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Supplement for the Period April—June 1981

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International Coal Refining Company
Allentown, Pennsylvania

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Note

Articles in this Supplement were previously withheld from publication to avoid premature disclosure of inventions. Three articles were published prior to June 1984 and are not republished here. The article on page 1 was included in the April-June 1981 Quarterly Technical Report (DOE/OR/03054-4), the article on page 41 was included in the January-March 1982 Quarterly Technical Report (DOE/OR/03054-7), and the article on page 51 was published in the July-December 1983 Technical Report (DOE/OR/03054-94). Pagination has not been altered, since this could invalidate any cross-references.

CORROSION IN SRC-I COAL LIQUID DISTILLATION TOWERS

K. L. Baumert,* A. A. Sagüés,**
B. Davis,** and C. H. Jewitt***

During pilot plant operations for the SRC-I process, severe corrosion developed in the solvent fractionation towers. This high corrosion rate on carbon and types 304 and 316 stainless steel occurred when the pilot plant was processing coal with 0.15-0.39 wt % chloride. Subsequent stream analysis revealed that synergistic effects of inorganic chloride, phenols, and nitrogen compounds were responsible for corrosion most severe in the 200°C (400°F) boiling range. Liquid analysis also revealed the most likely path of chlorides to the tower.

INTRODUCTION

In the SRC-I process, solvent refining converts high-sulfur, high-ash coal to a clean-burning solid product: SRC (solvent-refined coal). This process is developing in the Wilsonville Pilot Plant which was commissioned in late 1973. Using ground coal and purchased hydrogen, the Wilsonville pilot process involves preheating a slurry of coal in coal-generated solvent and hydrogen, dissolving coal, separating mineral matter by filtration or critical solvent deashing, and recovering solvents and SRC, which is solidified on chilled plates. The process gases are cleaned with caustic soda and recycled with makeup hydrogen added. Figure 1 is a schematic of the Wilsonville Pilot Plant.

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PLANT EXAMINATION AND LEAK DETECTION

During pilot plant runs, the process was more efficient when heavier boiling solvent fractions were used for the coal slurry. Accordingly, an atmospheric fractionation column was installed after the vacuum tower to accomplish a more precise solvent separation. The fractionation tower was purchased used and is designed for full vacuum, 0.97-megapascals (MPa) (140-psi) pressure and 149°C (300°F). The tower is approximately 16.15 m (15 ft) long with a 0.914-m (3-ft) internal diameter. The bottom 9.14 m (30 ft) are clad with type 304 stainless steel; the upper 7 m (20 ft) of the tower are carbon steel. The original trays in this tower were type 316 stainless steel and numbered from the top of the tower; trays 1 through 9 were in the area where no stainless cladding is on the tower walls. The tower is oversized for the needs of the SRC-I process. The fractionation tower was lifted into position and secured in December 1976 and came on stream in approximately May 1977 during Indiana (Ind) #5 coal processing.

From late September in 1978 through January of 1979, Kentucky (Ky) #6 and #11 coals were processed in the pilot plant. During this time several leaks developed in unused nozzles in the solvent tower, which included both stainless-clad and carbon-steel nozzles. In February of 1979, the plant began to process Ky #9 Pyro coal. Shortly thereafter, severe leaks were noticed in the fractionation tower which was taken out of service after a total stream time of approximately 280 hours. Examination of the internal parts revealed that most severe corrosion occurred in the tray-15 area in the approximate boiling range of 200°C (400°F). The type-316 tray at location 15 was paper-thin; much of the cladding thickness on the vessel in this area was also lost. The area on the tower wall immediately above the cladding (between trays 9 and 10) was severely corroded also. The weld joining the uppermost cladding to the vessel wall had lost approximately 6.35 mm (¼ in.) of metal. Some stress corrosion cracking occurred in the tower; however, it is not known whether previous tower operation caused this cracking. There was also one rather severe temperature excursion recorded during Wilsonville tower operation.

Table 1 lists the coals processed in the pilot plant while the fractionation tower was operating. Amex Balle Ayr and Ind #5 coals were among those first processed and contained very low chlorine. Subsequently, Kentucky coals #6, #11, and #9, were processed and found to contain approximately 0.25% chloride. Because the severe corrosion was noticed during these runs, corrosion was assumed to occur during the high chloride coal runs. In more recent runs this assumption was verified. When Ky #9 Fies coal containing 0.07% chloride was processed without adding sodium carbonate, fractionation-tower corrosion rates were very low. Subsequent operation with Ky #9 Dotiki, containing a nominal 0.25% chloride, increased solvent-tower corrosion rates around tray 15 to 5-7.6 mm/yr (200-300 mils/yr). Prior to plant stream analyses, a correlation existed between solvent-tower corrosion rates and chloride amounts in the feed coal.

The tower was repaired in early 1979; type 321 stainless steel was used for replacement trays (Figure 2). The interior diameter cladding of the tower was raised approximately 1.2 m (4 ft); consequently, the cladding now ends between trays 7 and 8. Finally, external bypass loops were installed on the tower. Fluid in these loops is characteristic of trays 1, 9, and 15. Sample taps and corrosion probes were placed in these loops to measure on-stream corrosion rates and remove samples at any time.

In June 1979 during a short run before sodium carbonate was added, a very good correlation arose between the corrosion-probe readings and the thickness measurements of trays 9 and 15. This correlation suggested intolerably high corrosion rates of 7.6-26 mm/yr (300-1,000 mils/yr); hence, 1 wt % sodium carbonate was added. Since June of 1979, during high-chloride processing, 1 wt % sodium carbonate was added to the feed in order to maintain low fractionation-tower corrosion rates. The corrosion rates are measured daily; the plant adjusts feed-coal carbonate according to the amount of corrosion reported in the tower. More recently, a Ky #9 Fies coal was processed without sodium carbonate. During this run, corrosion probes detected essentially no corrosion in the tower.

CHEMICAL ANALYSIS

Most analytical results presented here are for two sets of samples collected at Wilsonville. One set was collected on July 20, 1979 when the unit was processing Ky #9 coal with maximum sodium carbonate addition: 9.07 kg/ton (20 lb/ton) of coal. Another set of samples collected December 15, 1979 using Ky #9 coal was processed with low sodium carbonate addition: 1.13 kg/ton (2.5 lb/ton). The fractionation-tower corrosion rate was low during maximum sodium carbonate addition. Conversely, during low sodium carbonate addition, the corrosion rate became unacceptably high.

After samples were collected December 15, 1979, larger amounts of sodium carbonate were added; a few days later corrosion rates decreased to an acceptable level. Consequently, the material(s) responsible for the severe corrosion should have been present in the December 15 samples. Table 2 provides the sodium carbonate addition rate and corrosion rates measured in the fractionation tower bypass when the preceding samples were collected.

Samples were analyzed for total chloride and water soluble chloride. The water-soluble inorganic chloride was removed from the organic sample by washing with distilled water.

Table 3 shows results for the chloride analysis (as ppm) around the tower for December 1979 samples. Because the sodium carbonate addition was changed during the interval sampled (Table 1), the analyses may reflect this change. Nevertheless, the three sampling periods are consistent and results follow the same trend.

The chloride data clearly demonstrate important points concerning tower corrosion.

- Tray-1 chloride content is lower than that in the tower feed; the chloride is organic rather than water-soluble chloride.

- Tray 15 chloride content is two-to-three times higher than that in the feed; more chloride is present as inorganic rather than organic chloride.

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- Chloride builds up in the tower, which is operated so that the flow from the bottoms is at least twice (and usually much greater than twice) the flow overhead. The bottoms flows over tray 15 and the overhead passes tray 1. If 350 ppm is the total feed chloride content and 94 ppm is the overhead with a 2-to-1 split bottoms-to-overhead, the maximum chloride content of tray 15 is calculated as follows: (350)(3) = (2)(x) ± (1)(4). Hence, the maximum chloride is 487 ppm. As the bottom flow increases to more than that of the overhead, maximum chloride in tray 15 decreases accordingly.

- Chloride concentration builds up in tray 15, but this elevated concentration does not appear to carry through to tower trays, because the bottoms chloride content is calculated as less than chloride in tray-15 liquid.

Sample liquids collected in trays 1, 9, and 15 during maximum sodium carbonate addition were also analyzed; each sample contained less than 10 ppm total chloride.

One question raised by the severe corrosion problem at Wilsonville and Ft. Lewis pilot plants was whether chloride was reacting with some component of the organic liquid to form a highly corrosive material. However, two samples containing several hundred ppm chloride were separated into phenolic, basic amine, and neutral fractions. After separation, each fraction contained the same organic chlorides concentration as in the as-received sample. Therefore, it is unlikely that chloride is concentrated in one, or a few, organic compounds.

Two small samples collected from the Wilsonville plant were acquired from the materials and ceramics group of the Oak Ridge National Laboratory (ORNL). One liquid was collected during Ind #5 coal processing when no corrosion was occurring in the fractionation tower. The other sample was collected on February 1, 1979 during Ky #9 coal processing when the corrosion rate was unacceptably high. Indiana coal contained 0.02 wt % chloride; Kentucky coal contained 0.25 wt % chloride.

Total chloride reported in Table 3 agrees well with values ORNL reported after analyzing portions of the same samples. As with the other tower liquids, the major portion of the chloride in the Ky-#9, coal-derived liquid is present in water-soluble, inorganic form. Organic chloride is present in similar amounts in both liquids. Significant differences in the nitrogen and sulfur content of either the feed liquid or the wash water from both coal-derived liquids did not appear (Table 4).

Samples were also obtained from June 24 through June 28, 1979 during Wilsonville Ky #9 coal processing (Lafayette coal). Sodium carbonate was first added June 28, 1979. Thus, these samples were collected shortly after the rebuilt tower was returned to operation but prior to sodium carbonate addition. Corrosion probes in the tower indicated a high corrosion rate during this period (Table 5). Total chloride values measured on tray samples collected June 24 and 26 (Table 6) show the same trend as in samples collected six months later. On both days tray-1 liquid contained less than 100 ppm chloride; nearly all chloride was organic chloride. Tray 15 (or tower bottoms) had a much higher total chloride content and a high water-soluble inorganic chloride content, which was not so high as in the December 15, 1979 samples.

Some of December 12, and 14, 1979 samples were analyzed for ammonia. The as-received sample was washed with water, which was made basic with sodium hydroxide; an NH_3-sensitive electrode determined NH_3 content. Ammonia measured by this method was low. For samples with 100-530 ppm Cl, ammonia was only about 10% of the amount of inorganic chloride present (Table 7). Before we can conclude that inorganic chloride is not present as ammonium chloride, this method must be evaluated further. However, ammonia and chloride data obtained by three labs using different techniques show a higher chloride than ammonia content.

Liquids from trays 1, 9, and 15 of the fractionation tower were analyzed for acidic compounds. Extraction with 5-10 wt % aqueous sodium bicarbonate is frequently used to separate carboxylic acids from mixtures. Carboxylic acids are not present as a major fraction of the tower liquids; instead, they varied from 0.6 wt % for tray-1 liquids to

0.1 wt % for tray-15 liquids. Because the bicarbonate solution probably removed some water-soluble phenols; these carboxylic acids concentrations should be considered the maximum concentration of carboxylic acids that may be present.

Phenols, extracted into sodium hydroxides and recovered by reacidification, comprise 35 to 55 wt % of the tower tray liquids (Table 8). The phenol content is highest for tray 1 and decreases as the tray number increases. (Trays are numbered from the top so that the uppermost tray is 1; the bottom tray is 20.) Phenolic content in the July 20, 1979 sample measured during maximum sodium carbonate addition, is very similar to that of samples collected at minimum sodium carbonate addition. In samples from the same tray, the ratios of individual phenolic compounds are nearly identical for both sample dates; liquids composition differences are within experimental error.

LABORATORY STUDIES

A series of experiments investigated the corrosivity of tower liquids and the effect of adding other species. These studies were conducted in an all-glass apparatus under total reflux conditions, an N_2 blanket, and at the liquid boiling temperature. The corrosion coupons were AISI 1050 carbon steel (CS) and type 304L stainless steel (SS) and electrically insulated from each other. Weight loss measurements taken at five-hour intervals were used to evaluate corrosion rates.

The first group of experiments used July 24, 1979 as-received, tray-9 liquid collected when the sodium carbonate addition rate was 1 wt % of the coal charge, and no corrosion was observed in the tower. Tray-9 liquid boiling temperature was 220°C (428°F). The corrosion rates were about 1.27 mm/yr (50 mils/yr) for CS and less than 0.127 mm/yr (5 mils/yr) for SS. At that point, approximately 1,000 ppm wt % ammonium chloride (NH_4Cl) was added to the liquid, resulting in about 19 mm/yr (750 mils/yr) corrosion rates for CS and 12.7 mm/yr (500 mils/yr) for SS. In consecutive five-hour tests this high corrosion rate diminished gradually as increasing amounts of metal were removed. Eventually, corrosivity returned to values observed prior to NH_4Cl

addition. When examined during high corrosivity, the SS coupons showed a dark, adherent, and sulfur-rich corrosion scale much like that observed in the corroded fractionation-tower, stainless steel trays. The CS coupons were free of the adherent scale; this was also observed in CS tower components.

Described below are the second group of experiments using different fractions of tower liquid (Table 10).

Experiment A

First, a portion of the as-received, tray-9 sample was separated as shown in Figure 3. The oil (I) fraction exhibited low levels of corrosivity. Adding NH_4Cl did not enhance that corrosivity and resulted in deposition of a white precipitate in the cooler portions of the corrosion apparatus. Reintroducing the phenolic (I) fraction resulted in precipitate dissolution and return to high corrosion rates.

Experiment B

Another experiment involved tray-15 liquid extracted on April 4, 1980, during high sodium carbonate addition. Separations were made using the method in Figure 4. The base corrosivity of the phenolic fraction (III) was low. Adding NH_4Cl resulted in virtually no changes in corrosion rates or in formation of a white deposit in cooler portions of the apparatus. Adding the amphoteric fraction (III) resulted in high corrosion rates and in dissolution of the white deposit.

Experiment C

The sequence of experiments was completed by testing the corrosivity of the oil (II) fraction; the white deposit appeared in all cases. Reintroducing part of the phenolic fraction (III) to this oil-NH_4Cl-basic mixture (II) to obtain about 25 wt % phenols resulted in return to high corrosion rates and dissolution of the white deposits.

DISCUSSION

The first group of experiments indicated that ingredients necessary for enhanced corrosivity (other than the appropriate type and quantity of chloride) are present in the tower even when sodium carbonate is being added to plant coal feed. This conclusion is consistent with results of stream chemical analyses in which the only significant effect of sodium carbonate addition was reduced inorganic chloride levels.

Results from the second group of experiments permit closer identification of species that play an important role in the corrosion process. Experiments A and C indicate that neutral fractions with or without basic compounds fail to provide the adequate liquid environment for high corrosivity in the presence of chlorides. In all cases a white, chloride precipitate (not necessarily ammonium chloride), suggests that corrosivity is low, because chloride does not stay in solution. Experiments A and C also show that when phenols are added to mixtures including both neutral and basic fractions with ammonium chloride, high corrosivity returns. This result implies that phenols provide an adequate medium to keep in solution inorganic chlorides necessary for corrosion.

Experiment B shows that high corrosivity can exist in the absence of the neutral fraction. Phenolic compounds by themselves, however, do not exhibit corrosive synergism with ammonium chloride. Restoration of the amphoteric fraction (Experiment B) or the basic fraction (Experiment C) is necessary for keeping the chloride in solution and for high corrosivity to occur. Although these findings can be extrapolated to other situations, nearby cold surfaces within the apparatus permit chloride removal from the liquid. The distillation tower, however, behaves more like a closed, high-temperature system which retains chloride in solution for longer periods. Under these circumstances the phenolic compounds alone may be able to retain chloride concentration great enough to result in significant corrosion.

The corrosion experiment results support the hypothesis of a corrosion mechanism involving HCl formation through inorganic chloride decomposition. The phenols can provide a suitable polar medium; whereas, the amphoteric or the basic fractions contribute compounds with a variety of base strengths. These compounds will react with initial ammonium chloride to produce inorganic chlorides of suitable solubility and dissociation temperatures.

CONCLUSIONS

The chemical analyses show:

- High chloride concentration in the fractionation tower samples occurred when high corrosion rates were observed.

- The majority of the chloride is present as inorganic chloride in the bottom of the tower where higher corrosion rates were observed.

- That similar organic chloride concentrations were observed in three organic fractions (phenolic, basic amine, and neutral), indicates that severe corrosion rates are not caused by a highly corrosive organic chloride.

- Liquids in trays 1, 9, and 14 have 35-50 wt % phenol concentrations; the highest phenol concentration is in tray 1 where low corrosion occurs.

- The basic amine fraction corresponds to at least ten times the chloride concentration; this result and the low ammonia concentration indicate that some of the chloride is present as basic nitrogen "inorganic" compounds.

- Chloride analyses of samples collected during December 1979 suggest the following pathway for chloride through distillation:

  Coal ---> Dissolver ---> Flash vessel ---> Vacuum tower ---> Fractionation tower

- With the exception of chloride, constituents necessary for enhanced corrosion are part of the coal-derived liquids even if Na_2CO_3 is added to the feed.

- Laboratory tests suggest that necessary ingredients for corrosion to occur are chloride, phenols, and amphoteric or basic fractions (probably nitrogen compounds).

Table 1

Analyses of Coals Processed with the Fractionation Column in Service

Coal	Analyses, wt % S	Cl	Ash
Amax Bell Ayr (Emery)	1	0.01-0.12	8.5
Indiana #5	4	0.01-0.07	11
Kentucky #6 and 11	2-5.3	0.20	
Kentucky #9 (Pyro)		0.24	
Kentucky #9 (Lafayette)		0.18-0.25	
Kentucky #9 (Fies)		0.07	
Kentucky #9 (Dotiki)		0.24	

Table 2

Plant Feed Parameters and Fractionation Tower Corrosion Rates During Periods of Sample Collection

Date	Na_2CO_3 added: kg/ton (lb/ton) coal	Chlorine in coal, wt %[a]	Corrosion rate measured at[b] [mm/yr (mils/yr)] Tray #9	Tray #15
7/25/79	9.07 (40)	0.25	0.025 (<1)	0.025 (<1)
12/12/79	2.27 (10)	0.18	0.229 (9)	0.686 (27)
12/13/79	2.27 (10)	0.18	0.940 (37)	3.05 (120)
12/14/79	2.27 (10)	0.18	0.940 (37)	4.72 (186)
12/15/79	2.27 (10)	0.18	2.36 (93)	11.1 (436)
12/16/79	3.4 (15)	0.18	6.35 (205)	8.15 (321)
12/17/79	3.4 (15)	0.18	3.3 (130)	5.18 (204)
12/18/79	3.4 (15)	0.18	1.42 (56)	1.96 (77)

[a] In all instances feed coal was Kentucky #9 (Lafayette).

[b] Corrosion rates were measured by 321SS Corrosometer (c) probe wire.

Table 3

Chloride in Fractionation Tower Feed

Feed	Chloride (ppm) Indiana #5	Chloride (ppm) Kentucky #9
Tower feed, total sample	133	820
Tower feed, water washed	141	254
Wash water	6	570

Table 4

Sulfur and Nitrogen in Fractionation Feed

	Indiana #5		Kentucky #9	
	Sulfur	Nitrogen	Sulfur	Nitrogen
Column feed, total sample	0.26 wt %	0.59 wt %	0.30 wt %	0.61 wt %
Column feed, water washed	--	0.53 wt %	--	0.60 wt %
Wash water	Trace	29 ppm	Trace	96 ppm

Table 5

Effect on Tower Corrosion of Adding Na_2CO_3 to Coal Feed

Date	Na_2CO_3 added kg/ton (lb/ton) coal	Average corrosion rate measured at Tray 15[a] mm/yr (mils/yr)
6/24/79 to 6/27/79	9.07 (20)	8.62 (339)
6/28/79 to 7/1/79	9.07 (20)	NA NA
7/1/79 to 7/6/79	9.07 (20)	<0.25 (<10)

[a] Corrosion rates were measured by 321SS Corrosometer (c) probe wire.

Table 6

Constituents in Tower Liquids Collected Prior to Carbonate Additions[a]

Date and location		H_2O soluble chloride	Chloride	Sulfate	Ammonia
6/24/79	Tray 1	<2	41	<10	550
	Tray 15	170	410	<10	480
	Bottoms	280	510	no extract	310
6/26/79	Overheads	<2	270	insufficient sample	1,450
	Tray 1	2	74	<10	650
	Tray 9	140	620	<10	580
	Tray 15	220	1,350	<10	420
6/28/79	B131B Bottoms	420	900	no extract	300

[a] All values in parts per million.

Table 7

Ammonia Extracted with Water from Fractionation Tower Organic Layer

			Water extract	
Feed	Date	Sample	Cl (ppm)	NH_3 (ppm)
Tray 1	12/15/79	1	1.9	2.2
		2	1.8	2.3
	12/14/79		2.5	2.1
Tray 9	12/15/79	2	151	3.8
	12/14/79		42	2.2
Tray 15	12/15/79	1	194	26
		2	532	30
	12/14/79		370	17
Feed	12/14/79		212	17

Table 8

Phenol Composition of Fractionation Tower Trays

	Sample	
Tray	7/20/79	12/15/79
1	51 wt %	45 wt %
9	47 wt %	43 wt %
15	37 wt %	34 wt %

Table 9

Summary of Corrosion Experiments

Experimental medium	Corrosion rate, mm/yr (mils/yr)[a]				Comments
	CS		SS		
First group					
Tray 9, as received	1.02	(40)	0	(0)	--
Tray 9, 4,000 ppm NH_4Cl[b]	19.1	(750)	12.7	(500)	--
Second group					
A					
Tray 9, oil (I)	0	(0)	0	(0)	--
Oil (I) + 1,000 ppm NH_4Cl[c]	1.78	(70)	0	(0)	White deposit
As above + phenolic (I) fraction	26.7	(1,050)	20.3	(800)	Deposit dissolved
B					
Tray 15, phenolic (III)	0	(0)	--	--	--
Tray 15, phenolic (III) + 1,000 ppm NH_4Cl	0	(0)	--	--	White deposit
As above + amphoteric III	High	High			Deposit dissolved
C					
Tray 15, oil (II) + 1,000 ppm NH_4Cl	Low	Low			White deposit
As above + basic (II)	Low	Low			White deposit
As above + phenolic (III) (to 25% phenols)	High[d]	High[d]			Deposit dissolved, after 10 hr

[a] Corrosion rates are average for the first short period of exposure, unless indicated otherwise.

[b] Ammonia chloride concentrations shown in the table are computed on the basis of the whole liquid sample before separation.

[c] This medium contained a small amount (<1,400 ppm of the whole liquid) of ammonium carbonate. This addition was shown previously IMMR-48-PD-23-80 to have no significant effect on the corrosion behavior.

[d] These rates were reached only during the second exposure, coinciding with the complete dissolution of the white deposit.

Figure 1
Block Flow Diagram of the SRC-I Process

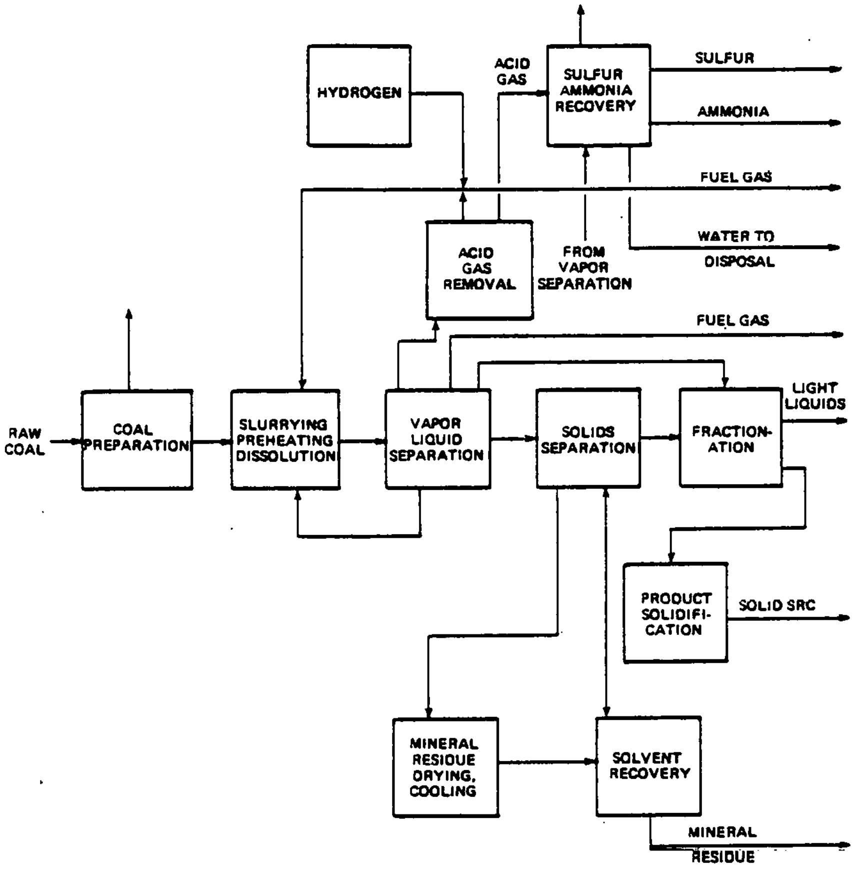


Figure 2
T-105 Fractionation Column
Wilsonville SRC Pilot Plant

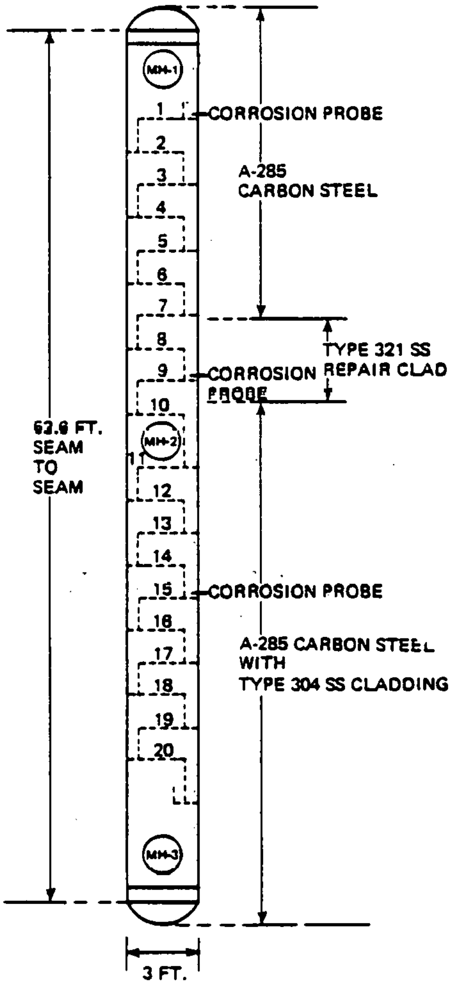


Figure 3
Phenol — Oil Separation Method

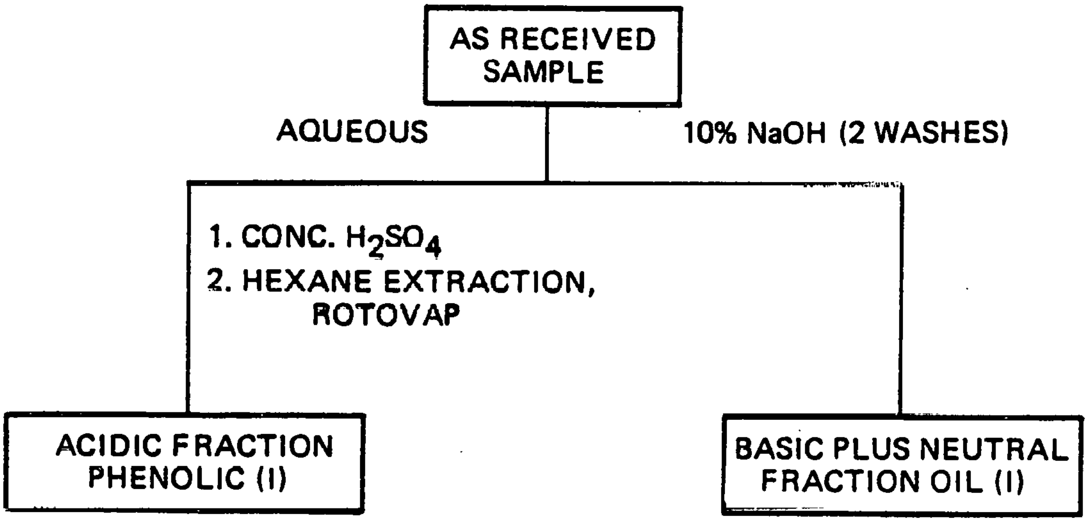
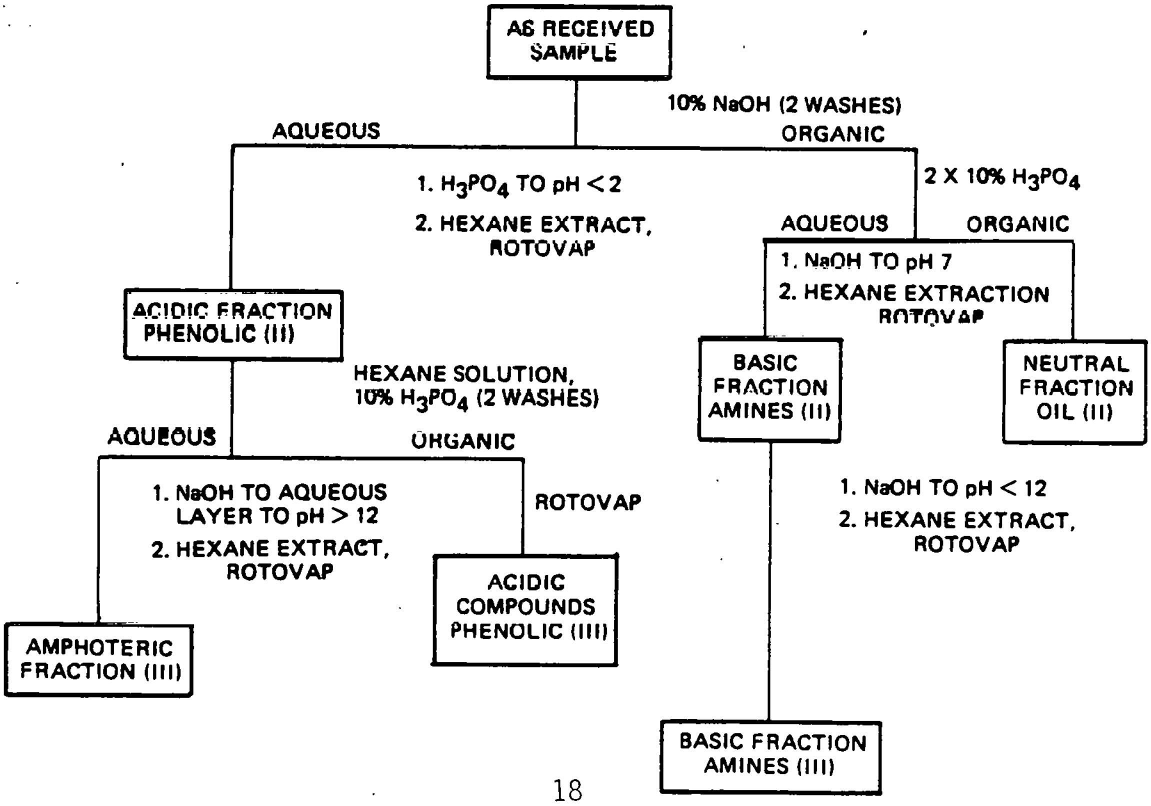


Figure 4
Separation Method for Phenols and Amines



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