/v	15	nil	nil																			
Mercaptans		nil	nil	nil																			
Methanol	Vol.%(dry)	nil	3)	4)																			
<div>1) Methane and higher hydrocarbons: In January through March, 1978, a study was made by AECI on the hydrocarbon content of the synthesis gas after desulfurization and after CO shift and CO<sub>2</sub> wash. The average results of six analyses each were in ppm v/v.</div> <table><tr><th></th><th>Gas ex H<sub>2</sub>S Absorber<sup>2</sup></th><th>Gas ex CO<sub>2</sub> Adsorber<sup>2</sup></th></tr><tr><td>CH<sub>4</sub></td><td>140</td><td>382</td></tr><tr><td>C<sub>2</sub>H<sub>4</sub></td><td>1.4</td><td>0.6</td></tr><tr><td>C<sub>2</sub>H<sub>6</sub></td><td>15</td><td>13</td></tr><tr><td>C<sub>3</sub>H<sub>8</sub></td><td>5</td><td>nil</td></tr><tr><td>C<sub>2</sub>H<sub>2</sub></td><td>nil</td><td>nil</td></tr></table> <div>2) Not determined; design number is &lt; 2 ppm total sulfur 3) Not determined; design number is 0.12 Vol. % 4) Not determined; design number is 0.05 Vol. %</div>							Gas ex H <sub>2</sub> S Absorber <sup>2</sup>	Gas ex CO <sub>2</sub> Adsorber <sup>2</sup>	CH <sub>4</sub>	140	382	C <sub>2</sub> H <sub>4</sub>	1.4	0.6	C <sub>2</sub> H <sub>6</sub>	15	13	C <sub>3</sub> H <sub>8</sub>	5	nil	C <sub>2</sub> H <sub>2</sub>	nil	nil
	Gas ex H <sub>2</sub> S Absorber <sup>2</sup>	Gas ex CO <sub>2</sub> Adsorber <sup>2</sup>																					
CH <sub>4</sub>	140	382																					
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C <sub>3</sub> H <sub>8</sub>	5	nil																					
C <sub>2</sub> H <sub>2</sub>	nil	nil																					
DEP.:	NAME	DATE:																					

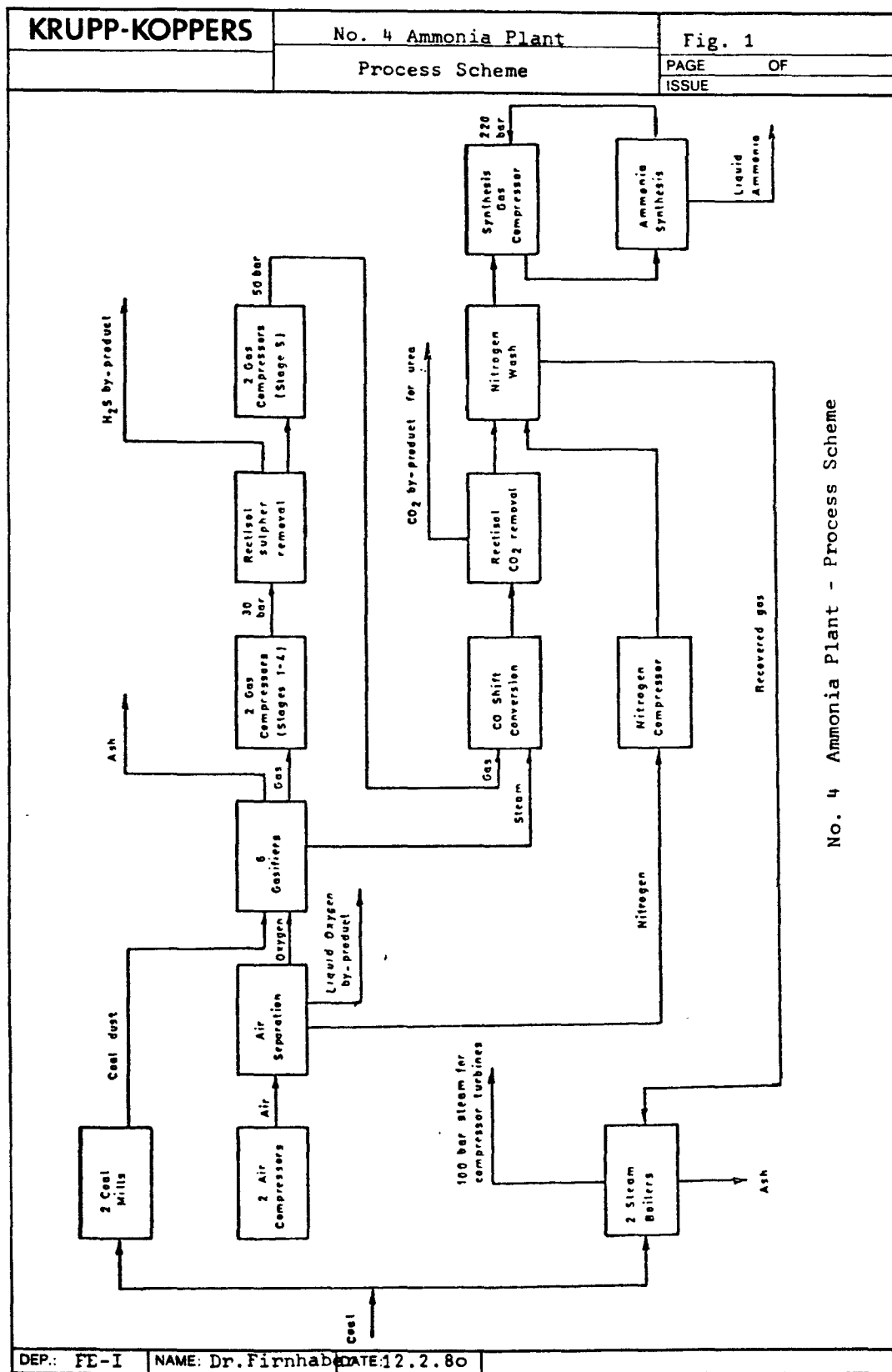
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AGT. FE-1		NAME: Dr. Firnhaber		DATUM: 12.2.1980		KRUPP KOPERS				Water Analyses		Table 3		SEITE VON		AUSGABE	
Sample		Fresh Wash Water Input (PSE)	Cooling Water Input	Effluent from Settling Pond		Condensate from Raw Gas Compressor		Diluted Condensate from Rectisol Unit									
Date		12-11-79	19-11-79	12-11-79	19-11-79	12-11-79	19-11-79	12-11-79	19-11-79								
Flow Rate	m <sup>3</sup> /h	215	54	230	230	9.3	9.0	4.1	3.6								
Temperature	°C	23	30	23	23	32	34	50	54								
pH		6.8	8.5	8.0	9.4	8.2	8.0	9.1	8.1								
Total suspended solids	mg/l	nil	0	nil	nil	nil	12	70	20								
Total dissolved solids	mg/l	1580	1460	1580	1530	260	170	1390	1640								
Hardness	mg/l	452	621	420	664	60	46	691	554								
Alkalinity, p-Value	as	0	0	0	44	0	0	0	0								
m-Value	CaCO <sub>3</sub>	26	167	126	79	2990	2690	78	144								
Conductivity	µmhos/cm	2300	1900	2100	2100	6000	5500	1800	2000								
COD	mg/l	38	118	353	43	644	569	28	1000								
NH <sub>3</sub>	mg/l	73	2.4	38	28	973	900	26	49								
CN <sup>-</sup>	"	0.2	1.2	0.2	0.2	7.2	10.5	2.8	ND								
SCN <sup>-</sup>	"	2.1	2.1	1.3	2.2	10.9	17.1	110	137								
H <sub>2</sub> S	"	nil	traces	nil	nil	43.9	53.5	1.1	4.5								
S <sub>2</sub> O <sub>3</sub> <sup>--</sup>	"	nil	nil	nil	nil	4.8	7.8	18.5	16.4								
SO <sub>3</sub> <sup>--</sup>	"	nil	nil	3.6	nil	nil	nil	nil	nil								
SO <sub>4</sub> <sup>--</sup>	"	584	853	752	706	56	49	461	541								
PO <sub>4</sub> <sup>3-</sup>	"	10	2.4	5	0.4	2	3	0.1	2.8								
Cl <sup>-</sup>	"	185	172	145	163	23	13	153	158								
Methanol	mg/l							< 0.1	< 0.1								
Dissolved oxygen *	mg/l																

Note: The term "nil" indicates a concentration below the limit of detection which is for the applied analytical methods below 1 mg/l

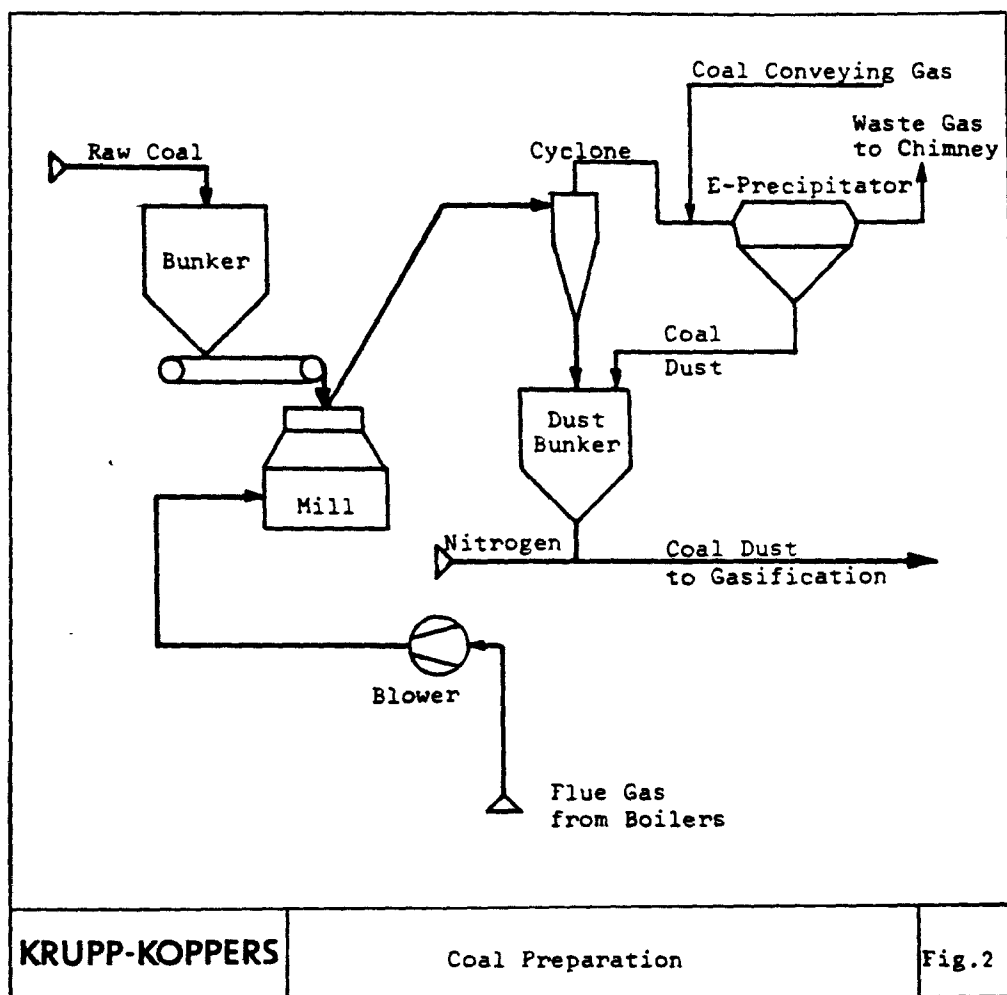
\* Dissolved oxygen was measured in January, 1980 while the plant was at full production rates by AECI personnel using a T.O.A. dissolved oxygen meter. The results obtained were:  
 Fresh Wash Water Input - 3.0 mg/L  
 Effluent from Settling Pond - 6.8 mg/L  
 Diluted Condensate from Rectisol Unit - 0.4 mg/L



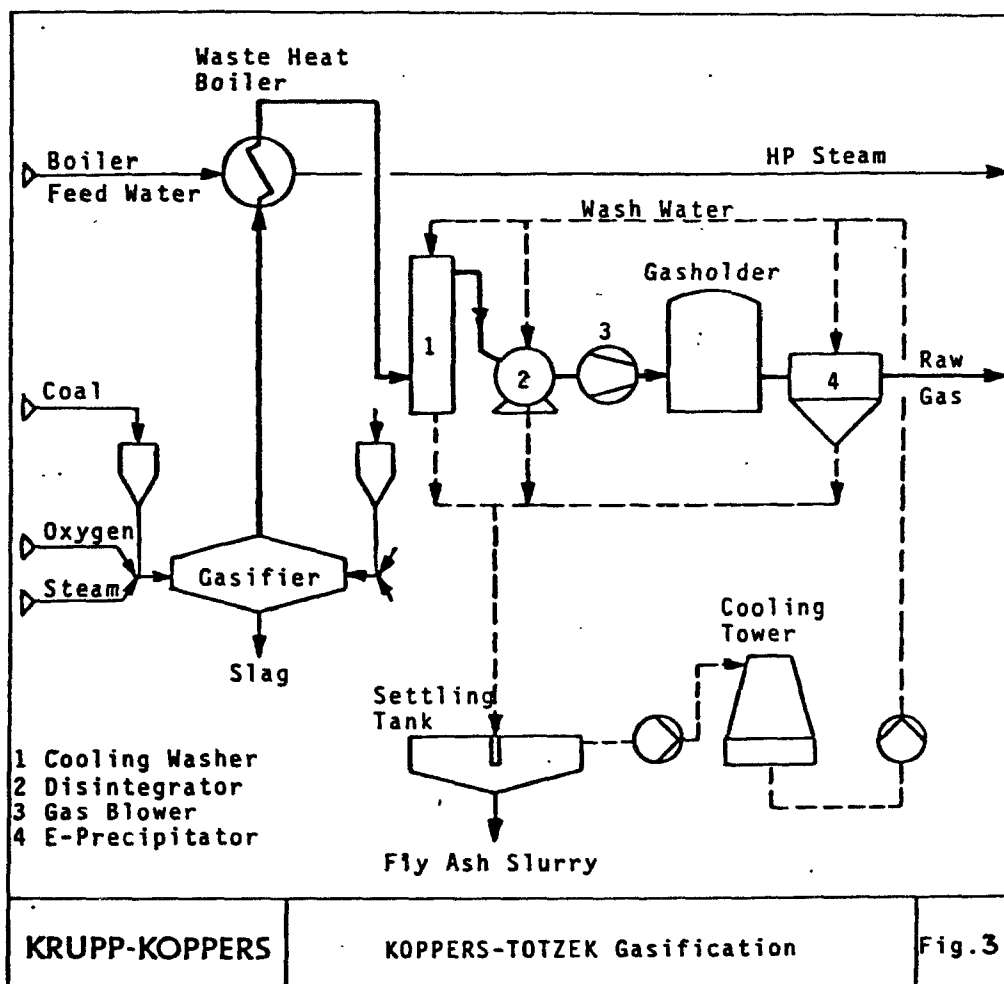
No. 4 Ammonia Plant - Process Scheme

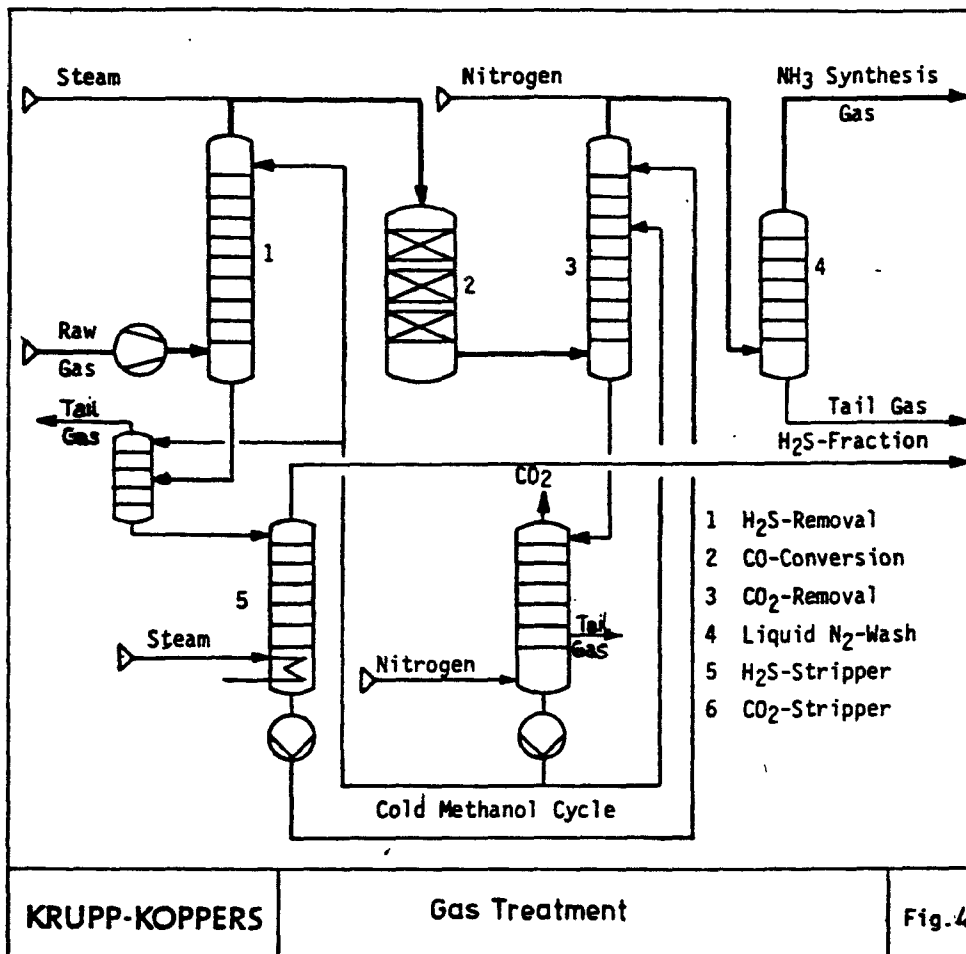
DEP: FE-I NAME: Dr. Firnhaber DATE: 12.2.80

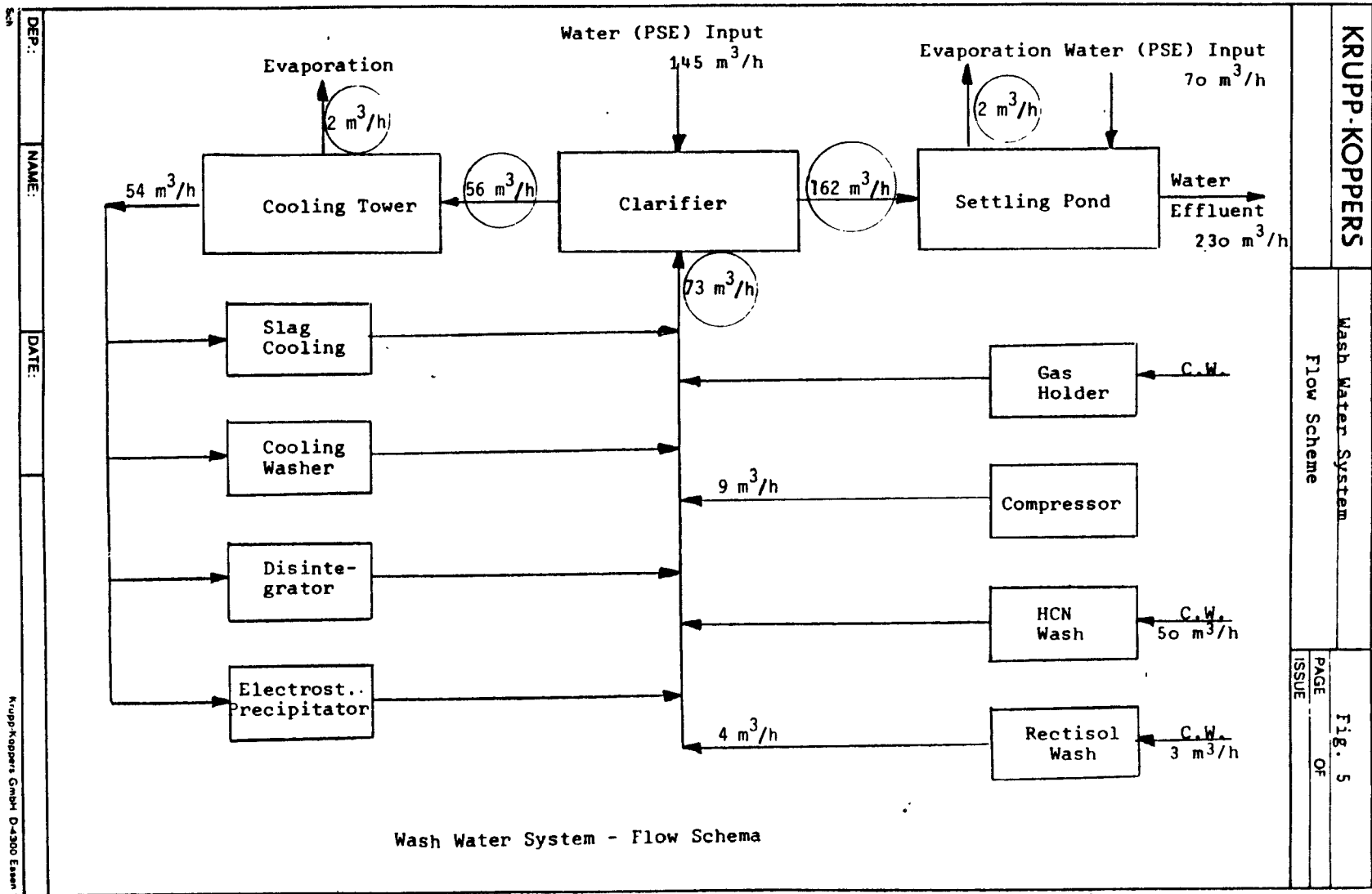
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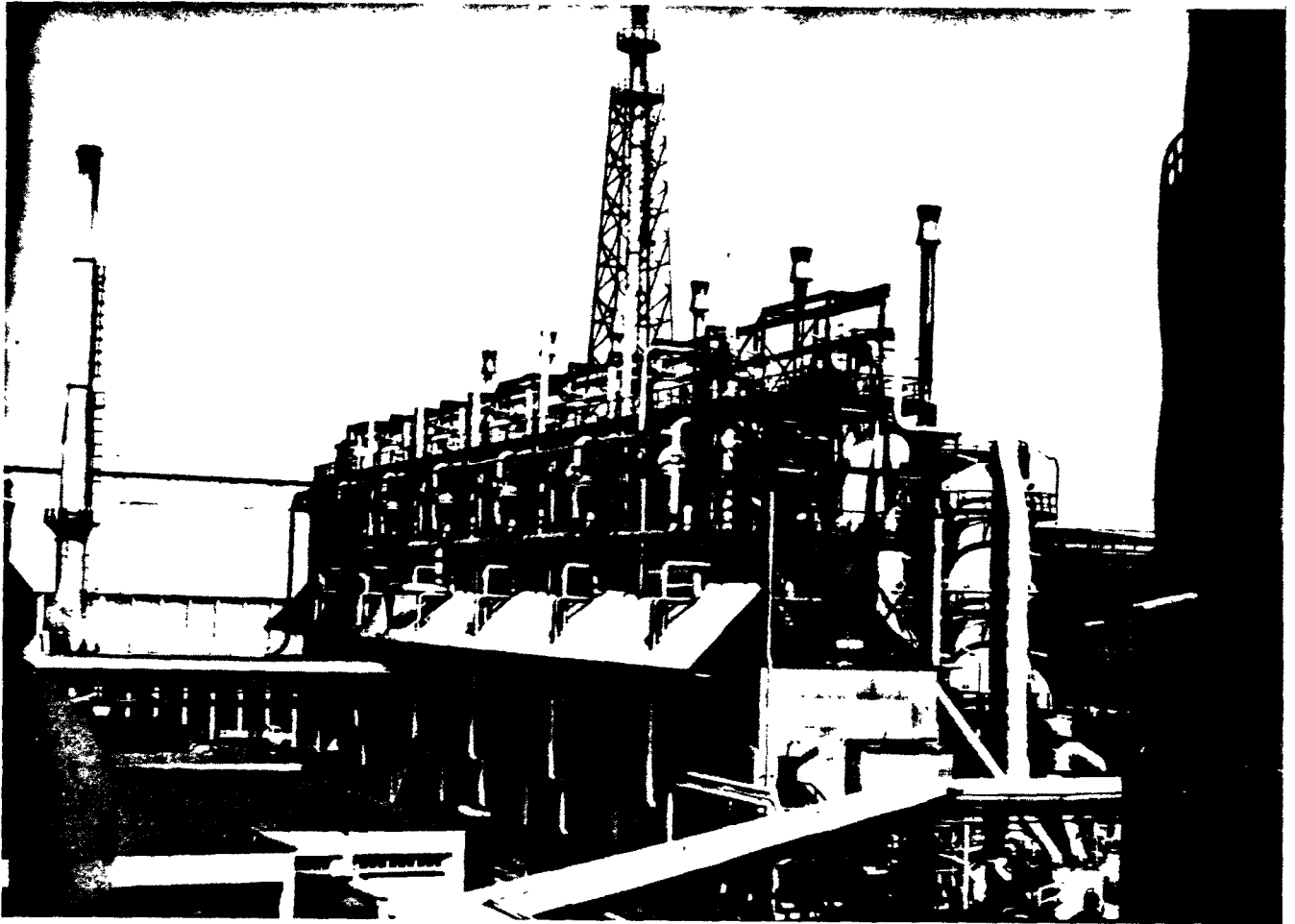


Fig. 6: Gasification plant with 6 Koppers-Totzek  
gasifiers at the Nitrogen Fertilizer Works  
Modderfontein, South Africa.  
Capacity: 1.000 tons/day ammonia

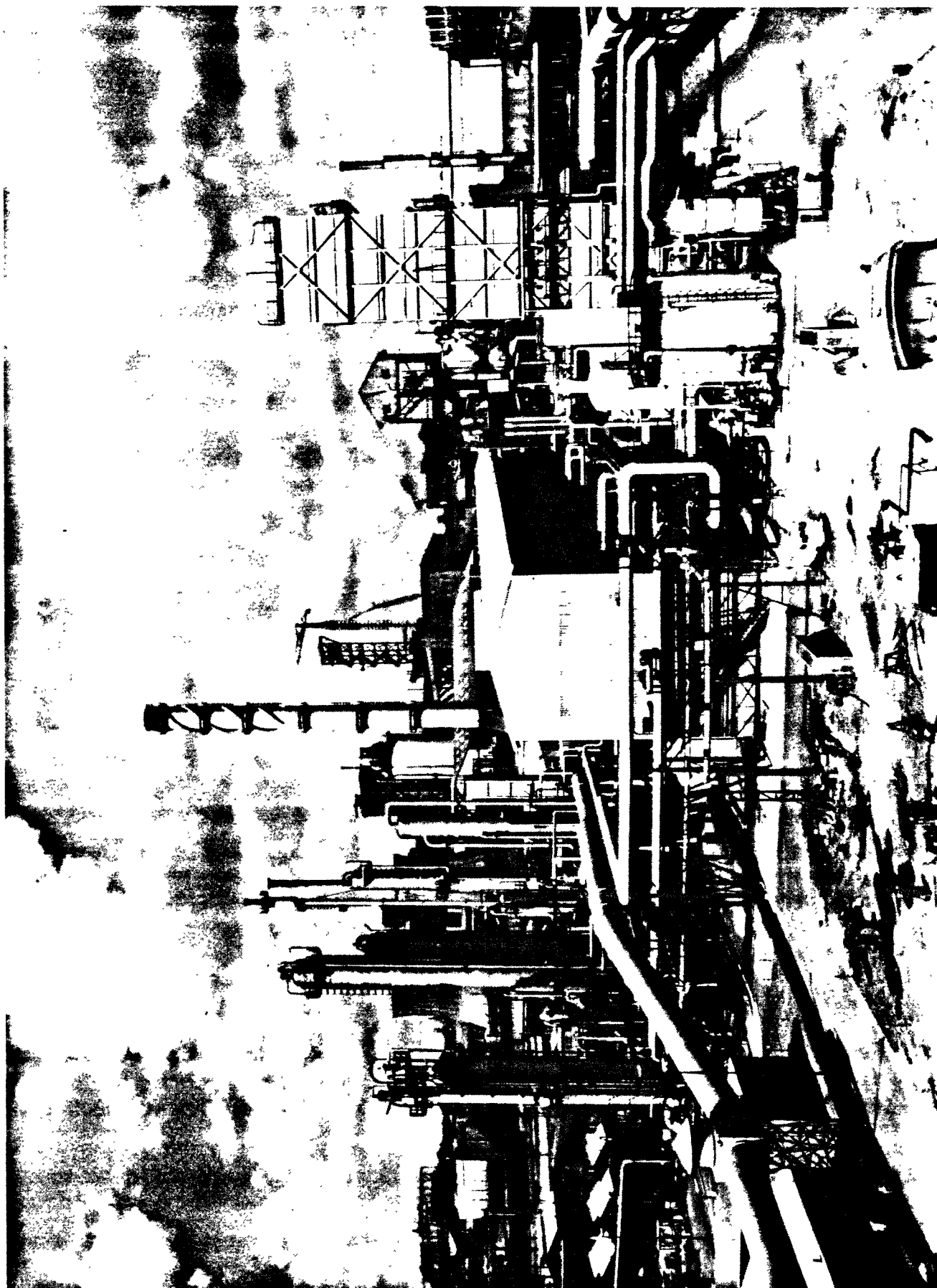


Fig. 7: Gas treatment unit and compressor house at the Nitrogen Fertilizer Works Modderfontein, South Africa.

TECHNICAL REPORT DATA (Please read instructions on the reverse before completing)		
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16. ABSTRACT The report gives results of a source test program at a Koppers-Totzek (K-T) coal gasification plant operated by AECI, Ltd. at Modderfontein, Republic of South Africa. EPA is interested in the K-T process because process economics and demonstrated commercial reliability make it a viable prospect for U.S. applications. Responsibilities for sampling, analysis, and engineering descriptions of the plant were shared by TRW and Krupp-Koppers GmbH of Essen, Federal Republic of Germany. EPA's phased approach for environmental assessments was followed. Level 1 and Level 2 data were collected along with priority pollutant screening data. Much of the effort was focused on wastewater streams. Wastewater treatment, consisting of a clarifier and settling pond, was adequate to produce a final discharge that had lower pollutant levels than the fresh input waters supplied to the plant. The report contains complete data and describes the K-T process and the Modderfontein plant. The Source Test Evaluation (STE), intended as an initial effort, was somewhat limited in scope. Recommendations for future STE programs are provided.		
17. KEY WORDS AND DOCUMENT ANALYSIS		
a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Pollution Coal Gasification Assessments Waste Water Water Treatment	Pollution Control Stationary Sources Koppers-Totzek Process Source Testing	13B 13H 14B
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