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WILSONVILLE SRC-I PILOT PLANT

- I. FRACTIONATION AREA CORROSION STUDIES
- II. HOT vs. NORMAL SEPARATOR MODE OF OPERATION

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SPECIAL TOPICS FROM THE WILSONVILLE SRC PILOT PLANT PROGRAM

Jun Mounq Lee

ABSTRACTS

I. Fractionation Area Corrosion Studies

Extensive corrosion studies in solvent recovery columns (T105 Fractionation Column, T-102 Vacuum Column and T104 Light Organics Recovery Column) have been done during the last two years (1979 and 1980) with different coals (mainly Kentucky #9 Lafayette, Dotiki and Fies). Sodium carbonate (0.1-1.1% of coal) was added as neutralizer to control corrosion rate. Chloride balance runs were made for isolation of corrosive streams with high chlorine content. Caustic wash program of V164 (T104 Feed Tank) inlet streams have been developed for selective treatment of corrosive streams as an alternative means for possible replacement of sodium carbonate addition.

High chlorine content coals such as Kentucky #9 Lafayette and Dotiki (0.2-0.3%) were very corrosive, compared to low chlorine content coal, Kentucky #9 Fies (<0.1%).

Sodium carbonate addition (0.6-0.7% of coal) reduced corrosion rate from 500 MPY to insignificant level of less than 5 MPY.

Caustic wash of solvents could reduce corrosion rate by 50%, removing most corrosive compounds present in the 440-480°F boiling fraction.

II. Hot vs. Normal Separator Mode of Operation

Extensive studies for the hot separator mode of operation have been done by bypassing the E102 Product Cooler and the B103 Vacuum Column Slurry Preheater, in order to save substantial energy by elimination of dissolver slurry cooling (0.3 MM BTU/hr) and reheating for solvent recovery (1 MM BTU/hr). Impacts of the hot separator mode on plant operability, product quality and Kerr-McGee CSD Unit recovery have been studied.

The hot separator mode of operation was operable by controlling the V103 temperature to 740°F. Higher flow rate of the V105

oil stream (400-500 lb/hr) was observed in the hot separator mode of operation, compared to that (300 lb/hr) in the normal separator mode of operation.

In the hot separator mode of operation with 740°F of the V103 High Pressure Separator temperature, it was observed that pre-asphaltene contents increased in the SRC products such as V110 L/F SRC and CSD feed; CSD unit recovery was not affected significantly; solvent quality was not affected significantly.

When anthracene oil as start-up solvent and 30 wt % of LSRC in the process solvent were used, similar hydrogen transfer mechanism was observed, based on comparisons with product quality (BS/BI ratio in CSD feed), CSD unit recovery and solvent quality.

I. FRACTIONATION AREA CORROSION STUDIES

SOLVENT RECOVERY COLUMNS

BOILING POINT FRACTIONS

KENT # 9 - DOTIKI

2/26/80 - 4/4/80

<u>BP FRACTIONS</u>	<u>SOLVENT STREAM (WT %)</u>					
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>
< 350°F	11	10	4	1	14	-
350-450°F	10	9	11	7	75	2
450-550°F	58	59	64	48	11	48
550-650°F	16	16	16	22	-	24
650°F-EP	5	6	5	22	-	26

CORROSION EXPERIENCE

<u>DATE</u>	<u>COAL USED</u>	<u>CHLORINE, WT %</u>
74-JUN 77	(NO T-105)	
	KENT # 14	NA
	ILL. # 6	0.05-0.16
	PITT. # 8	0.10
	AMAX	<0.03
	KENT # 14	0.02-0.09
	EMERY	0.01-0.12
JUN 77	(T-105 INSTALLED)	
JUN 77-SEP 78	INDIANA V	0.01-0.07
SEP 78-JAN 79	KENT # 6/11	0.07-0.29

LEAKS IN T-105 & T-102 AREAS

FEB-MAY 79	KENT # 9(PYRO)	0.20-0.45
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HIGH CORROSION IN T-105 & T-102

ORNL & APCI INSPECTIONS

<u>DATE</u>	<u>COAL USED</u>	<u>CHLORINE, WT %</u>
6 FEB-15 JUN 79	T-105 OUT OF SERVICE FOR REPAIRS	
MAY 79-FEB 80	KENT # 9 (LAFAYETTE)	0.26-0.29
	HIGH CORROSION IN T-105 & T-102 350-500 MPY WITH 321 SS (T-105) 200 MPY WITH 316 SS (T-105) 250 MPY WITH 304 L SS (T-105) 30 MPY WITH 1010 CS (T-102) REPAIRS FOR T-105 & T-102 ORNL INSPECTION OF T-105 (DEC)	
28 JUN 79	Na ₂ CO ₃ ADDED AS NEUTRALIZER VERY EFFECTIVE IN CORROSION CONTROL	
FEB-MAY 80	KENT # 9 (DOTIKI)	0.28-0.48
	HIGH CORROSION IN T-105 & T-102 130 MPY WITH 316 SS (T-105) 100 MPY WITH 1010 CS (T-102) Na ₂ CO ₃ ADDITION TO CONTROL	

<u>DATE</u>	<u>COAL USED</u>	<u>CHLORINE, WT %</u>
MAY -AUG 80	KENT # 9 (FIES)	0.06-0.14
	LOW CORROSION IN T-102 40-50 MPY WITH 1010 CS (T-102) NO Na_2CO_3 ADDITION	
3-21 AUG 80	KENT # 9 (DOTIKI)	
	CHLORIDE BALANCE RUNS FOR ORNL & IMMR HIGH CORROSION IN T-105 & T-102 150 MPY WITH 321 SS (T-105) 200 MPY WITH 1010 CS (T-102) NO Na_2CO_3 ADDITION	
22 AUG 80	T-105 OUT OF SERVICE FOR REPAIRS	
AUG 80-MAR 81	KENT # 9 (FIES)	0.06-0.15
	MODERATE CORROSION IN T-102 50-100 MPY WITH 1010 CS (T-102) Na_2CO_3 ADDITION TO CONTROL	

T-105 CORROSION INSPECTION

(1) TRAYS EXAMINATION

MATERIAL: 321 SS (0.135 IN TH)

PERIOD: 15 JUN-15 SEP 79

COAL USED: KENT # 9 (LAFAYETTE)

Na₂CO₃ ADDITION TO CONTROL (28 JUN)

HIGHEST CORROSION RATE

60-70 MPY BETWEEN TRAYS 15 & 18

OPERATION TEMPERATURE: 450-500°F

(2) SHELL EXAMINATION (JAN 81)

1ST PICTURE

TRAY SECTION AREA BETWEEN TRAYS 9 & 10

SHELL: CARBON STEEL (3/8 IN TH)

CLADDING (7/64 IN TH): 321 SS TOP

309 SS WELDING

304 SS BOTTOM

2ND PICTURE

TRAY SECTION AREA BETWEEN TRAYS 17 & 18

SHELL: CARBON STEEL

CLADDING: 304 SS TOP

304 SS WELDING

304 SS BOTTOM

MORE CORROSION RATE IN LOWER SECTION (2ND) AND IN
DOWNCOMER THAN TRAY SECTION

PERIOD OF SERVICE: JUN 77-AUG 80

EFFECTS OF DIFFERENT COALS

CORROSION PROBE (40 MILS) TESTS

NO Na_2CO_3 ADDITION

<u>PERIOD</u>	<u>COLUMN</u>	<u>CORROSION RATE</u>		
		<u>TRAY</u>	<u>MATERIAL</u>	<u>MPY</u>
(1) KENT # 9 (LAFAYETTE), 0.26-0.29 WT % CL				
6/24-27/79	T-105	15	321 SS	340
		9	321 SS	520
1/14-16/80	T-105	15	304L SS	230
		9	316 SS	180
	T-102	PACKING	1010 CS	7

SIX DAYS RESPONSE TIME

(2) KENT # 9 (DOTIKI), 0.28-0.48 WT % CL

3/6-7/80	T-105	15	304L SS	NA
		9	316 SS	130
	T-102	PACKING	1010 CS	100

NINE DAYS RESPONSE TIME

<u>PERIOD</u>	<u>COLUMN</u>	<u>CORROSION RATE</u>		<u>MPY</u>
		<u>TRAY</u>	<u>MATERIAL</u>	
(3) KENT # 9 (FIES), 0.06-0.15 WT % CL				
5/25-7/29/80	T-105	15	304L SS	0
		9	316 SS	0
5/25-6/17/80	T-102	PACKING	1010 CS	2
6/21-29/80				15
7/7-15/80				42
7/15-25/80				50

T-104 BOTTOM TO T-102 TRAY 8 SINCE JULY

THIRTY DAYS RESPONSE TIME

9/8-16/80	T-102	PACKING	1010 CS	120
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RESIDUAL EFFECT BY DOTIKI (90-180 MPY)

EFFECT OF T-104 BOTTOM

KENT # 9 (DOTIKI), 0.28-0.48 WT % CL

CORROSION PROBE TESTS

NO Na_2CO_3 ADDITION

<u>PERIOD</u>	<u>COLUMN</u>	<u>TRAY</u>	<u>MATERIAL</u>	<u>MPY</u>
(1) T-104 BOTTOM TO T-102 TRAY 8				
8/3-13/80	T-105	15	304L SS	0
		9	316 SS	0
	T-102	PACKING	1010 CS	180
(2) T-104 BOTTOM TO T-105				
8/13-21/80	T-105	15	304L SS	160
		9	316 SS	0
	T-102	PACKING	1010 CS	90

EFFECT OF Na_2CO_3 ADDITION AS NEUTRALIZER

(1) KENT # 9 (LAFAYETTE), 0.26-0.29 WT % CL

(2) AMOUNT OF Na_2CO_3 ADDITION
KENT # 9 (LAFAYETTE), 0.26-0.29 WT % CL

(3) KENT # 9 (DOTIKI), 0.28-0.48 WT % CL

T-102 PACKING AREA, 1010 CS

2/26/80 COAL FED

3/4-6/80 100 MPY

3/7/80 Na_2CO_3 ADDITION (1.1 % OF COAL)

3/7-11/80 20 MPY

3/11-4/8/80 0 MPY

(4) KENT # 9 (FIES), 0.06-0.15 WT % CL

T-102 PACKING AREA, 1010 CS

9/3/80 COAL FED

9/3-8/80 40 MPY

9/8-16/80 120 MPY

9/16/80 Na_2CO_3 ADDITION (0.7 % OF COAL)

9/25-11/8/80 2 MPY

CHLORIDE BALANCE (ORNL)

KENT # 9 (DOTIKI), 0.28-0.48 WT % CL

NO Na_2CO_3 ADDITION

TEST PERIOD: AUGUST 3-21, 1980

- (1) T-104 BOTTOM STREAM CARRIES CORROSIVE CHLORIDE IN T-102
OR T-105
- (2) Na_2CO_3 ADDITION REMOVES CORROSIVE WATER-SOLUBLE INORGANIC
CHLORIDE. (IMMR)
- (3) CORROSION AGENTS: PHENOLS (TAR ACIDS)
BASIC NITROGEN COMPOUNDS
WATER-SOLUBLE CHLORIDES

TOTAL CHLORIDE CONTENT OF WILSONVILLE SAMPLES IS AFFECTED
BY ROUTING OF T-104 BOTTOMS

SRC-I SOLVENT RECOVERY AREA

--- = ALTERNATIVE ROUTE

Total Chloride (wt ppm)

T-104 Bottoms to T-102

T-104 Bottoms to V-160

oml

SODIUM CONTENTS IN SRC PRODUCTS

(KENT # 9 COAL)

<u>COAL</u>	<u>RUN (MB)</u>	<u>Na₂CO₃ ADD LB/ BATCH</u>	<u>COAL</u>	<u>Na₂O IN ASH, WT %</u>		
				<u>V-110 SRC</u>	<u>ASH CONC.</u>	<u>CSD SRC</u>
DOTIKI	202A	0	0.5	-	0.5	-
	203DE-208AB	40	0.5	4.9	2.7	4.4
FIES	209AB-212AB	0	0.7	1.4	0.8	2.7
	217AB-221G	25	0.9	15.0	3.8	19.5
	222ABC-225I	15	0.6	7.7	2.1	6.1

<u>COAL</u>	<u>Na₂CO₃ ADD LB/BATCH</u>	<u>RATIO</u> <u>(Na ADD + Na IN COAL)</u>		<u>Na RATIO IN ASH</u> <u>(ASH CONC./COAL)</u>
		<u>Na IN COAL</u>		
DOTIKI	0	1.0		1.0
	40	11.3		5.4
FIES	0	1.0		1.1
	15	3.3		3.5
	25	6.9		4.2

CAUSTIC WASH STUDIES (CATALYTIC)

OBJECTIVE: SELECTIVE TREATMENT OF CORROSIVE STREAMS

LAB TEST RESULTS:

(1) CAUSTIC AND WATER EXTRACTION

<u>EXTRACTION</u>	<u>SAMPLE</u>	<u>DATE</u>	<u>Na₂CO₃</u> <u>LBS</u>	<u>EXTRACTION</u> <u>YIELD, WT %</u>
CAUSTIC	V-105 OIL	2/12/80	40	15.1 ± 0.6
	V-164 OIL	2/28/80	NONE	22.7
	V-164 OIL	10/11/80	25	29.4
	T-104 BOTTOM	2/28/80	40	21.1
WATER	V-164 OIL	10/11/80	25	1.5

(2) CORROSION TEST (1010 CS)

<u>SAMPLE</u>	<u>DATE</u>	<u>TREATED</u> <u>SAMPLE</u>	<u>TEST TIME</u> <u>HRS</u>	<u>CORROSION RATE, MPY</u>	
				<u>WIRE</u>	<u>COUPON</u>
V-164 OIL	10/11/80	ORIGINAL	47.5	23.5	22.4
		CAUSTIC	47.5	13.0	13.1
		WATER	47.5	22.0	23.7
V-164 OIL	2/28/80	ORIGINAL	87	-	20.2
T-104 BOT.	2/28/80	ORIGINAL	215	7.9	10.9
		CAUSTIC	215	5.0	5.4

FUTURE CORROSION STUDIES

- (1) CONTINUOUS CORROSION RATE TEST UNIT
- (2) CORRELATIONS BETWEEN PLANT AND LAB TEST DATA
- (3) IDENTIFICATION OF CORROSIVE COMPONENTS
- (4) CORROSION ABATEMENT STUDY
- (5) BETTER CHLORIDE BALANCE

SUMMARY

- (1) HIGH CHLORINE COAL CORROSIVE (0.2-0.3%)
- (2) Na_2CO_3 EFFECTIVE NEUTRALIZER (0.7%)
- (3) 440-480°F FRACTION MOST CORROSIVE
- (4) CAUSTIC WASH REDUCES CORROSION BY 50%

II. HOT VS NORMAL SEPARATOR MODE OF OPERATION

DEFINITION

(1) NORMAL SEPARATOR MODE

USE E102 PRODUCT COOLER

B103 VAC. COL. SLURRY PREHEATER

(2) HOT SEPARATOR MODE

BYPASS E102 PRODUCT COOLER

B103 VAC. COL. SLURRY PREHEATER

OBJECTIVE

- (1) SUBSTANTIAL ENERGY SAVINGS BY ELIMINATION OF:
 - o DISSOLVER SLURRY COOLING (0.3 MM BTU/HR)
 - o REHEATING FOR SOLVENT RECOVERY (1MM BTU/HR)
- (2) IMPACTS OF HOT SEPARATOR MODE ON
 - o PLANT OPERABILITY
 - o PRODUCT QUALITY
 - o KERR-McGEE CSD UNIT RECOVERY

PLANT OPERABILITY

(1) SOLIDS ACCUMULATION IN V103

RUN 205-RUN 208 (3 APR-8 MAY 1980)

HOT SEPARATOR MODE, DOTIKI

V103 TEMP: 790-815°F

PROBLEMS: V103 LEVEL CONTROL

HIGH PRESSURE LETDOWN VALVE CONTROL

SOLIDS ACCUMULATION (1 MAY): 70% OF V103 VOLUME

ANALYSIS OF SOLIDS

ASH	=	63.6%	VOL. MATTER	=	11.5%
CARBON	=	26.1%	FIXED CARBON	=	24.9%
HYDROGEN	=	1.1%			
SULFUR	=	8.3%	SULFIDE	=	4.8%
NITROGEN	=	0.4%	ORGANIC	=	2.7%
CHLORINE	=	0.5%			

SOLUTION: REMOVE INSULATION (RUN 210)

LOWER V103 T TO 750°F

(2) OPERATION TEMPERATURE

<u>COAL</u>	<u>SEPARATOR MODE</u>	<u>RUN (MB)</u>	<u>T, °F</u>			
			<u>R101</u>	<u>V103</u>	<u>V110</u>	<u>T-102</u>
DOTIKI	NORMAL	204C	841	645	591	553
	HOT	206A&B,	840	800	693	596
		208AB				
FIES	NORMAL	209AB	840	710	633	559
	HOT	210AB	834	740	675	588
	HOT	219AB,	840	680-710	628-639	597
		220AB&DE				
	HOT	225BC,F, G & I	842	719-739	658-678	605
FIES	NORMAL	217AB&CD	785	550-600	509	547
	HOT	221B&G	768	648	590	594
	HOT	222ABC	754	647	590	597

DEMO PLANT CONDITION RUNS

<u>SEPARATOR MODE</u>	<u>T, °F</u>		
	<u>V103</u>	<u>V110</u>	<u>T-102</u>
NORMAL	650	590	555
HOT	740	680	600

BETTER HEAT EXCHANGER REQUIRED FOR PRECISE CONTROL
OF V103 T AND HEAT RECOVERY

(3) V105 OIL FLOW RATE

<u>SEPARATOR MODE</u>	<u>COAL</u>	<u>RUN (MB)</u>	<u>V105 OIL LB/HR</u>
NORMAL	DOTIKI	204C	340
	FIES	209AB	350
		217AB&CD	60
HOT	DOTIKI	206A&B, 208AB	580-610
	FIES	210AB	480
		219AB, 220AB&DE	230-310
		225BC,F, G&I	290-340
		221B & G	60-80
		222ABC	50

HIGHER DESIGN CAPACITY OF T-104 COLUMN REQUIRED FOR HOT
SEPARATOR MODE

OR

VACUUM FLASH OF V-105 OIL BEFORE FEEDING OVERHEAD TO PRESENT
T-104 AND BOTTOM TO T-102

PRODUCT QUALITY

(1) V110 L/F SRC

KENT # 9 DOTIKI

R101 T = 840°F, P = 2,100 PSIG

Na₂CO₃ ADDITION = 40 LB/BATCH

LSRC ADDED-FREE BASIS

KENT # 9 FIES

R101 T = 840°F, P = 2,100 PSIG

Na₂CO₃ ADDITION, LB/BATCH

209AB & 210AB	0
219AB, 220AB & DE	25
225BC, F, G & I	15

LSRC ADDED-FREE BASIS

KENT # 9 FIES

R101 T = 754-785°F, P = 2,000 PSIG

Na₂CO₃ ADDITION, LB/BATCH

217AB & CD 25

221B & G 25

222ABC 15

(2) CSD FEED

<u>RUN</u>	<u>COAL</u>	<u>SEPARATOR MODE</u>	<u>V103 T, °F</u>	<u>BS/BI IN V110 SRC</u>
209AB	FIES	NORMAL	710	2.0
210AB	FIES	HOT	740	2.1

<u>T102 BT T, °F</u>	<u>BS/BI IN CSD FEED</u>	<u>PREASPHALTENE % IN CSD FEED</u>	<u>CSD (B) RECOVERY, %</u>
559	3.2	18.8 (26.0) ^(A)	77-81
588	1.7	29.7 (41.6) ^(A)	79

(A) SOLVENT, UC AND ASH FREE BASIS.

(B) FORCED ASH BALANCE.

RUN 210AB IN HOT SEPARATOR MODE HAS HIGHER PREASPHALTENE
CONTENT IN THE CSD FEED.

CSD RECOVERY NOT AFFECTED SIGNIFICANTLY.

(3) CSD RECOVERY AND SOLVENT QUALITY

<u>COAL</u>	<u>SEPARATOR MODE</u>	<u>RUN (MB)</u>	<u>BS/BI IN CSD FEED</u>	<u>CSD^(A) RECOVERY, %</u>	<u>SOLVENT^(B) QUALITY, %</u>
DOTIKI	NORMAL	204C	3.1	79.0	70.4
	HOT	206A	1.5	68.6	66.7
	HOT	206B	1.8	72.1	66.8
	HOT	208AB	2.3	73.4-77.0	69.6
FIES	NORMAL	209AB	3.2	77-81	71.6
	HOT	210AB	1.7	79	69.1
	HOT	219AB	5.5	84.7-85.2	76.0/75.2
	HOT	220AB	5.7	84.5-84.9	78.1/79.8
	HOT	220DE	4.3	84.9-86.5	81.1
	HOT	225BC	3.8	85.6	-/82.2
	HOT	225F	3.9	82.6	-/78.5
	HOT	225G	3.6	88.9	-/81.2
	HOT	225I	4.2	82.2	-/75.6
FIES	NORMAL	217AB	3.7	89.6-89.7	81.7/79.3
	NORMAL	217CD	4.8	90.0-91.0	82.6/73.4
	HOT	221B	3.9	89.3	82.5-85.8/80.4
	HOT	221G	5.0	89.1	85.8/75.1
	HOT	222ABC	3.4	87.9-88.8	-/74.8

(A) FORCED ASH BALANCE.

(B) WITH LSRC/WITHOUT LSRC.

HOT FLASH MODE WITH DOTIKI COAL SHOWS
THAT:

LOWER BS/BI IN CSD FEED (1.5 vs. 3.1)
LOWER CSD RECOVERY (69 vs. 79%)
LOWER SOLVENT QUALITY (66.7 vs. 70.4%)

DEMO PLANT CONDITION RUNS (219AB, 220AB & DE) AFTER
30% LSRC RUNS IN PROCESS SOLVENT (216 AND 217) SHOW
THAT:

HIGH BS/BI IN CSD FEED (4.3-5.5)
HIGH CSD RECOVERY (85-87%)
HIGH SOLVENT QUALITY (76.0-81.1%)

DEMO PLANT CONDITION RUNS (225BC, F, G & I) WITH
ANTHRACENE OIL AS START-UP SOLVENT SHOW THAT:

HIGH BS/BI IN CSD FEED (3.6-4.2)
HIGH CSD RECOVERY (82-89%)
HIGH SOLVENT QUALITY (75.6-82.2%)

30% LSRC RUNS (217AB & CD, 221B & G, 222ABC) IN
PROCESS SOLVENT SHOW THAT:

HIGH BS/BI IN CSD FEED (3.4-5.0)
HIGH CSD RECOVERY (88-91%)*
HIGH SOLVENT QUALITY (81.7-85.8%)

* LSRC FREE BASIS: 78%

SIMILARITY IN HYDROGEN TRANSFER MECHANISM WITH ANTHRACENE
OIL AND WITH LSRC

CURRENT STUDIES IN PROGRESS

(1) NEW V-103 HIGH PRESSURE SEPARATOR

- o SHORTER RESIDENCE TIME
- o TEMPERATURE VARIATION

(2) NEW HEAT EXCHANGER TO PRECISELY CONTROL V-103 TEMPERATURE

- o 650°F-780°F

SUMMARY

(1) HOT SEPARATOR MODE OPERABLE

- o V103 TEMPERATURE 740°F
- o V105 OIL 400-500 LB/HR

(2) HOT SEPARATOR MODE WITH V103 T = 800°F

- o HIGH SOLIDS ACCUMULATION IN V103
- o INCREASE PREASPHALTENE CONTENT
- o DECREASE SOLVENT QUALITY
- o DECREASE CSD RECOVERY

(3) HOT SEPARATOR MODE WITH V103 T = 740°F

- o INCREASE PREASPHALTENE CONTENT
- o CSD RECOVERY NOT AFFECTED SIGNIFICANTLY
- o SOLVENT QUALITY NOT AFFECTED SIGNIFICANTLY

(4) SIMILAR HYDROGEN TRANSFER MECHANISM WITH ANTHRACENE OIL AND LSRC