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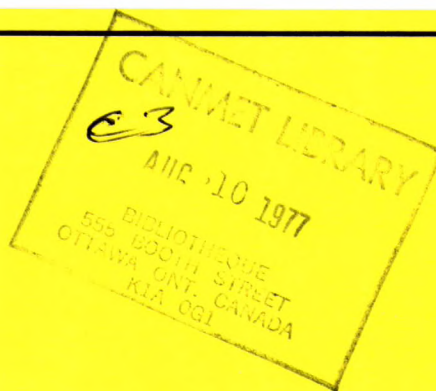
Canada Centre
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**PILOT PLANT THERMAL HYDROCRACKING OF
GCOS (GREAT CANADIAN OIL SANDS) BITUMEN:
3. EFFECT OF HEAVY OIL RECYCLE ON REACTOR
FOULING**

C.P. Khulbe, B.B. Pruden, J.M. Denis and W.H. Merrill

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(GREAT CANADIAN OIL SANDS) BITUMEN
3. EFFECT OF HEAVY-OIL RECYCLE ON REACTOR FOULING

by

C.P. Khulbe^{*}, B.B. Pruden^{*}, J.M. Denis^{**}
and W.H. Merrill^{***}

ABSTRACT

Thermal hydrocracking of Athabasca bitumen was carried out in a pilot-scale reactor at a pressure of 13.9 MPa, a liquid hourly space velocity (LHSV) of one, and temperatures of 450 and 460°C with recirculation of a portion of the heavy-oil product to the reactor bottom. The pilot plant was operated for 21 days (R-450) and 13 days (R-460) to determine the effect of heavy-oil recirculation on deposition of solids in the reactor and hot separator.

The effect of heavy-oil recirculation in both runs was to reduce substantially the deposition of solids in the reactor and hot separator compared to once-through runs at the same pitch conversion. The suppression of the formation of deposits was ascribed to a short "first pass" residence time and a high ash content of the reactor fluid.

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CANADIAN OIL SANDS) SUR UNE INSTALLATION
PILOTE

3. L'EFFET DU RECYCLAGE D'HUILE LOURDE SUR L'ENCRASSEMENT DU REACTEUR

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SOMMAIRE

Le procédé d'hydrocraquage thermique a été utilisé sur du bitume de l'Athabasca à l'aide d'un réacteur pilote, sous une pression de 13.9 MPa, à une vitesse spatiale liquide horaire (LHSV) de 1 et à des températures de 450°C et 460°C. De plus, pendant l'opération, une recirculation d'une partie de l'huile lourde a été effectuée par le fond du réacteur. L'essai à une température de 450°C (R-450) dura 21 jours et l'autre à 460°C s'échelonna sur 13 jours. Par la suite, l'effet de cette recirculation de l'huile lourde sur le dépôt de solides formé dans le réacteur et le séparateur à haute température a pu être déterminé.

L'effet du recyclage du produit d'huile lourde au cours des deux essais a été de réduire une quantité appréciable du dépôt de solides dans le réacteur et le séparateur à haute température à comparer avec les opérations d'une seule marche au même taux de conversion du brai. L'élimination de dépôts est due à un court temps de séjour du liquide dans le réacteur ainsi qu'à un fort contenu en cendre.

Droits de la Couronne réservés

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INTRODUCTION

This is the third report describing the effect of heavy-oil recycle on thermal hydrocracking pilot plant operation, product distribution and reactor fouling. The objectives of this research program have been documented in the first two reports (1,2) of the series. The first report was devoted to the effect of heavy-oil recycle on pilot plant operation and the second to a comparison of product qualities from a recycle run and a once-through run. Variations in the product quality during the plant operation for a recycle run were also documented in the second report. This report describes the effect of experimental parameters which apparently prevented deposition of solids in the reactor or hot separator.

The bitumen contains about 51 wt % pitch (fraction boiling above 524°C). It is necessary to convert pitch into a lower boiling fraction in order to obtain better yields of distillable hydrocarbons (fraction boiling below 524°C). Different processes, such as delayed coking, fluid coking and flexicoking have been developed for the first-stage refining of heavy oil and bitumens but the distillate obtained from these processes has a maximum yield of 76 wt % (3,4) and high amounts of undesirable coke are formed as by-product. A thermal hydrocracking process (5) capable of competing with these methods for heavy-oil processing has been developed at the Energy Research Laboratories, Department of Energy, Mines and Resources. The development of the hydrocracking process was based on short four-hour experiments (5) which yielded little information on the operation of the process over the extended periods necessary for a commercial plant. However, even these experiments indicated that at certain conditions, for example above 440°C at 7 MPa pressure or above 465°C at 10.4 MPa pressure, deposits were formed which filled one-third of the reactor after operation for several short runs. These deposits were found to be composed of coke-like benzene-insoluble organic matter and mineral matter with coke being the major constituent.

In order to determine the ability of the process to operate satisfactorily, experiments were conducted for two to three weeks in the temperature range 450-470°C with pressures at 13.9 to 24.2 MPa and with LHSV of 2. This work is reported in reference 6 where it is shown that the best operating conditions for the reduction of reactor deposits occur at the highest pressure and temperature. The pilot plant was operated for nearly 500 hours

at a pressure of 24.2 MPa and a temperature of 470°C, without any operational problems and with negligible solid deposits in the reactor and hot separator.

The present work was undertaken for two reasons: (a) to demonstrate that the plant could be operated without the formation of deposits at lower pressures (<24.2 MPa), recycling a portion of the heavy oil and (b) to show that increasing the linear velocity would have a beneficial effect in reducing reactor fouling since the liquid linear velocity increases with reactor scale-up.

LITERATURE SURVEY

Information on the reduction of coke formation and its effect on thermal hydrocracking together with the expected improvements in equipment due to heavy-oil recycle are documented in this section. Wolk et al. (7) observed that, for a coke-free condition, the temperature of the hot separator and the linear liquid velocity should be maintained between 260 and 427°C and 0.18 and 30 cm/sec, respectively. These conditions represent a LHSV of 1.6 to 275 for the present system. It has been observed that an increase in LHSV results in a decreased pitch conversion (5) which is undesirable, and an LHSV above 4 is impractical.

McColgan et al. (8) have observed that with a feed containing a high concentration of mineral matter, the bitumen could be hydrocracked at higher temperatures with no coking problem. Chervenak et al. (9) observed that a feed containing a large quantity of mineral matter (>1 wt %) has less tendency to coke compared with feed containing less than one wt %. They also noted that fouling of the reactor by coke deposition could be avoided if the hydrogen content of pitch in the liquid product was increased to more than 6.8 wt %. Athabasca topped bitumen* generally contains about 0.5 wt % mineral matter and in this case, it would be necessary to add an appropriate hydrogenation catalyst with the feed to increase the hydrogen content of pitch in the liquid product. This procedure would increase the process operating cost

* Topping of bitumen implies removal of light fractions. In this case all material boiling below 260°C has been removed.

because of the expensive catalyst and high hydrogen consumption, with its attendant high production cost.

Layng (10) claimed that if the reactor effluents were quenched, no coke deposited in the separator. This method reduced fouling of the hot separator but it had little effect on reactor fouling.

Wolk (11) claimed that, by maintaining the mineral matter content in the fluid between 4 and 10 wt %, coke deposition could be avoided. This could be achieved either by adding mineral matter to the feed or by recirculating the under-flow of the hot separator to the reactor bottom.

Schuman (12) suggested the use of coke carriers such as sand, quartz, alumina, zircon, beryl or bauxite. In this case, coke would deposit on the carriers instead of the reactor wall and would be carried out of the reactor with the reactor effluents. This procedure may increase operational problems associated with erosion.

Considering all of the above points, it was decided to recirculate a portion of heavy ends to the reactor bottom. This should result in the following improvements:

1. The linear liquid velocity in the reactor or hot separator should be much higher than the velocity based on fresh feed flow. The pitch conversion should not be affected significantly because of the constant LHSV based on fresh feed.
2. The mineral matter content of the reactor fluid should increase.
3. The velocity of the liquid at the bottom of the separator, where coke deposition is evident, should be greater by 7 or 8 times. This increased velocity should help in reducing coke deposition.
4. Because of higher liquid velocities, the liquid near the wall should be replaced more efficiently, preventing excessive coke formation on the reactor walls.

APPARATUS AND ANALYTICAL PROCEDURE

The apparatus, operation, analytical procedures and properties of the feed have been documented in previous reports (1,2).

RESULTS AND DISCUSSION

Experiments were conducted at two different temperatures*, 450 and 460°C ($\pm 1^\circ\text{C}$), to determine the effect of heavy-oil recycle. These runs will be referred to as R-450 and R-460, respectively. Reaction conditions for these runs were given in an earlier report (1) under pilot plant run numbers R-2-1-2 and R-2-2-4 respectively. The results of equipment inspections after the completion of the runs are given in Table 1. This table indicates that little coke deposited on reactor or hot separator walls after 300 hours of operation. To assess the effect of heavy-oil recycle it was necessary to analyse these runs on the basis of liquid flow, reactor-fluid analysis and analysis of deposits in the reactor after the completion of the run.

1. Liquid Flow in the Reactor and Separator

Feed and product evaporation in the reactor resulted in a lower apparent liquid flow rate than the actual feed rate. Consequently, the hold-up time for once-through runs was more than the reciprocal of the space velocity. Table 2 shows the values of the average liquid flow, residence time for the first feed pass and average residence time of the recycled oil. These calculations were based on a 25% gas hold-up in the reactor and a liquid volume of 300 cc in the separator (1). The recycle-oil flow was calculated from a heat balance around the preheater. The average residence time of the heavy oil was calculated by dividing the system liquid volume by the recycle-oil withdrawal rate. The density of the recycle oil was calculated to be approximately 1.0 g/cm^3 (13), and this number was used in the computations. Fouling of the reactor was evident in the case of once-through runs with a relatively long first-pass residence time. On the other hand, little coke was found in the reactor during the recycle runs in which the residence time for the first feed pass was shorter. It seems that the first feed-pass is important in determining the amounts of coke deposited in the reactor. Heavy oil, which stayed in the system from 4 to 5 hours on the

* One twenty-four hour recycle run at 440°C and LHSV of 0.5 was made, but is not included in this report.

average, is apparently more stable with respect to coke formation.

2. Reactor Fluid

Samples of the reactor fluid were taken at the bottom of the reactor, point 1; the middle of the reactor, point 3; and the top of the reactor, point 5. No reactor fluid samples were collected for short runs or from points 2 and 4 for recycle runs.

Table 3 shows the analysis of the reactor fluid for run R-450. The maximum build-up of mineral matter and insolubles occurred at the bottom of the reactor. Ash content of the feed was about 0.57 wt %. Because there was no withdrawal of RO for the first six hours of the run, the ash content rapidly increased to 3% in this time. The ash content of the reactor fluid at the bottom of the reactor was 6.34 wt % on the fifth day; this increased to 20.08 wt % and then decreased slightly in the later stages of the run. The decrease in the ash content of the reactor fluid was due to the removal of ash in reactor samples*. The concentration of sulphur, nickel, vanadium and insolubles followed a similar trend to that observed for ash (Fig. 1). The changes in the properties of the reactor fluid sampled at the middle of the reactor are shown in Fig. 2. The concentrations of mineral matter and insolubles were nearly constant at this point.

Table 4 shows the analyses of the reactor fluid for run R-460. Due to relatively shorter run time, reactor fluid samples were withdrawn on the fourth and ninth days only. The analyses of these samples show that the maximum build-up of mineral matter and insolubles was at the bottom of the reactor and the accumulation increased with operating time, consistent with run R-450.

The accumulation of solids in the reactor may have been due to the tendency for larger particles in the feed to settle if the flow of liquid and the turbulence induced by the gas were not sufficient to carry them out of the reactor. Alternatively, there may have been some tendency for solid particles to agglomerate because of adsorption of larger oil molecules such as asphaltenes and resins, which might act as binders.

* Sample volume is ~100 cc for reactor samples, and the portion of the reactor which has a high ash concentration is 300-500 cc by calculation using the results of successive reactor samples.

3. Analyses of Residue Samples Obtained from Different Sections of the Pilot Plant after Completion of Each Recycle Run

Table 5 shows analyses of the residue samples obtained after the completion of the recycle runs. In each case, the amount of residue obtained was only a few grams. Ash concentration was high, 51.1 and 17.7 wt % for runs R-450 and R-460, respectively, in samples collected from the reactor wall. The hydrogen content was greater in the sample obtained for run R-460. The samples obtained from the hot separator contained 50.6 wt % ash for R-450 and 37.9 wt % for R-460. Sulphur was about 10 wt % for each of the two runs. The hydrogen content of these samples was less than the samples obtained from the reactor. The samples collected from the thermowell (hot separator) were similar in composition to those obtained from the walls of the hot separator.

The quantity of total residue obtained was less than for R-450 although the reactor temperature was greater for run R-460. The ash, sulphur and insoluble contents were greater in the inspection samples obtained from run R-450. The only difference in the pilot plant for these runs was the inside diameter of the pump suction transfer line for the recycle oil. The inside diameter of this line was 0.3175 cm for run R-450, whereas it was 0.481 cm for run R-460. The recycle-oil flow rate increased to 12,700 g/h in R-460 from 9,060 g/h in R-450 because of the increased size of the transfer line. The turbulence in the reactor increased with increased recycle-oil flow and possibly suppressed coke deposition during run R-460.

4. General Discussion

The feed in the present investigation had only 0.57 wt % solids and the pitch (liquid product) had less than 6.8 wt % hydrogen content (about 6.0 wt %) but even after being on-stream for about 21 days, little coke was found in the reactor, which is contrary to the findings of Chervenak et al. (9).

The liquid velocities in the reactor and in the separator were less than 0.6 cm/sec and the separator temperature was 450°C which corresponds to the coking zone as suggested by Wolk et al. (7), but the separator was clean on inspection after the run.

On a once-through basis, the residence time can be considerably longer than the reciprocal of the space velocity because of the vaporization of the feed and liquid products. The average residence time for the first feed pass (0.3 h) in the recycle runs was much shorter than for a single pass or once-through operation (1.5 h). However, the mean residence time of the heavy oil can be considerably longer (3.8 h); some of the heavy oil could be in the system for one pass (0.3 h) and theoretically some could be in for the entire run. The results of the present investigation suggest that the heavy oil which stayed in the system for more than 3.8 h is more stable with respect to coke formation.

The ash content at all points of the reactor was more than 4 wt %. There was mineral matter build-up in the reactor to above the 4 wt % level in earlier runs at 13.89 MPa without recycle (6). However, in these runs up to one-third of the reactor was filled with coke after 21 days of operation. It would therefore appear that the build-up of mineral matter in the reactor was not the only contributing factor in the reduction of coke deposits. It is possible that random motion of inert particles in the ebullient mass caused them to rub against each other and against the walls of the vessel so that formation of deposits was minimized. This scouring action could help to prevent agglomeration of the particles and plugging of the vessel and would be expected to be more intense for the recycle runs than for the once-through runs because of the higher liquid velocity. It appears that a high ash content in the reactor is not the only parameter in reducing reactor fouling; others such as liquid linear velocity are equally important.

CONCLUSIONS

The fouling of the reactor or hot separator due to coke and sludge deposits could be very much reduced by recirculating a portion of the heavy ends. Although the hydrogen content of pitch in liquid product was less than 6.8 wt %, little coke was found in the reactor or hot separator even after more than 300 hours of operation. This is contrary to the findings of Chervenak et al. (9).

The quantity of deposits found in the reactor and hot separator after shut-down was less for the higher temperature run. These deposits were found to contain less ash and more coke at the higher temperature because of an increased recycle-oil flow rate for the higher temperature run.

The shorter first feed-pass residence time coupled with high ash content in the reactor fluid are possible reasons for reduced coke deposition.

High linear velocities have been shown to have a beneficial effect on reactor operation. This is important when considering scale-up since larger reactors will have higher linear velocities for the same LHSV than smaller reactors.

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TABLE I

Condition of Equipment After Completion of the Runs

	R-450	R-460
Reactor wall	Light dusting of powdery material on the walls	Coated with black powder
Reactor outlet cap	Hard foam-like deposit	Small amount of bubbly oil
Reactor inlet cap	Light dusting of powdery material	Clean
Hot receiver	Black powdery material on the walls less than 2 mm thick on the lower 1 m portion	Thin layer of black powder in the lower 1 m portion
Down-pipe in hot separator	Caked with hard black material	Thin layer of black powder
Preheaters	Caked with black material	Small amount of solids on the wall

TABLE 2

Average Liquid Flows, Velocities and Residence Times

Run No.	82-3-1*	R-450	67-4-1*	R-460
Average liquid flow, g/h	2192	11261	1955	14906
Recycle oil withdrawal rate, g/h	-	976	-	748
Superficial liquid velocity in the reactor, cm/sec	0.053	0.274	0.048	0.360
Superficial average residence time for the first pass, h	1.54	0.30	1.53	0.23
Total residence time for the recycle oil, h	-	3.8	-	5.4
Liquid velocity in the separator, cm/sec	0.059	0.244	0.037	0.330

* Once-through runs at 450°C (82-3-1) and 460°C (67-4-1) were described in a previous report (2).

TABLE 3

Properties of the Reactor Fluid for Run R-450

Sample Point No.	Day No.	PI * wt %	BI ** wt %	Ash wt %	CCR *** wt %	S wt %	Ni ppm +	V ppm +	Asphaltene wt %	Coke wt %
1	5	36.6	20.8	6.34	35.8	4.87	2353	8763	15.8	14.46
	9	40.6	23.4	12.94	43.5	7.52	9619	3.37 %	17.2	10.46
	14	46.7	32.0	19.92	49.2	10.06	1.82 %	5.03 %	14.7	12.08
	17	46.0	32.1	20.08	48.2	10.66	2.13 %	6.41 %	13.9	12.02
	19	42.0	26.1	16.63	45.3	9.20	1.59 %	4.89 %	15.9	9.47
	21	38.0	21.8	13.29	40.1	8.20	1.23 %	3.21 %	16.2	8.51
3	5	34.0	7.8	4.14	32.5	4.10	560	2291	26.2	3.66
	9	33.0	12.5	4.05	31.8	4.06	371	1451	20.5	8.45
	14	32.0	11.9	4.06	31.0	3.79	376	1288	20.1	7.84
	17	33.5	12.5	3.46	29.8	3.91	370	1318	21.0	9.04
	19	32.0	10.9	4.33	31.3	3.96	377	1340	21.1	6.57
	21	30.4	9.9	3.79	29.7	4.04	309	1015	20.5	6.11

Continued

TABLE 3 (Cont'd)

Properties of the Reactor Fluid for Run R-450

Sample Point No.	Day No.	PI * wt %	BI ** wt %	Ash wt %	CCR *** wt %	S wt %	Ni ppm +	V ppm +	Asphaltene wt %	Coke wt %
5	5	-	-	0.88	2.02	4.26	-	-	-	-
	9	-	-	-	-	-	-	-	-	-
	14	32.5	12.7	4.08	31.4	3.78	310	1832	19.8	8.62
	17	32.1	11.2	3.76	30.9	3.84	311	1060	20.9	7.44
	19	32.5	11.7	4.48	32.0	3.77	368	1356	20.8	7.22
	21	27.5	9.8	3.46	29.2	3.99	241	827	17.7	6.34

* Pentane-insoluble ** Benzene-insoluble *** Conradson carbon residue + per cent where marked, otherwise parts per million (ppm)

Asphaltene = PI-BI
Coke = BI-Ash

TABLE 4

Properties of the Reactor Fluid for Run R-460

Sample Point No.	Day No.	PI * wt %	BI ** wt %	Ash wt %	CCR *** wt %	S wt %	Ni ppm	V ppm +	Asphaltene wt %	Coke wt %
1	4	44.4	18.9	5.75	39.7	4.13	1166	4095	25.5	13.15
	9	42.3	23.8	9.36	44.5	5.76	5402	1.85 %	18.5	14.43
3	4	42.7	19.0	5.60	41.0	3.86	676	2195	23.7	13.40
	9	40.9	16.8	5.43	40.2	4.09	1072	4027	24.1	11.37
5	4	45.7	20.8	6.48	43.3	3.76	670	2108	24.9	14.32
	9	42.6	26.0	5.17	43.5	4.27	1660	5522	16.6	20.83

* Pentane-insoluble ** Benzene-insoluble *** Conradson carbon residue

+ per cent where marked, otherwise parts per million (ppm)

Asphaltene and coke defined on page 13

TABLE 5

Analyses of the Residue Samples Obtained from
Different Sections of the Pilot Plant

Section		Wall				Thermocouple Well (Hot Separator)	
		Reactor		Hot Separator		R-450	R-460
Run Number:		R-450	R-460	R-450	R-460		
PI *	wt %	97.3	90.3	97.2	99.1	98.2	95.0
BI **	wt %	92.3	88.0	94.3	98.0	95.2	89.7
Ash	wt %	51.1	17.7	50.6	37.9	55.3	31.9
Asphaltene	wt %	5.0	2.3	2.9	1.1	3.0	5.3
Coke	wt %	41.2	70.3	43.7	60.1	39.9	57.8
CCR ***	wt %	97.0	87.1	98.3	93.4	84.9	95.2
S	wt %	20.48	5.24	10.44	9.0	12.18	7.55
Ni	wt %	4.5	1.0	2.0	1.5	2.5	1.6
V	wt %	13.2	3.2	5.9	4.8	7.5	4.5
N	wt %	1.1	1.52	1.06	0.35	1.02	1.44
C	wt %	39.07	74.51	36.05	50.94	36.05	56.22
H	wt %	1.94	3.95	1.88	2.46	1.88	2.83
Sp gr		2.48	-	2.15	-	2.27	-

*PI Pentane-insoluble ** BI Benzene-insoluble

*** CCR Conradson carbon residue

Asphaltene and coke defined on page 13

TABLE 5 (cont'd)

Analyses of the Residue Samples Obtained from
Different Sections of the Pilot Plant

Section		HCP Bottom Cap		Bottom of HCP, first 2'		Preheaters	
		R-450	R-460	R-450	R-460	R-450	R-460
PI *	wt %	79.1	-	97.3	-	89.2	-
BI **	wt %	62.9	-	91.5	-	84.3	-
Ash	wt %	28.6	36.3	45.7	-	60.1	-
Asphaltene	wt %	16.2	-	5.8	-	4.9	-
Coke	wt %	34.3	-	45.8	-	24.2	-
CCR ***	wt %	69.1	97.7	85.1	-	86.4	-
S	wt %	7.08	-	12.52	-	24.62	-
Ni	wt %	2.2	-	2.7	-	7.3	-
V	wt %	3.3	-	8.3	-	14.9	-
N	wt %	1.42	-	1.23	-	0.6	-
C	wt %	61.44	-	44.61	-	23.52	-
H	wt %	4.15	-	2.36	-	2.27	-
Sp gr		1.58	-	2.07	-	-	-

Section	Top Cap Reactor		Bottom Cap Reactor	
	R-450	R-460	R-450	R-460
Ash wt %	36.3	-	-	20.6
CCR*** wt %	69.4	-	-	91.0

* PI Pentane-insoluble ** BI Benzene-insoluble *** CCR Conradson carbon residue

Asphaltene and coke defined on page 13

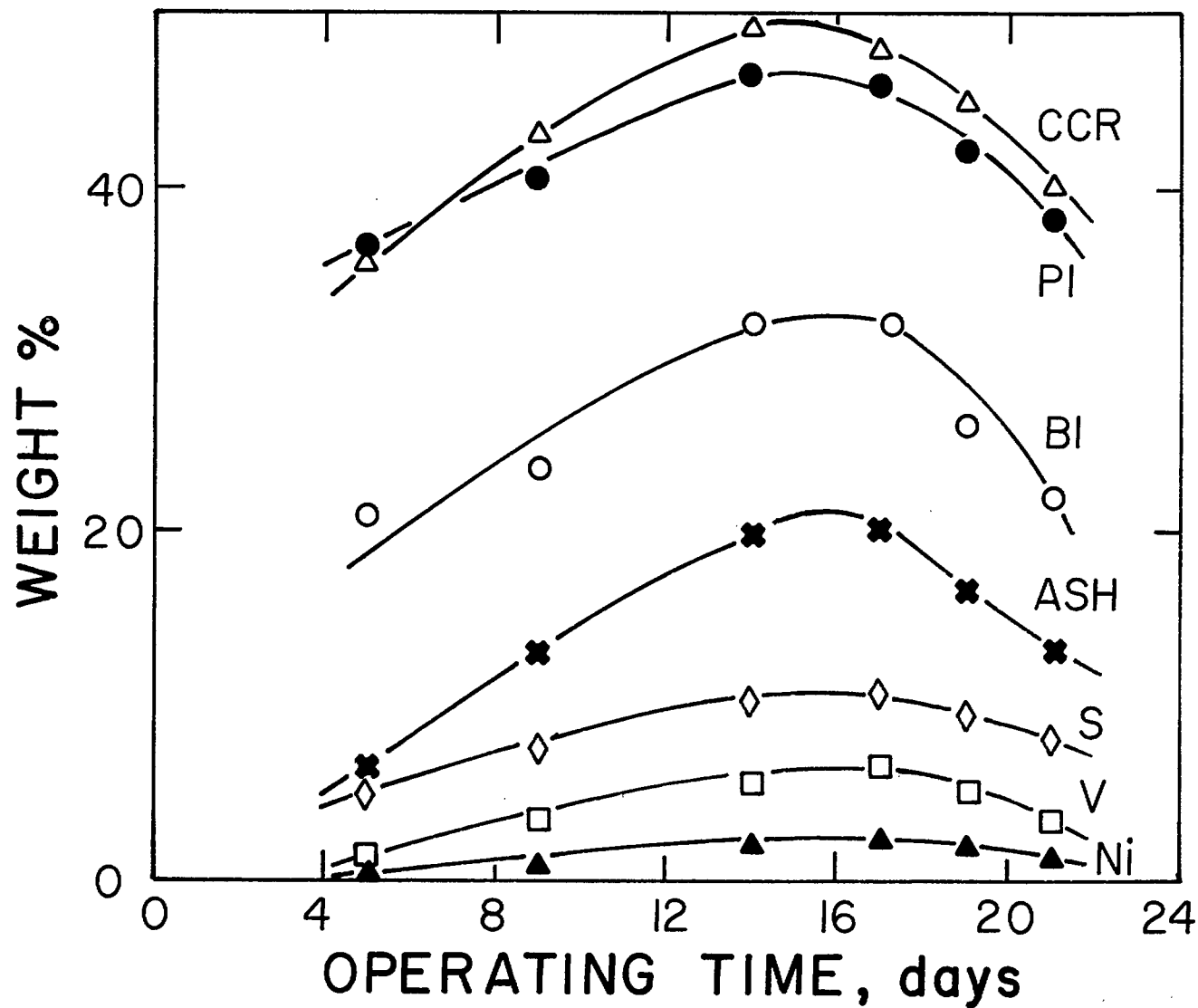


FIGURE 1 - Variations in the Reactor Fluid Properties for Run R-450
Samples Taken at the Bottom of the Reactor

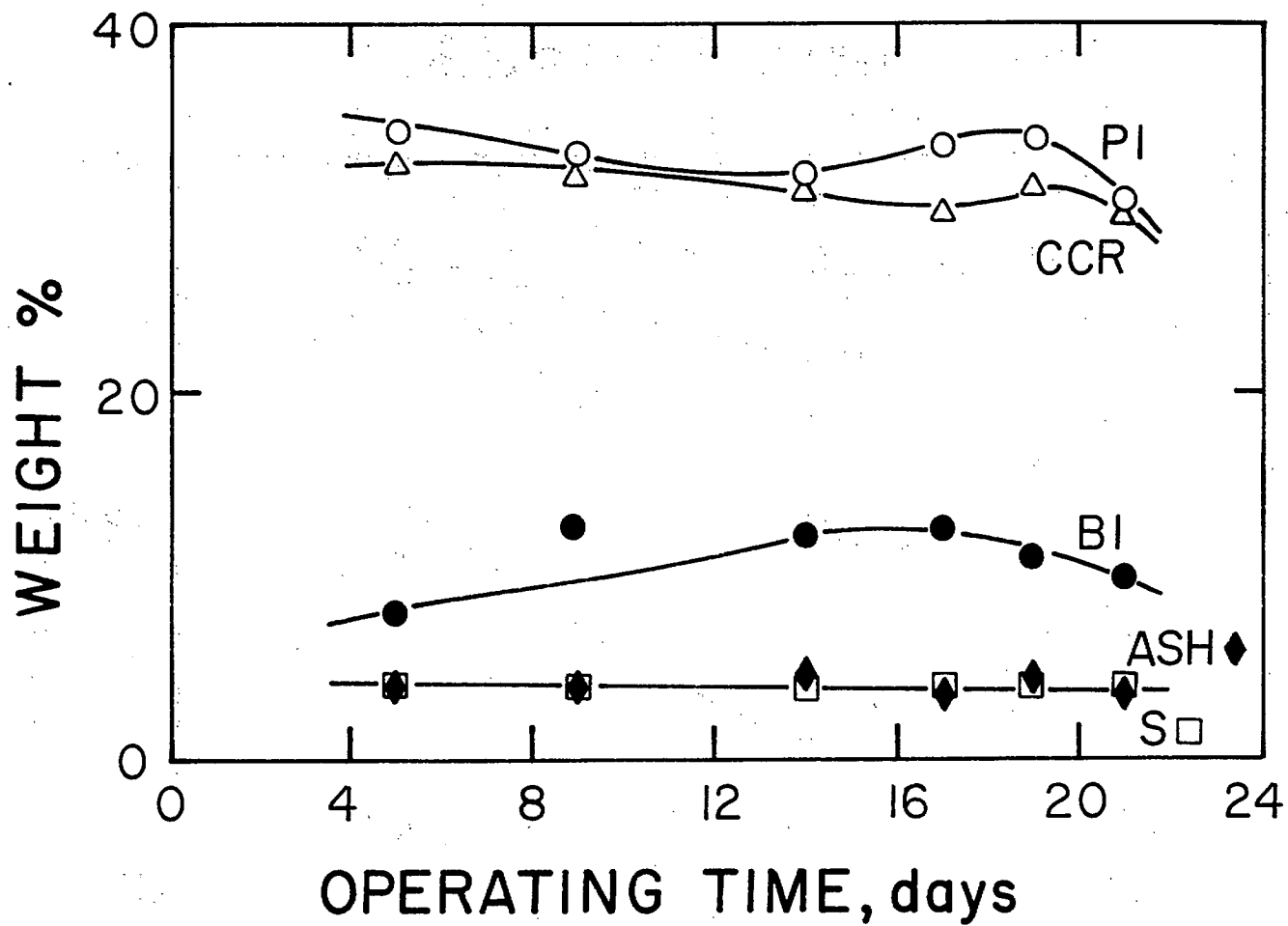


FIGURE 2 - Variations in the Reactor Fluid Properties for Run R-450
Samples Taken Half-way up the Reactor

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