Hold – not to be distributed – all copies with author

Energy, Mines and Resources Canada
Énergie, Mines et Ressources Canada

CANMET
Canada Centre for Mineral and Energy Technology
Centre canadien de la technologie des minéraux et de l'énergie

EFFECT OF FINELY DIVIDED SUB-BITUMINOUS COAL ADDITION ON HYDROCRACKING PILOT PLANT OPERATION

R. Ranganathan, B.B. Pruden and J.M. Denis

JUNE 1977

ENERGY RESEARCH PROGRAM
ENERGY RESEARCH LABORATORIES
CANMET REPORT 77-62

Available by mail from:

Printing and Publishing
Supply and Services Canada,
Ottawa, Canada K1A 0S9

CANMET
Energy, Mines and Resources Canada,
555 Booth St.,
Ottawa, Canada K1A 0G1

or through your bookseller.

Catalogue No. M38–13/77-62 Price: Canada: $1.50
ISBN 0-660-10018-5 Other countries: $1.80

Price subject to change without notice.

En vente par la poste:

Imprimerie et Édition
Approvisionnements et Services Canada,
Ottawa, Canada K1A 0S9

CANMET
Énergie, Mines et Ressources Canada,
555, rue Booth
Ottawa, Canada K1A 0G1

ou chez votre libraire.

N° de catalogue M38–13/77-62 Prix: Canada: $1.50
ISBN 0-660-10018-5 Autres pays: $1.80

Prix sujet à changement sans avis préalable.

EFFECT OF FINELY DIVIDED SUB-BITUMINOUS COAL ADDITION ON HYDROCRACKING PILOT PLANT OPERATION

by

R. Ranganathan*, B.B. Pruden** and J.M. Denis***

ABSTRACT

The thermal hydrocracking process was evaluated when Athabasca bitumen was reacted with hydrogen, with or without the addition of sub-bituminous coal. The hydrocracking was carried out at 10.44 MPa pressure and temperatures of 450°C and 455°C.

The addition of coal improved plant operation by decreasing the total system pressure-drop and by reducing the amount of solids deposited. The total system pressure-drop was only about 400 kPa in the presence of coal, whereas without coal the pressure-drop was above 1200 kPa. Only 132 g of solids were deposited with coal compared with 6600 g without it. The coke precursors (asphaltenes and benzene insolubles) were also found to be less abundant in the presence of coal. The results clearly indicated that the addition of coal would make operation of the thermal hydrocracking process possible at low pressures.

** Project Leader, ** Section Head and *** Manager, Synthetic Fuel Research Laboratory, Energy Research Laboratories, Canada Centre for Mineral and Energy Technology, Dept. of Energy, Mines and Resources, Ottawa, Canada.*

L'EFFET DE L'ADDITION DE CHARBON SOUS-BITUMINEUX FIN SUR LES OPERATIONS D'HYDROCRAQUAGE D'UNE USINE PILOTE

par

R. Ranganathan*, B.B. Pruden** et J.M. Denis***

SOMMAIRE

Le procédé d'hydrocraquage thermique a été évalué lorsque que le bitume d'Athabasca avait réagi avec l'hydrogène, avec ou sans l'addition du charbon sous-bitumineux. L'hydrocraquage a été effectué à une pression de 10.44 MPa et à des températures de 450° et 455°C.

L'addition du charbon a amélioré les opérations de l'usine en décroissant l'abaissement total de la pression du système et en diminuant la quantité de résidus solides. L'abaissement total de la pression du système n'est que de 400 kPa en présence du charbon; en l'abscence de celui-ci, l'abaissement de la pression était supérieure à 1200 kPa. Seulement 132 g de solides ont été déposés avec le charbon et 6600 g en son abscence. Les précurseurs du coke, les asphaltènes et les benzènes insolubles, étaient moins abondants en présence du charbon. Les résultats démontrent clairement que l'addition du charbon rendrait possible le procédé d'hydrocraquage thermique à basse pression.

** Responsable des projets, **Chef de Section et ***Gérant, Laboratoire de recherche sur les carburants synthétiques, Laboratoires de recherche sur l'énergie, Centre canadien de la technologie des minéraux et de l'énergie, Ministère de l'Energie, des Mines et des Ressources, Ottawa, Canada.*

CONTENTS

	Page
Abstract	i
Sommaire	ii
Introduction	1
Experimental	2
Feedstock and Coal	2
Operational Procedures	2
Results and Discussion	3
Plant Operation	3
Reactor Fluid	5
Accumulation of Solid Deposits	5
Heavy Oil	6
Light Oil	6
General Discussion	7
Conclusions	9
Acknowledgements	9
References	10

TABLES

No.		Page
1.	Properties of Bitumen Feedstock	11
2.	Properties of White Wood Coal	12
3.	Reaction Conditions for Thermal Hydrocracking Experiments	13
4.	Properties of Reactor Fluid	14
5.	Details of Solid Deposits at the End of Run	15
6.	Analysis of Solid Deposits	16
7.	Preheater and Reactor Temperatures	17

FIGURES

1.	A Schematic of the Pilot Plant	18
2.	Feed, Product, Heavy-oil and Light-oil Flow Rates for Run NA 450	19
3.	Feed, Product, Heavy-oil and Light-oil Flow Rates for Run CA 450-4	20
4.	Feed, Product, Heavy-oil and Light-oil Flow Rates for Runs CA 450-5 and CA 455	21
5.	Total Pressure Drop in the Thermal Hydrocracking Pilot Plant	22
6.	Hydrogen Consumption and Hydrocarbon Gas Make During Thermal Hydrocracking	23
7.	Accumulation of Benzene Insolubles and Ash in the Bottom of the Reactor	24
8.	Heavy-oil Properties During Run NA 450	25
9.	Heavy-oil Properties During Run NA 450	26
10.	Heavy-oil Properties During Run CA 450-4	27
11.	Heavy-oil Properties During Run CA 450-4	28
12.	Heavy-oil Properties During Runs CA 450-5 and CA 455	29
13.	Heavy-oil Properties During Runs CA 450-5 and CA 455	30
14.	Light-oil Properties During Run NA 450	31
15.	Light-oil Properties During Run CA 450-4	32
16.	Light-oil Properties During Runs CA 450-5 and CA 455	33

INTRODUCTION

One of the objectives of CANMET (the Canada Centre for Mineral and Energy Technology) is to develop an economical process for upgrading bitumen and heavy oils. This is in keeping with the Energy Research Program of the Department of Energy, Mines and Resources and its policy of ensuring the effective use of Canada's mineral and energy resources. Thermal hydrocracking has been developed at the Energy Research Laboratories as a method of upgrading Athabasca bitumen (1).

The thermal hydrocracking process continues to present operational problems due to the formation of coke deposits in the reactor. Deposits form at the top of the reactor where the partial pressure of hydrogen and ash content are lowest. The deposition of coke affects plant operation by increasing the pressure-drop and fluctuations in gas flow, lowering the product quality, and eventually plugging the system. Plant operation is considered satisfactory when there are fewer fluctuations in pressure, a small pressure-drop, constant product quality, and constant gas flow. Several attempts have been made to improve plant operation. It has been reported that a higher operating pressure of 24.2 MPa reduced reactor fouling and improved plant performance (1). However, operating at high pressures involves higher capital and operating costs (2). Khulbe et al. (3) found that recycling a portion of the heavy-oil product reduced coke build-up on the reactor walls. The decrease was attributed to higher ash content and to shorter residence time of the liquid in the reactor.

Plant operation can be improved by either suppressing coke formation or preventing coke build-up on the reactor walls. Hence, coal would function to reduce the build-up of coke by acting as both a catalyst and scouring agent. It is also possible that some coke precursors might stick to the coal and leave the reactor, thereby helping to prevent coke build-up. Such a "getter" mechanism was described by Ternan et al. (4) who found from microscopic studies that coal particles act as sites for the deposition of coke. This work was based on bench-scale studies where bitumen was hydrocracked in the presence of a fixed bed of coal particles.

Khulbe et al. (5) used a Co-Mo-Al catalyst on a bituminous coal base in an attempt to improve pilot plant operation and reduce the deposi-

tion of solids. Even though improvements in pitch conversion and desulphurization resulted, significant solid deposits were found in the reactor. They recommended evaluating lower ranks of coals with higher softening temperature, as the bituminous coal could simply have melted and acted as a glue to cement mineral matter to the wall. It is possible that coke deposits in the reactor could also be caused by selective accumulation of Co-Mo metals in the reactor liquid. Even though the use of Co-Mo led to increased desulphurization and higher hydrogen consumption, coke deposition resulted.

In this report, the results obtained using a sub-bituminous coal without any catalytic additive are described. The effects of coal addition on plant operation and coke deposition are reported. The effect of adding coal on product quality and product distribution will be discussed in a subsequent report.

EXPERIMENTAL

Feedstock and Coal

The feedstock was topped Athabasca bitumen which had previously been separated from the tar sands of Alberta, supplied by Great Canadian Oil Sands Ltd., Fort McMurray, Alberta. The coal was of a sub-bituminous variety and was supplied by the White Wood Coal Mine, Calgary Power Ltd., Calgary, Alberta. Properties of the feedstock and coal are listed in Tables 1 and 2 respectively. The coal was crushed, dried at 100°C and sieved through 200 mesh (-74 μm). To obtain a homogeneous mixture, i.e., 2% coal in bitumen, 550 g of coal was mixed with about 4 kg bitumen at 120°C using a Hobart mixer. The mixture was initially stirred at low speed for 5 minutes and then at high speed for 5 minutes. It was poured into a feed tank containing about 30 kg of bitumen and mixed with a 3-bladed propeller of 0.1 m diam operating at 200 rpm (3.3 r/s). The tank was maintained at 110°C in a nitrogen atmosphere under a pressure of 240 KPa.

Operational Procedures

A schematic diagram of the thermal hydrocracking pilot plant is shown in Fig. 1. The reactor was 0.038 m in diam and 3.96 m long and has been described in detail with its equipment in earlier reports (1,3). For

start-up, the reactor and hot separator were heated to 350°C with hydrogen circulating at 5.58 m^3API/t. The reactor was then heated to 450°C while bitumen was fed at a rate of 13.5 kg/h. When reaction temperature was reached, additional bitumen containing 2% coal was introduced. The procedures for analysis of products were similar to those described for thermal hydrocracking (3). Additional samples were taken at the top, middle, and bottom of the tubular reactor to determine the distribution of ash and coke precursors.

The experiments and reaction conditions are listed in Table 3. A three-week run, CA 450-4, at 450°C, a liquid hourly space velocity of 3.0, and pressure of 10.44 MPa was conducted without adding coal, to assess plant performance. A three-week run, CA 455, at 455°C was also carried out with 2% coal additive. For this run, a slow start-up procedure was used because preliminary runs indicated that accumulation of mineral matter was helpful in preventing coke deposition. The experiment was carried out at 450°C for three days, after which the temperature was increased to 455°C. Comparisons of results for runs without coal, with coal at 450°C, and with coal at 455°C are given in the following section. As described in previous reports (1,5), the product was obtained in two streams, i.e., heavy oil and light oil. The heavy oil contained all of the pitch and some distillate, and the light oil contained no pitch and the remainder of the distillate. The proportion of the total distillate material in the light oil depended on the hot separator temperature and the gas/liquid ratio in the separator. The properties of the light and heavy oils are important to plant operation and are discussed in the following section.

RESULTS AND DISCUSSION

Plant Operation

The cause of shutdown for each run is given in Table 3. In previous studies with a coal-based catalyst, Khulbe et al. (5) suggested that the method of mixing coal and bitumen should be improved to prevent formation of small lumps. The method of preparing the coal slurry was improved for the experiments reported here by using minus 200-mesh coal (minus 74-μm)

and the Hobart mixer as described in the experimental section. This method improved operations considerably, but small lumps of agglomerated coal occasionally caused pumping problems and eventual shutdown of run CA 450-2. Run CA 450-1 was shut down because a hydrogen bypass valve was accidentally left open, resulting in hydrogen starvation in the reactor. Run CA 450-3 was shut down because of problems in controlling the liquid level in the hot separator. The CA 450-4 and CA 455 runs were completed without any problems. The run without any coal additive, NA 450, was shut down after 16 days of operation, when the recycle pump stopped operating properly. However, failure was anticipated and the plant shut down normally. The results of CA 450-1, CA 450-2 and CA 450-3 will not be discussed further because these runs were shut down after operating for only short periods.

The feed flow rates for NA 450, CA 450-4 and CA 455 were reasonably constant during operation, fluctuating about ±4%. It is seen in Fig. 2, 3 and 4 that for slight variations in feed rate, corresponding changes were observed in total product, heavy-oil, and light-oil flow rates. In previous work with a coal-based Co-Mo catalyst, it was observed that heavy oil increased with time, resulting from a decrease in conversion (5). It is seen from Fig. 2, 3 and 4 that no such increase took place.

The variation in total system pressure-drop is shown in Fig. 5 for runs NA 450, CA 450-4, CA 450-5 and CA 455. Run NA 450 showed three times the pressure-drop of the runs with coal additive. The pressure-drop for all runs was higher for the first four days of operation. After the initial period, the pressure-drop was constant. The reactor temperature during the first four days was at 450°C for CA 450-5 and was subsequently increased to 455°C for CA 455. This start-up was used in an attempt to build up coal and mineral matter in the reactor at less severe conditions. The pressure drop was slightly higher for CA 455 than for CA 450-4. This can be attributed to a higher operating temperature. Comparing NA 450 with CA 450-4 and CA 455 shows that adding coal to bitumen significantly decreases the overall pressure-drop caused by coking in the reactor, preheaters and the hot reactor-receiver crossover line, i.e., 400 KPa compared with 1200 KPa.

The hydrogen consumption and hydrocarbon gas make are shown in Fig. 6. At 450°C, hydrogen consumption was higher when coal was present.

The hydrogen consumption during start-up of CA 450-4 was close to that of NA 450 but increased with operating time and then stabilized. The hydrocarbon gas make for the thermal hydrocracking was the same at 450°C for runs with and without coal. After the first four days of operation, the hydrogen consumption and hydrocarbon gas make remained constant.

Reactor Fluid

The accumulation of ash and benzene insolubles in the reactor fluid is shown in Fig. 7. These samples were taken at the bottom of the reactor. It is seen that, for the run without coal, NA 450, the ash and benzene insolubles increased slowly. For the experiments in which coal was added, CA 450-4 and CA 455, the ash content increased rapidly and stabilized after 7 days. For benzene insolubles, a rapid increase followed by a slow increase was observed. The ash content of the reactor fluid in CA 450-4 was about 3 times that of NA 450. The properties of reactor fluid at different locations in the reactor are listed in Table 4. The asphaltene contents are lower for runs with coal than for runs without coal. This indicates that asphaltenes are converted to either BIOR (benzene insoluble organic residue) or oil, in the presence of coal.

Khulbe et al. (5) observed that the ash collected from the fluid in the bottom of the reactor increased to 67.7 wt % in 11 days of operation when a Co-Mo-based coal catalyst was used. This required withholding of catalyst to control ash accumulation. For the coal runs reported here, it was observed that ash content stabilized after 7 days and changes in operation were not necessary to control ash accumulation.

Accumulation of Solid Deposits

At the end of each run, the reactor and other parts of the pilot plant were checked and solid deposits were collected from the reactor and hot separator. Details of the deposits for each run are given in Table 5. Run NA 450 had hard coke deposits on the walls of the reactor and hot receiver. The experiments with coal produced significantly less deposits compared with runs without coal. For run NA 450, the solid deposits amounted to 6600 g after 16 days compared with 132 g after 21 days for CA 450-4 and 940 g after 22 days for runs CA 450-5 and CA 455 combined. Pruden et al. (1) reported

that, for thermal hydrocracking, a pressure of 24.1 MPa is necessary to reduce coke deposit. This indicated that coal addition improved plant operation by reducing solid deposits by a factor of 50 and allowed smooth operation at low pressures. Further, the microscopic studies showed that the solid deposits from CA 450-4 and CA 455 contained coal along with coke, whereas the deposits in NA 450 contained mostly coke.

Samples of the solid deposits obtained from CA 450-4 and CA 455 were analyzed and the analyses are given in Table 6. It is interesting to note that benzene insoluble, pentane insoluble, and ash contents for solids from the top section are higher than for solids from the bottom section. This trend is opposite to that observed for reactor liquid samples in Table 4. This trend is also the reverse of that observed in experiments with no solids addition, and further work is necessary to determine the nature of these deposits. It is surprising that the solids in the bottom section contained less ash than the reactor liquid samples. Obviously this is not simply caused by reactor liquid which has adhered to the wall on shutdown.

Heavy Oil

The properties of heavy-oil products are plotted in Fig. 8 to 13. Specific gravity, ash, vanadium, nickel, and sulphur of the heavy-oil product remained fairly constant during all runs. The product from runs with coal contained only slightly higher ash than did the product from the run without coal. The pentane insolubles and benzene insolubles were lower for the heavy oil from CA 450-4 than for the heavy oil from NA 450. It is seen from Fig. 11 that, for CA 450-4, the benzene insolubles decreased with operating time and then stabilized. However, for CA 455, the benzene insolubles appeared to increase towards the end of the run as can be seen in Fig. 13.

Light Oil

The properties of light-oil product are given in Fig. 14 to 16. The specific gravity of light-oil product from CA 450-4 was slightly higher than from NA 450. The nitrogen content was also slightly higher for light oil in CA 450-4, indicating that nitrogen compounds from the higher boiling fractions were transferred to lower boiling fractions. The sulphur contents for CA 450-4 light oil were initially high but decreased with operating time; on the other hand, nitrogen content appeared to increase with operating time.

GENERAL DISCUSSION

Operation of the pilot plant was extremely smooth when coal was added to the feed. The system pressure drop was low and temperatures were steady. It is seen from Table 7 that, for NA 450, the skin temperature at level 4 had to be raised by about 20°C to maintain reactor temperature. Preheater 3 also showed a drop in temperature of about 25°C suggesting a significant drop in heat transfer coefficient. These observations suggested solid deposits in the preheater and top section of the reactor. However runs CA 450-4 and CA 455 showed that increases in skin temperature were not necessary to maintain reactor temperature. Khulbe et al. (5) found that a 30°C increase in skin temperature at the top reactor section was necessary when a Co-Mo-Al/coal catalyst was used. The results reported here suggest that the performance of coal alone is better than with a Co-Mo-Al/coal catalyst. However the coal used by Khulbe et al. was a bituminous coal, whereas in the work reported here, a sub-bituminous coal was used. Additional work is necessary to establish the effects of different coal types.

It is seen from Fig. 7 that ash content in the reactor liquid in run CA 450-4 stabilized at 40% after about seven days. It is also seen that if all the coal ash and feed ash remained in the reactor, the reactor would have been full of solids within one day of operation. This calculation is based on voidage obtained from equations developed by Shah et al. (6). However, the experimental results showed that only a part of the ash remained in the reactor. Khulbe et al. (5) reported that when Co-Mo-Al/coal was used, the ash content increased to more than 60% in 11 days. They withheld the catalyst addition to prevent further build-up of ash in the reactor. It was also observed that the main constituent of the ash was Co-Mo catalyst.

The solids collected at the end of the runs showed them to be considerably reduced when coal was used. In CA 450-4 it was found that formation of solid deposits was in a swirl-like pattern, suggesting that some of the deposits (possibly coal) were left on the reactor wall during dumping of the reactor liquid. It should be noted that the solid deposits would also contain some coal along with coke.

Comparing the results with those from the Co-Mo-Al/coal runs indicated that the reduction of solid deposits was caused by differences in coal type or by adding Co-Mo-Al to the coal catalyst. It has been reported in the literature that passivation with hydrogen sulphide of the stainless steel reactor containing nickel, chromium and molybdenum reduces coke deposition for thermal cracking (7,8). Strong chemisorption of nitrogen compounds on the metallic components of the catalyst could also result in coke formation. Hence, it is possible that Co-Mo-Al in the coal catalyst might have been responsible for coke deposition in the previous studies (5). However, differences in coal type might also have been responsible for better operation. It is necessary to carry out studies with different types of coals, such as lignite, and with other catalysts like ferrous sulphate/coal, to obtain more information on the reduction of reactor fouling.

The results showed that asphaltene content of the reactor liquid was lower for runs with coal compared with runs without coal. Further, the pentane insolubles and benzene insolubles in heavy oils were lower for runs with coal. This is surprising because adding coal should result in increased pentane and benzene insoluble constituents. It is possible, however, that the reduction in pentane and benzene insolubles might have been caused by catalytic action of coal minerals.

CONCLUSIONS

Operation of thermal hydrocracking pilot plant improved considerably with the addition of coal because of low pressure-drop and reduced solids deposition. The system pressure-drop was only 400 KPa in the presence of coal, whereas with no coal, the pressure-drop was above 1200 KPa. The solid deposits in the reactor were only 132 g compared with 6600 g for runs without coal. It is significant to note that the amount of coke precursors was also reduced. Earlier reports indicated that, in the absence of coal, pressures of 24.1 MPa would be required to reduce coke deposits for thermal hydrocracking. The encouraging results obtained by adding coal show that successful operation can be achieved at lower pressures.

ACKNOWLEDGEMENTS

The authors wish to thank M.P. Pleet, R. Beer, C.A.Q. McNabb and the pilot plant staff for their cooperation and efforts. The contribution of E. Furimsky and his laboratory staff is gratefully acknowledge. The assistance of G. Noel in process calculations and of A. St. Louis for drafting is much appreciated.

REFERENCES

1. Pruden, B.B., Logie, R.B., Denis, J.M. and Merrill, W.H. "Thermal hydrocracking of Athabasca bitumen - reduction of reactor fouling"; CANMET Report 76-33; 33 p; Aug. 1976.

2. Stuckey, A.N., Fant, B.T., Duir, J.H. and Mickelson, G.A. "Operation VGO desulphurization unit at low pressure can help cut costs"; Adv. in Desulph. of Pet. Prod.; The Pet. Publ. Comp., Oklahoma; p 49-52; 1973.

3. Khulbe, C.P., Pruden, B.B., Denis, J.M. and Merrill, W.H. "Pilot plant thermal hydrocracking of GCOS (Great Canadian Oil Sands) Bitumen: 1. The effect of heavy oil recycle on the plant operation"; CANMET Report 76-28; 26 p; June 1976.

4. Ternan, M., Nandi, B.N. and Parsons, B.I. "Hydrocracking Athabasca bitumen in the presence of coal Part I: A preliminary study of the changes occurring in the coal"; Mines Branch Research Report R-276; 30 p; Oct. 1974.

5. Khulbe, C.P., Pruden, B.B. and Denis, J.M. "Hydrocracking of Athabasca bitumen: Effect of coal-base catalyst on pilot plant operation"; CANMET Report 77-35 ; 22 p; Jan. 1977.

6. Shah, A., Pruden, B.B. and Denis, J.M. "Thermal hydrocracking of Athabasca bitumen: Correlation of reactor voidage in vertical two-phase flow"; CANMET Report 77-48 ; 33 p; Dec. 1976.

7. Crynes, B.L. and Albright, L.F. "Pyrolysis of propane in tubular flow reactors"; Ind. Eng. Chem. Process. Des. Develop.; v. 8, no. 1, pp 25-31; Jan. 1969.

8. Slotboom, H.W. and Penninger, J.M.L. "The role of the reactor wall in the thermal hydrocracking of polyaromatic compounds"; Ind. Eng. Chem. Process. Des. Develop.; v. 13, no. 3, pp 296-299; Mar. 1974.

TABLE 1

Properties of Bitumen Feedstock

Specific Gravity 15/15°C		1.008
Sulphur	wt %	4.35
Nitrogen	wt %	0.41
Ash	wt %	0.53
Viscosity at 99°C	cst	124.0
Conradson Carbon Residue	wt %	12.5
Pentane Insolubles	wt %	15.4
Benzene Insolubles	wt %	0.44
Nickel	ppm (wt)	72
Vanadium	ppm (wt)	174

Distillation Analysis

Temp °C	wt %	Sp Gr	Sulphur wt %	Nitrogen wt %
IBP-200	1.8	0.816	1.89	0.010
200-250	2.1	0.861	1.30	0.010
250-333	8.8	0.907	1.70	0.017
333-418	17.0	0.951	2.98	0.084
418-524	15.3	0.989	3.97	0.21
+524	55.0	1.059	5.31	0.79

TABLE 2

Properties of White Wood Coal*

		As Received	Dry
Calorific Value	kJ/kg	23450	25900
Proximate Analysis			
Fixed Carbon	wt %	35.4	39.2
Volatile Matter	wt %	46.4	51.3
Ash	wt %	8.6	9.5
Moisture	wt %	9.6	0.0
Ultimate Analysis			
Carbon	wt %	60.7	67.1
Hydrogen	wt %	3.6	4.0
Sulphur	wt %	0.2	0.2
Nitrogen	wt %	0.8	0.9
Ash	wt %	8.6	9.5
Oxygen	wt %	16.5	18.3

* Coal from White Wood Coal Mine, Calgary Power Ltd., Alberta, Seam No. 3, SW 1/4, Section 14, Range 4, Township 53, West 5th Meridian

TABLE 3

Reaction Conditions for Thermal Hydrocracking Experiments*

Run No.	Feed Rate kg/h	Coal wt % of feed	Reactor Temp °C	Hot Separator Temp °C	Gas Flow m^3API/t	Duration of run h	Cause of Shutdown
NA 450	13.0	0	450	335 to 355	428	309	Hydrogen recycle gas pump failure; normal shutdown
CA 450-1	-	2	450	350	-	17	Operator's error; no H_2 flow; system coked up
CA 450-2	13.3	2	450	345 to 355	419	113	Feed pump failure due to lumps
CA 450-3	12.9	2	450	345 to 355	432	16	Problems with liquid fuel control in hot separator
CA 450-4	13.4	2	450	345 to 370	416	504	Completion of run
CA 450-5	13.3	2	450	340 to 375	419	74	
CA 455	13.4	2	455	365 to 375	418	443	Completion of run

* Pressure = 10.44 MPa, LHSV = 3.0, and gas flow at pressure = 55.7 ℓ/h

TABLE 4

Properties of Reactor Fluid

Run No.	Sample Point of Reactor	Day	PI* wt %	BI* wt %	Asphaltene wt %	Ash wt %	BIOR* wt %	CCR* wt %	S wt %	N wt %	V wt %	Ni wt %
NA 450	Bottom	5	26.9	7.1	19.8	4.7	2.4	26.0			0.19	0.11
		10	43.3	23.2	20.1	11.8	11.4	38.6			0.82	0.24
	Middle	5	22.8	4.9	17.9	2.1	2.8	21.9			0.68	0.03
		10	22.5	3.1	19.4	2.2	0.9	20.6			0.09	0.04
	Top	5	21.6	4.2	17.3	1.2	3.0	18.1			0.04	0.01
		10	-	10.6	-	1.7	8.9	22.4			0.04	0.02
CA 450-4	Bottom	7	56.4	45.4	11.0	35.7	9.7	50.9			-	-
		11	56.6	45.5	11.1	36.0	9.5	50.0			-	-
		16	59.0	50.1	8.9	38.5	11.6	55.8			-	-
		21	64.8	55.5	9.3	38.4	17.1	58.5			-	-
	Middle	21	63.2	57.8	5.4	40.6	17.2	62.8	-	-	-	-
CA 455	Bottom	3	-	-	-	19.7	-	-	-	-	-	-
		4	58.8	43.5	15.3	21.9	21.6	52.7	-	-	-	-
		21	-	-	-	-	-	50.6	6.1	0.53	-	-
		21	-	-	-	40.1	-	55.9	6.1	0.51	-	-
	Middle	21	-	-	-	31.9	-	55.2	4.53	0.65	-	-

*PI - Pentane Insolubles, BI - Benzene Insolubles, BIOR - Benzene Insoluble Organic Residue
CCR - Conradson Carbon Residue

TABLE 5

Details of Solid Deposits at the End of Run

Run No.	Location	Comments	Total Amount of Deposits g
NA 450 (16 Days)	Reactor	Hard coke deposits from about 0.3 m from the top to about 1 m from the bottom; the coke had to be burned at high temperatures to clean the reactor	6600
	Hot Receiver	Small deposits on down pipe and walls; heavy deposits on hot receiver bottom cap	
CA 450-4 (21 days)	Reactor	Top reactor cap was clean; top 1/3 of reactor had heavy deposits on walls; inlet funnel had hard black material; the deposits contain both coal and coke	132
	Hot Receiver	Small deposits on walls; down pipe clean	
CA 450-5 (3 days) CA 455 (19 days)	Reactor	Hard coke-like material near the top; cone-shaped shell at the top; thick sticky black substance on the inlet funnel; hard deposits on walls	940
	Hot Receiver	No deposits on walls; small amount of bubbly deposit on down tube	

TABLE 6

Analysis of Solid Deposits

Run No.	Sample Description	PI wt %	BI wt %	Ash wt %	CCR wt %	Sulphur wt %	Nitrogen wt %
CA 450-5 CA 455	1) Solids in the top section of the reactor	93.6	92.6	47.6	90.3	4.77	1.09
	2) Solids in the bottom section of the reactor	-	33.7	11.4	-	-	-
	3) Solids on the bottom cap	-	34.6	10.6	-	-	-
CA 450-4	1) Solids in the reactor	96.4	93.8	56.9	95.0	-	-
	2) Solids on the bottom cap	80.2	72.6	52.7	78.0	-	-

TABLE 7

Preheater and Reactor Temperatures

Location Measured	NA 450		CA 450-4		CA 455	
	Start	End	Start	End	Start	End
Preheater						
Skin Temperature						
1	355	375	370	370	348	350
2	400	401	395	400	400	395
3	450	455	450	450	450	450
Inside Temperature						
1	210	257	263	262	265	263
2	318	301	312	316	320	322
3	364	338	364	362	367	369
Reactor						
Skin Temperature						
1	457	456	455	451	455	469
2	449	446	449	448	439	446
3	440	441	440	429	437	439
4	447	464	442	442	450	444
Inside Temperature						
1	408	397	414	407	416	412
2	449	449	450	450	455	455
3	450	450	450	450	455	455
4	450	450	450	450	455	455
5	451	450	450	450	456	455

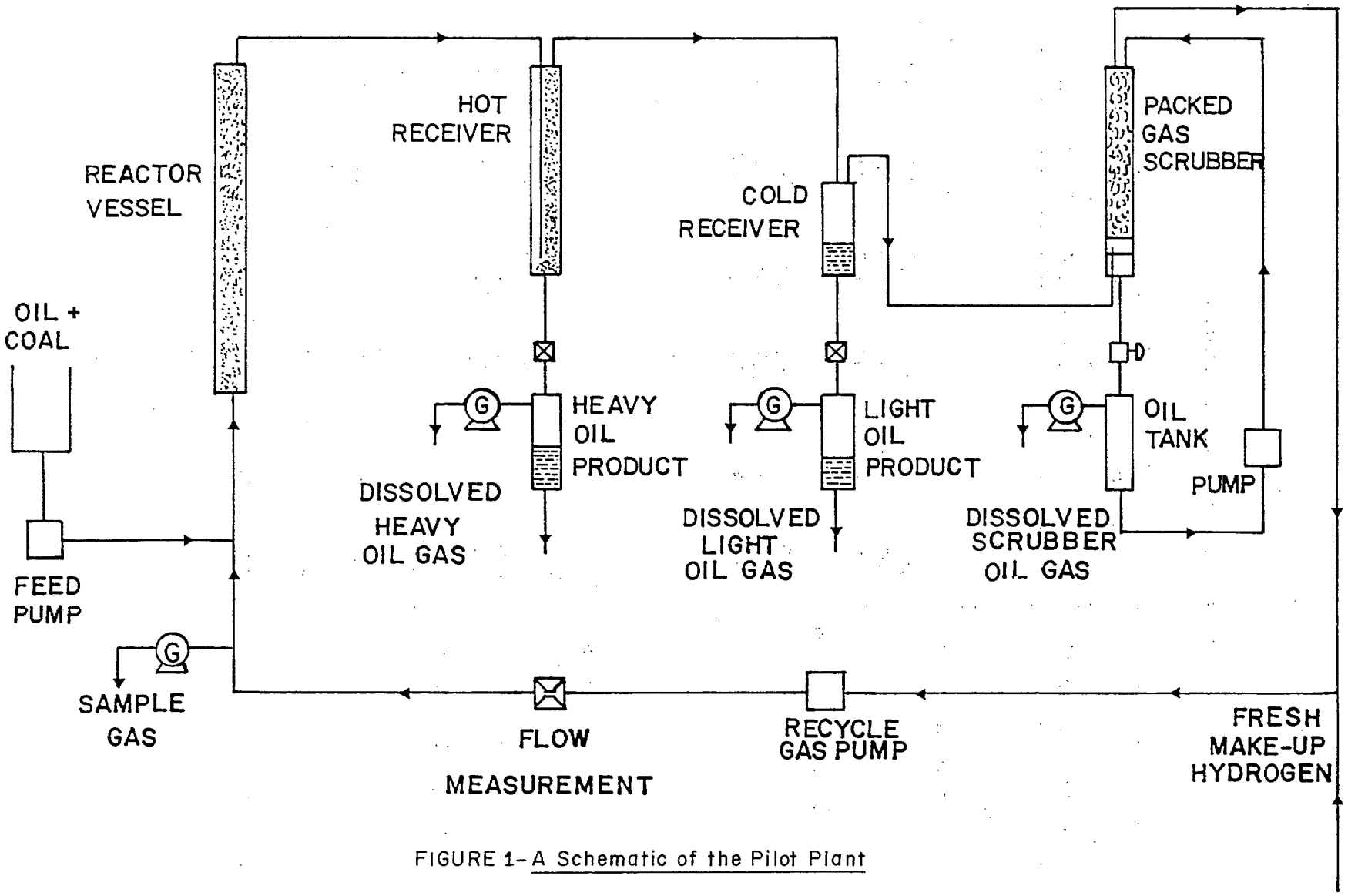

FIGURE 1- A Schematic of the Pilot Plant

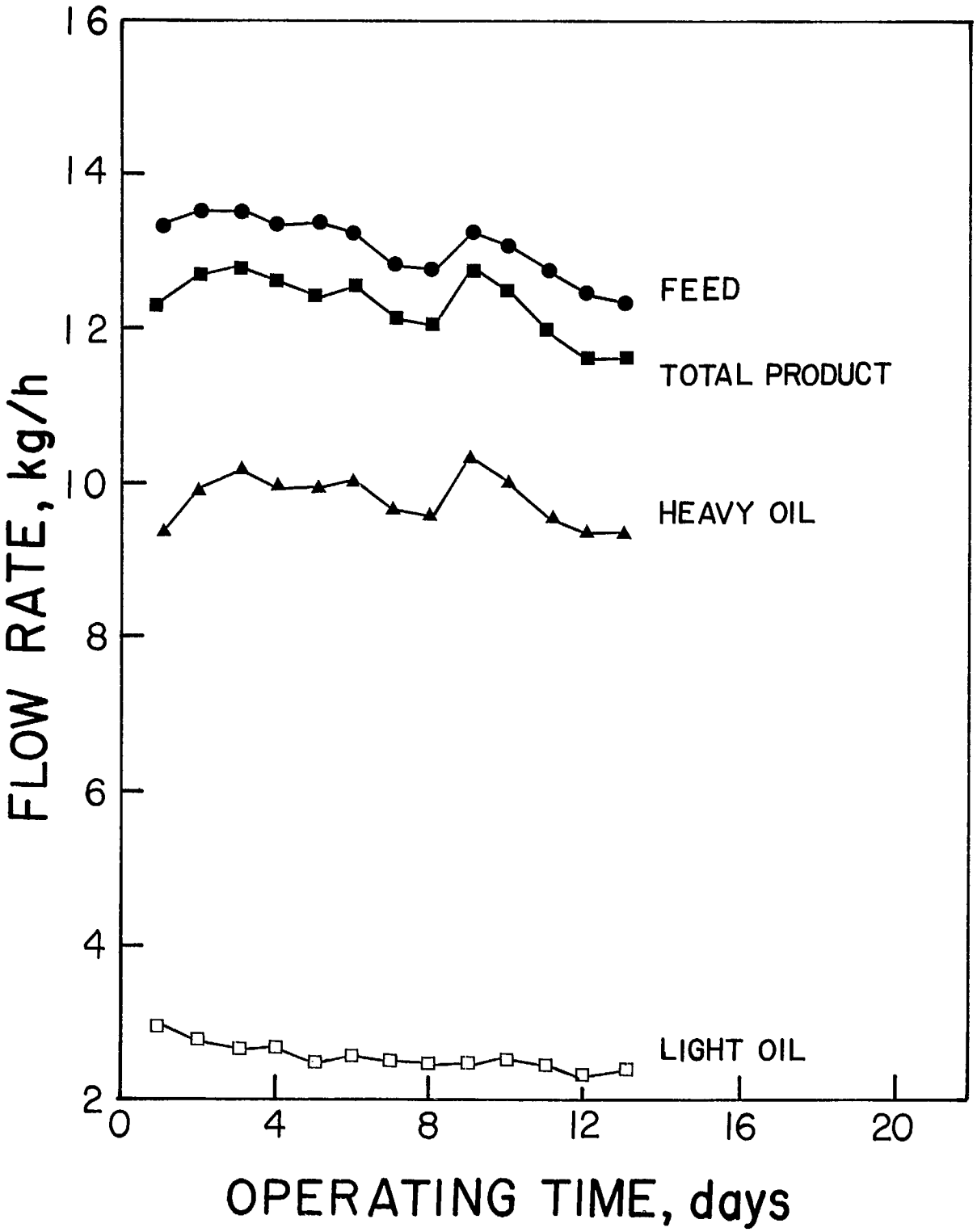

FIGURE 2 - Feed, Product, Heavy-oil and Light-oil Flow Rates for Run NA 450

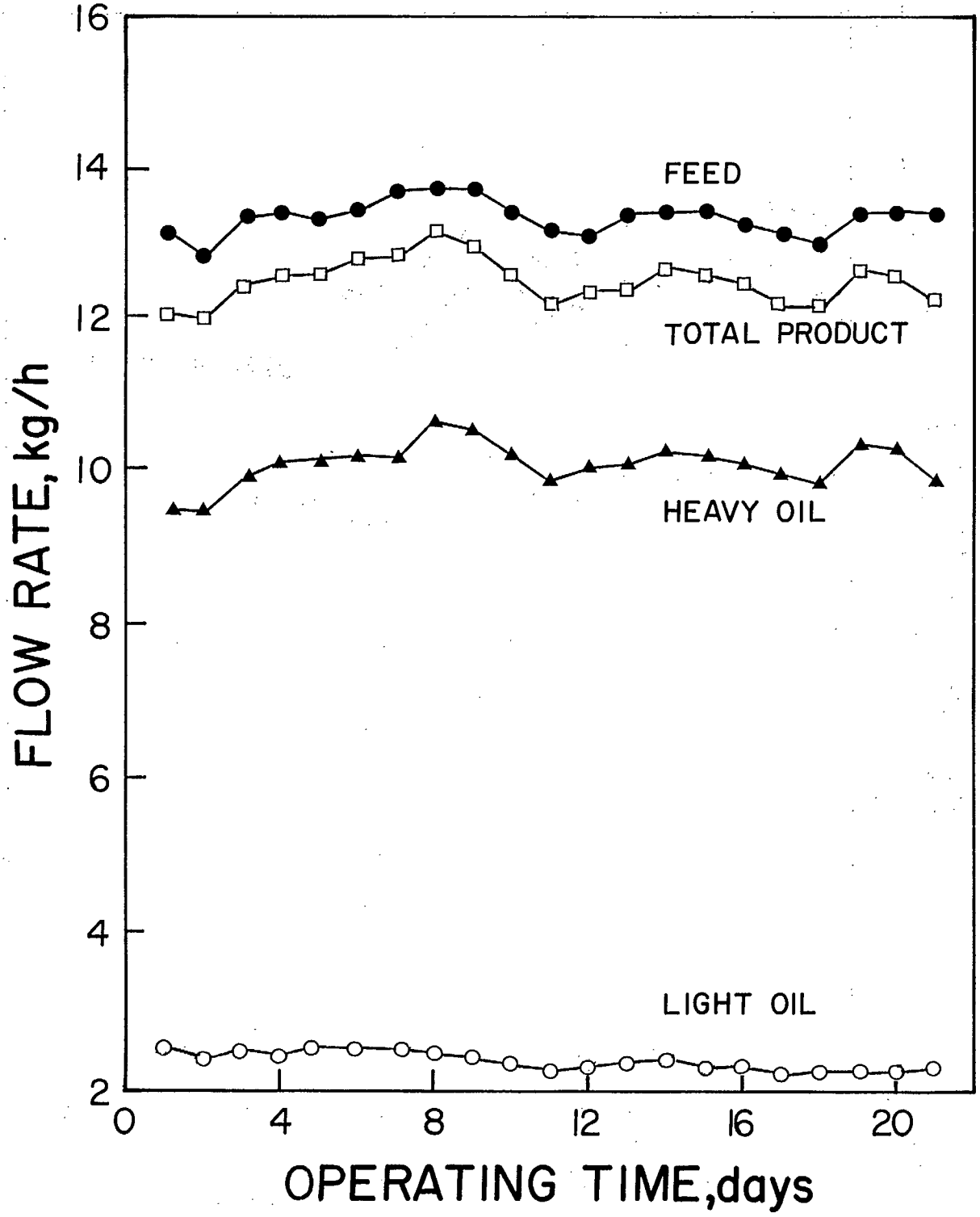

FIGURE 3 - Feed, Product, Heavy-oil and Light-oil Flow Rates for Run CA 450-4

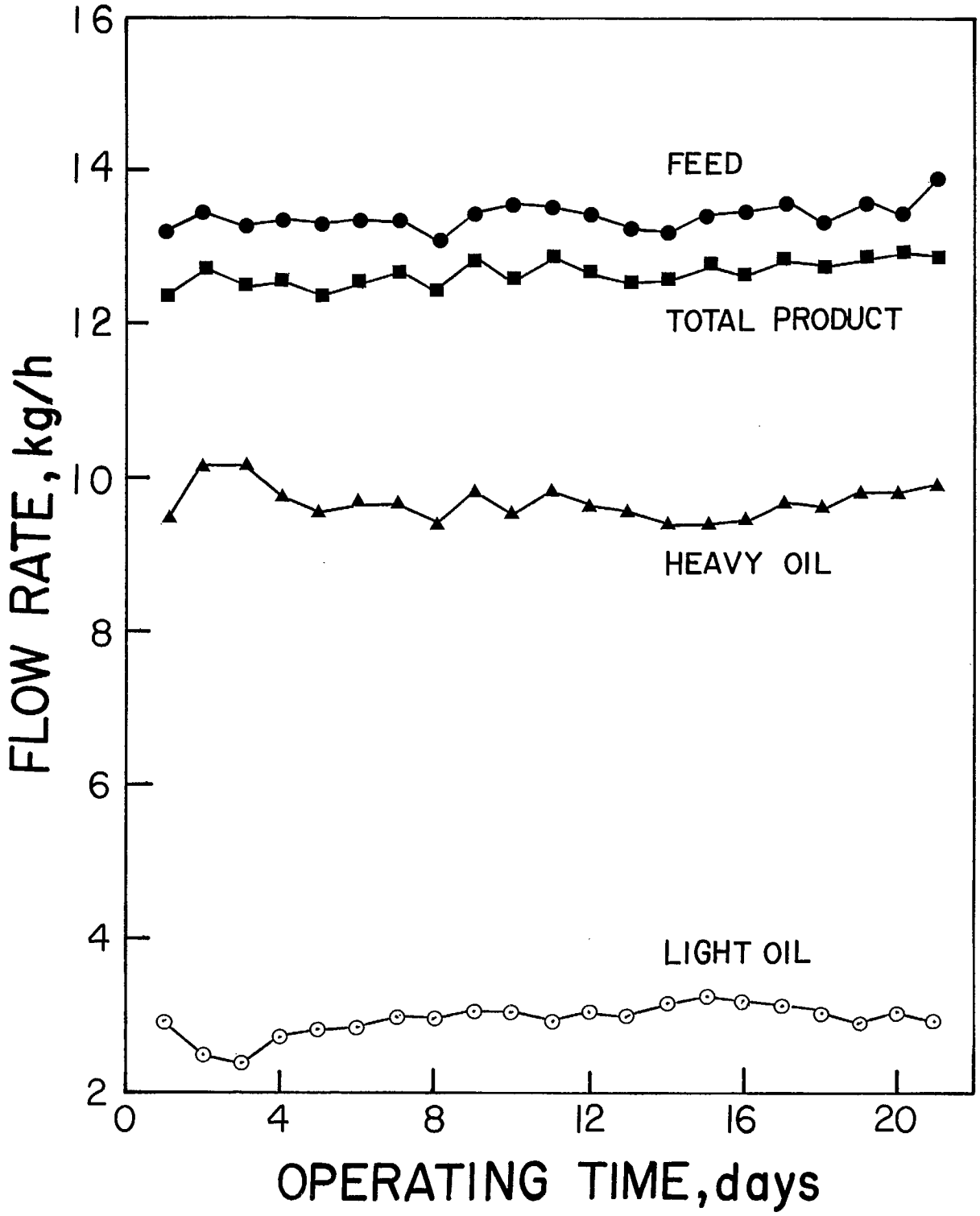

FIGURE 4 - Feed, Product, Heavy-oil and Light-oil Flow Rates for Runs CA 450-5 and CA 455

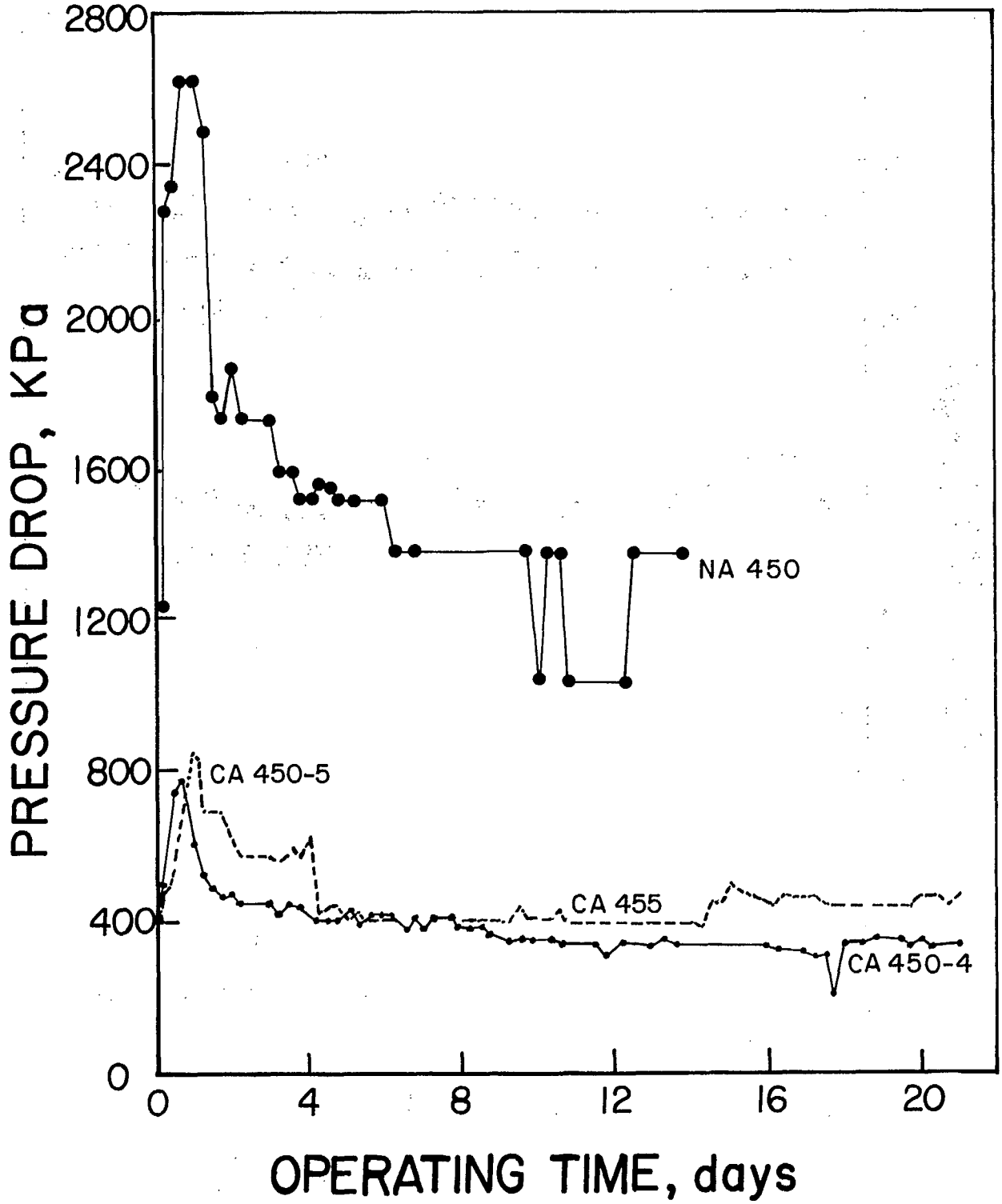

FIGURE 5 - Total Pressure Drop in the Thermal Hydrocracking Pilot Plant

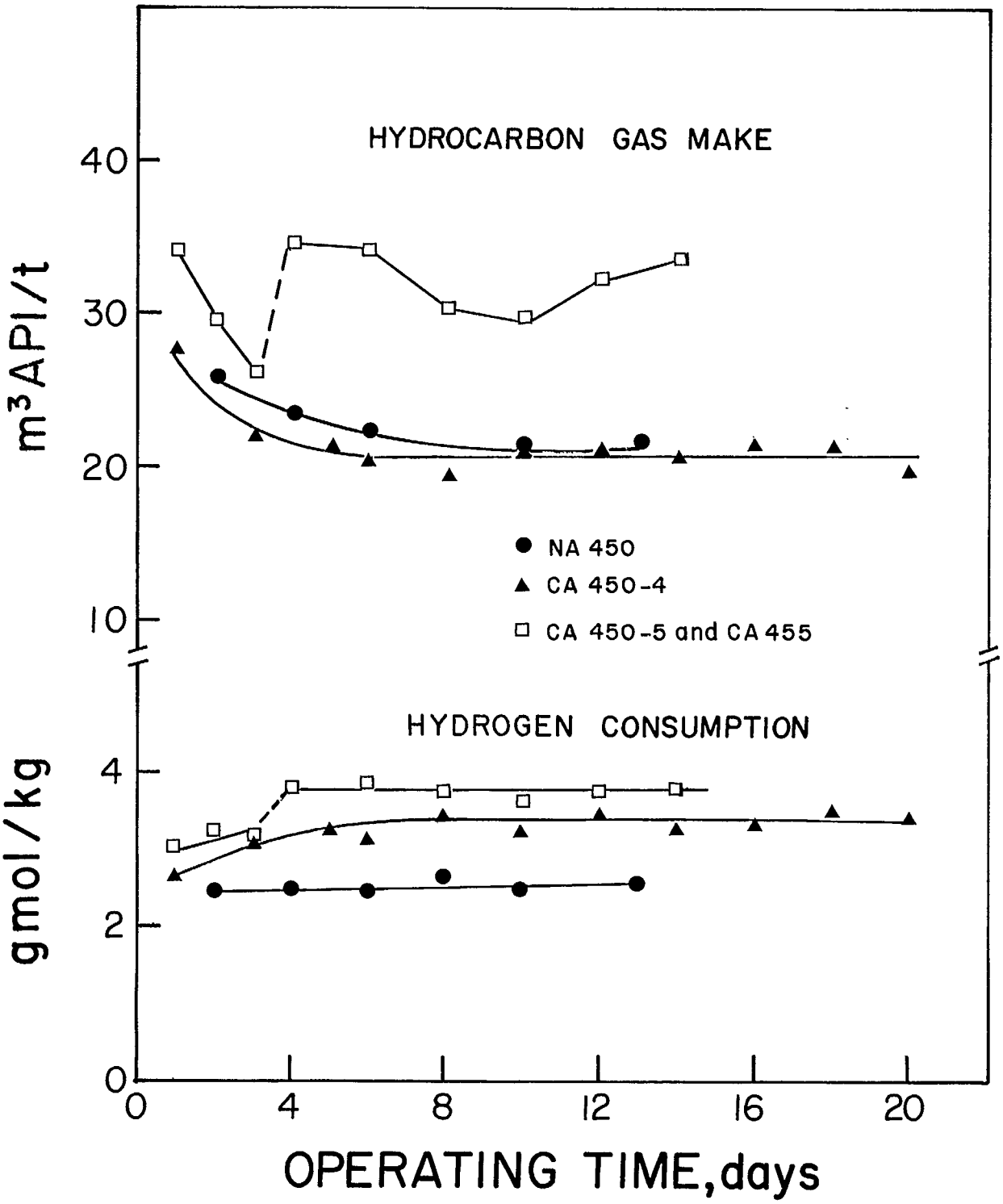

FIGURE 6 - Hydrogen Consumption and Hydrocarbon Gas Make During Thermal Hydrocracking

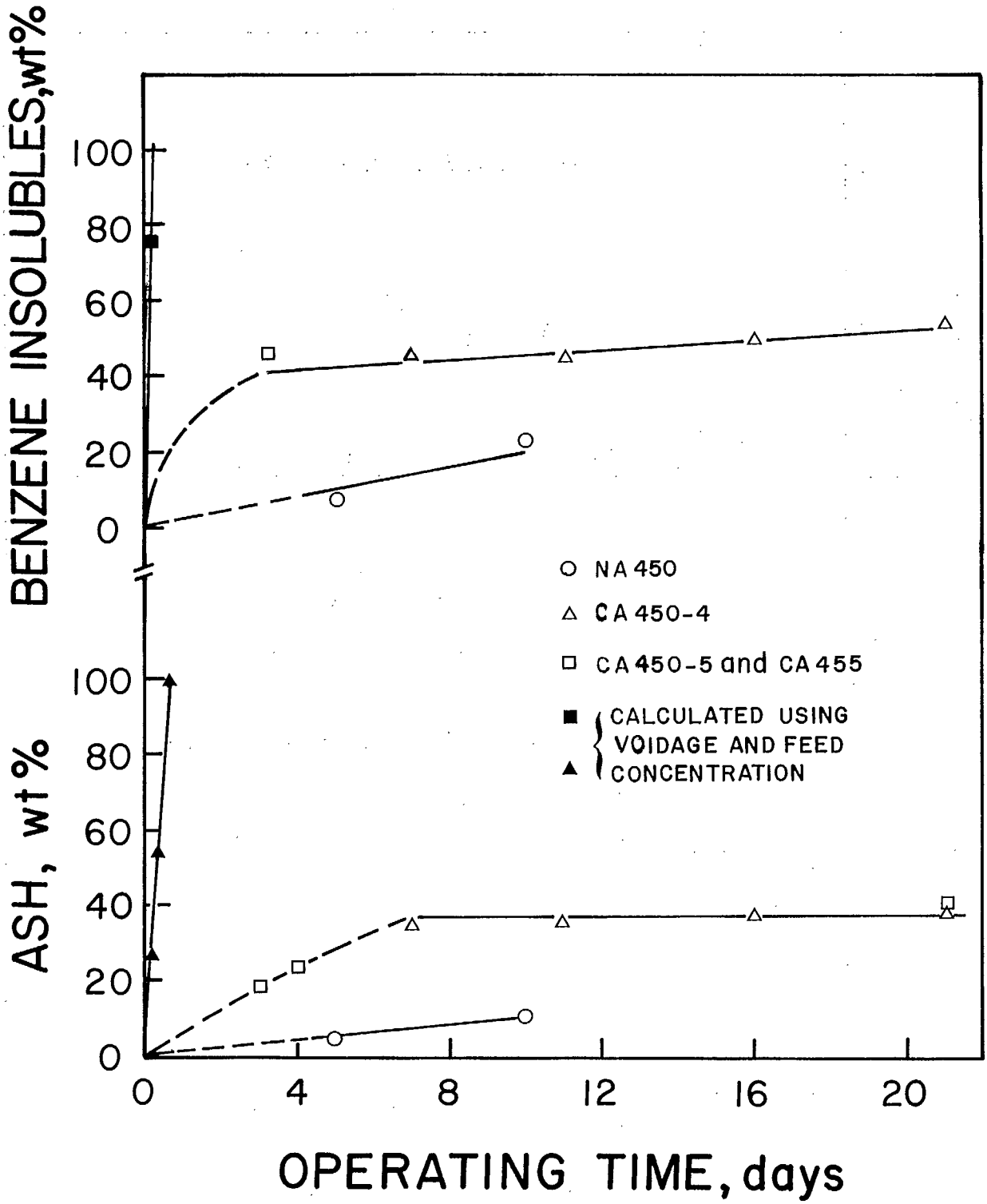

FIGURE 7 - Accumulation of Benzene Insolubles and Ash in the Bottom of the Reactor

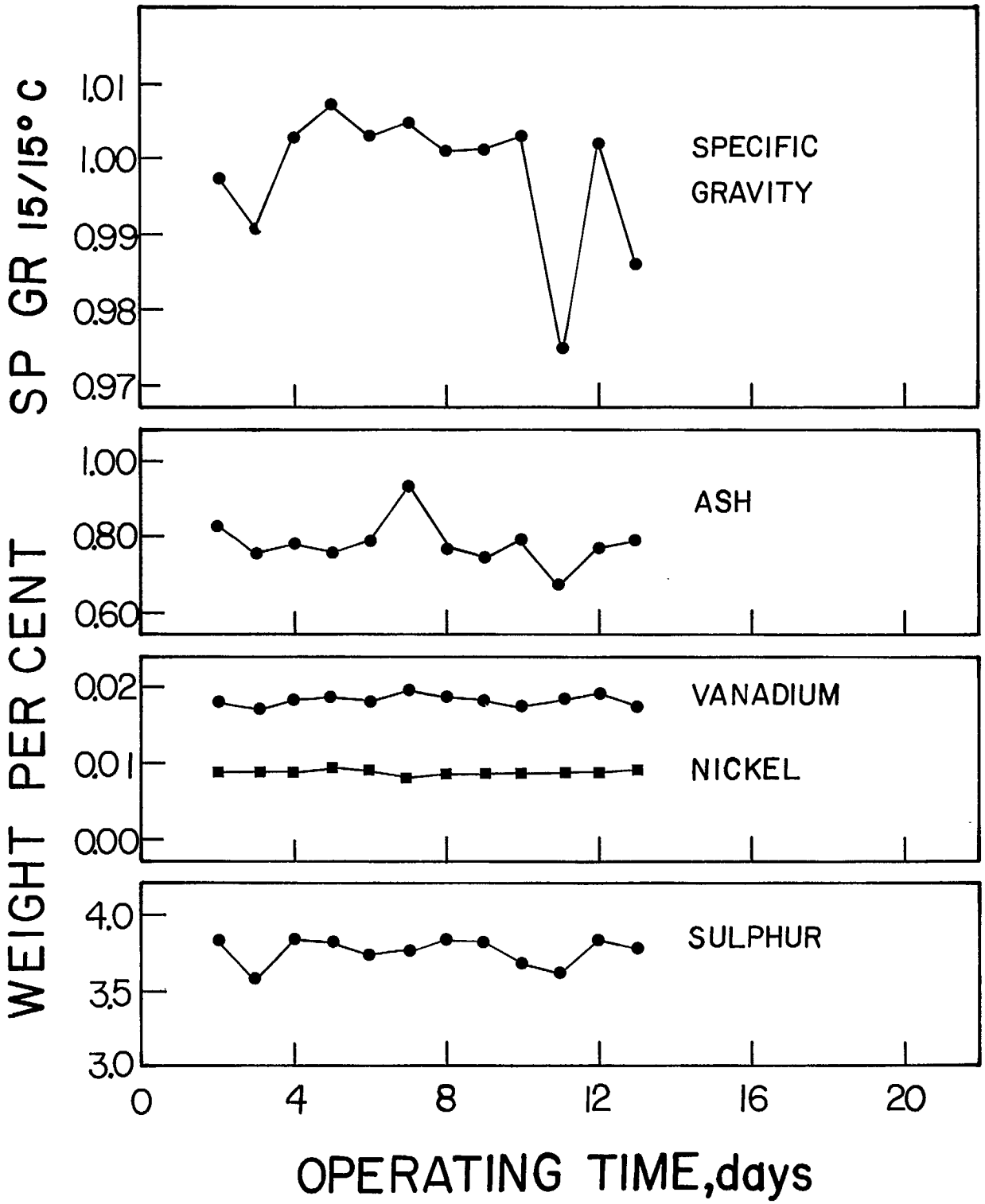

FIGURE 8 - Heavy-oil Properties During Run NA 450

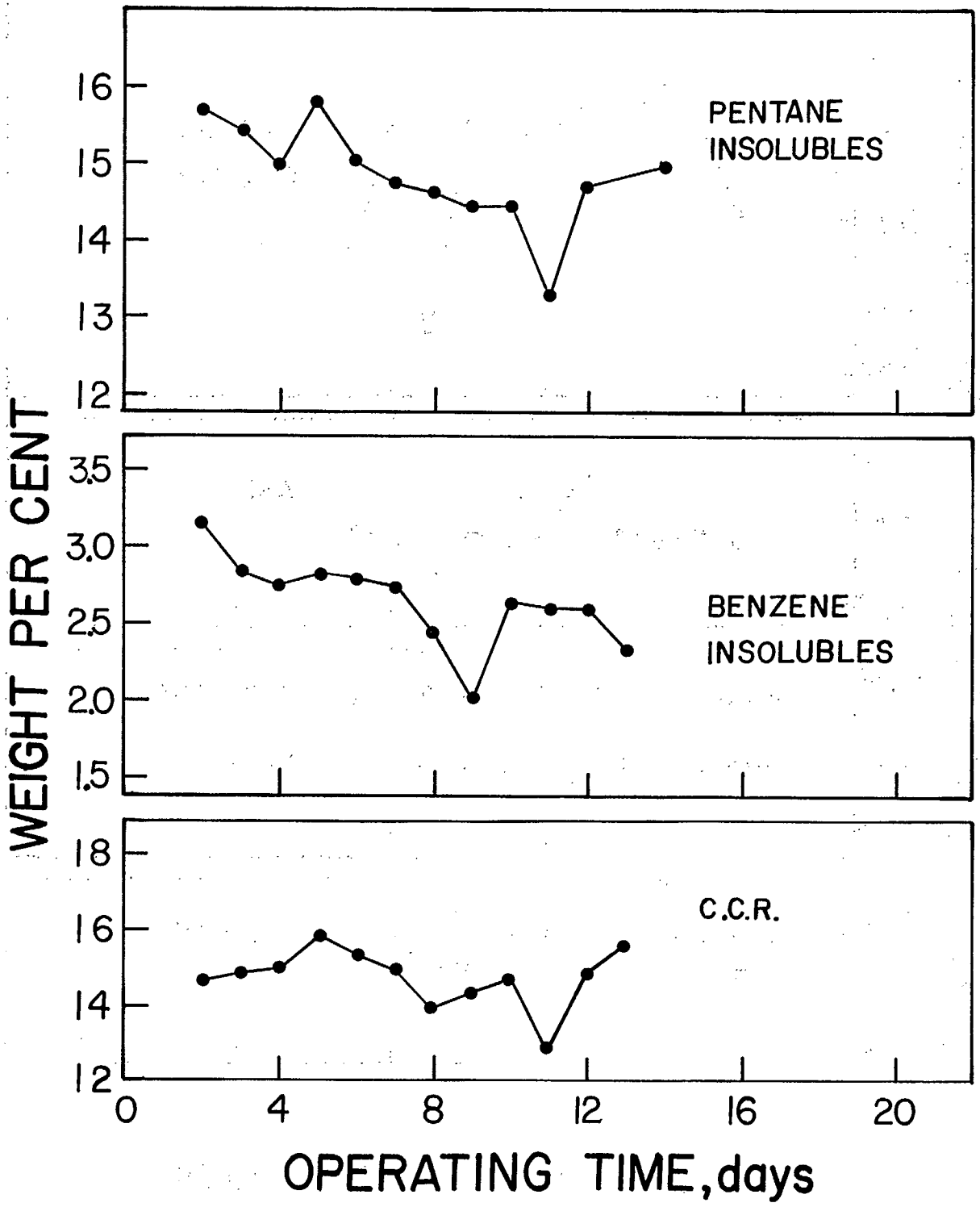

FIGURE 9 - Heavy-oil Properties During Run NA 450

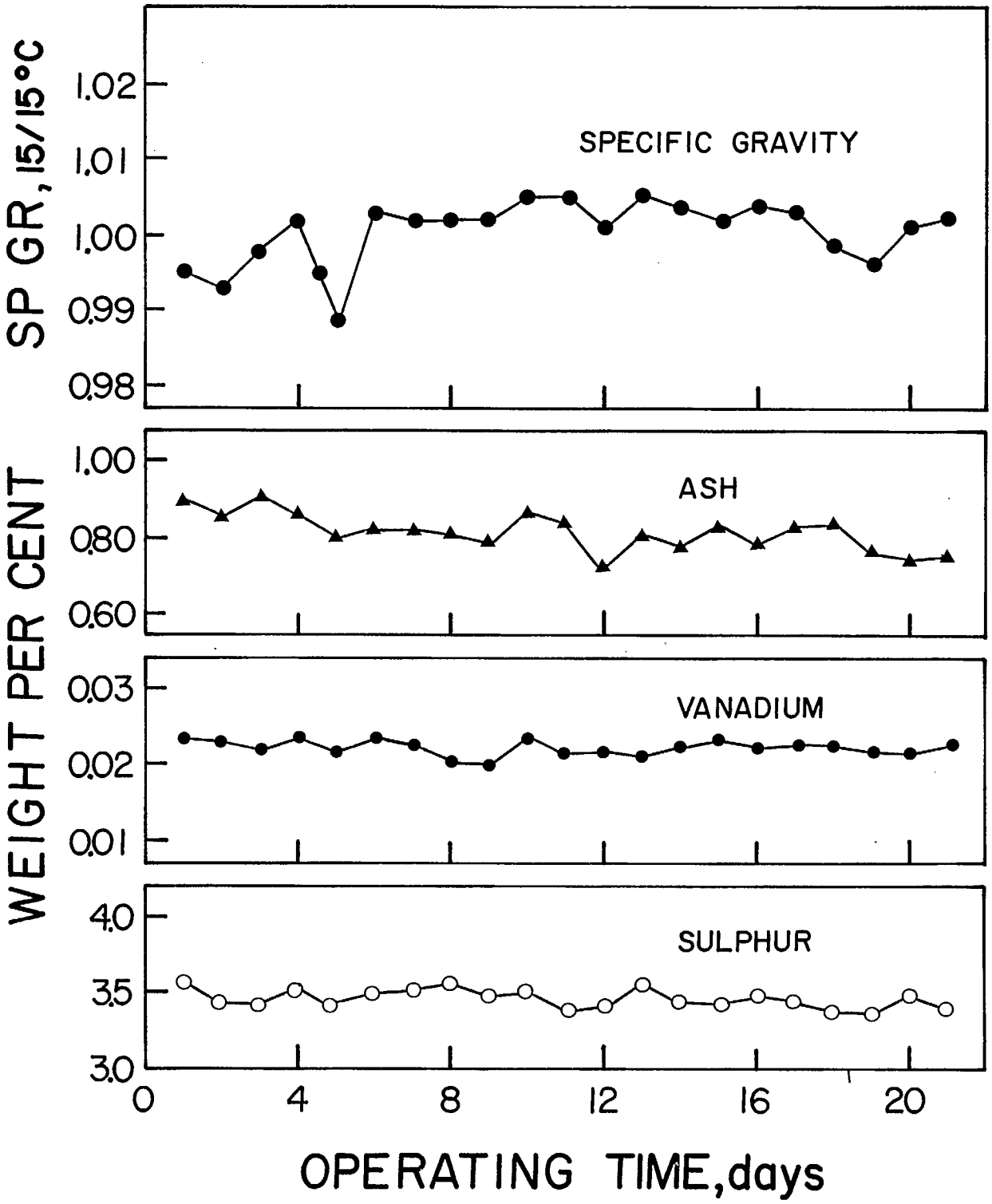

FIGURE 10 - Heavy-oil Properties During Run CA 450-4

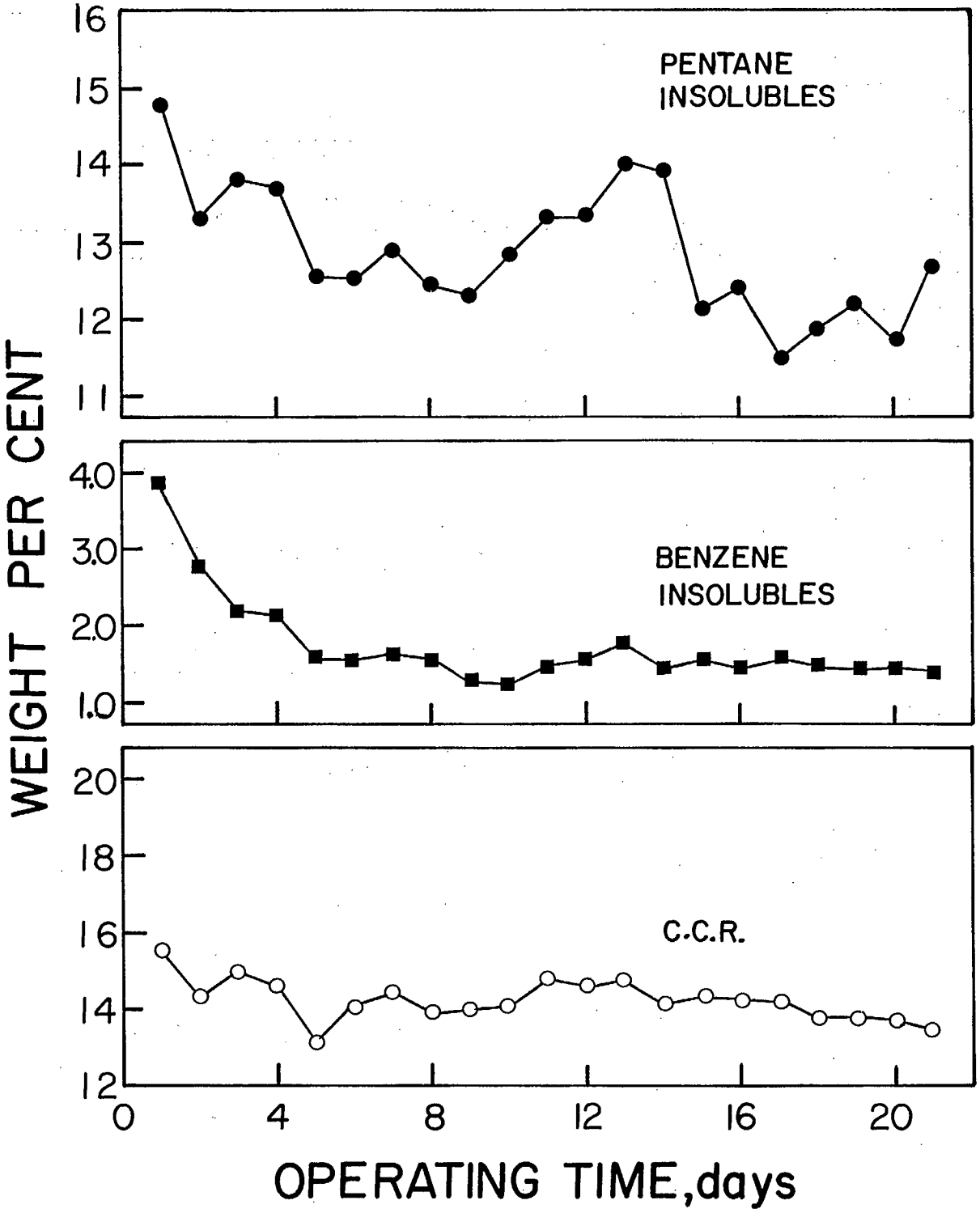

FIGURE 11 - Heavy-oil Properties During Run CA 450-4

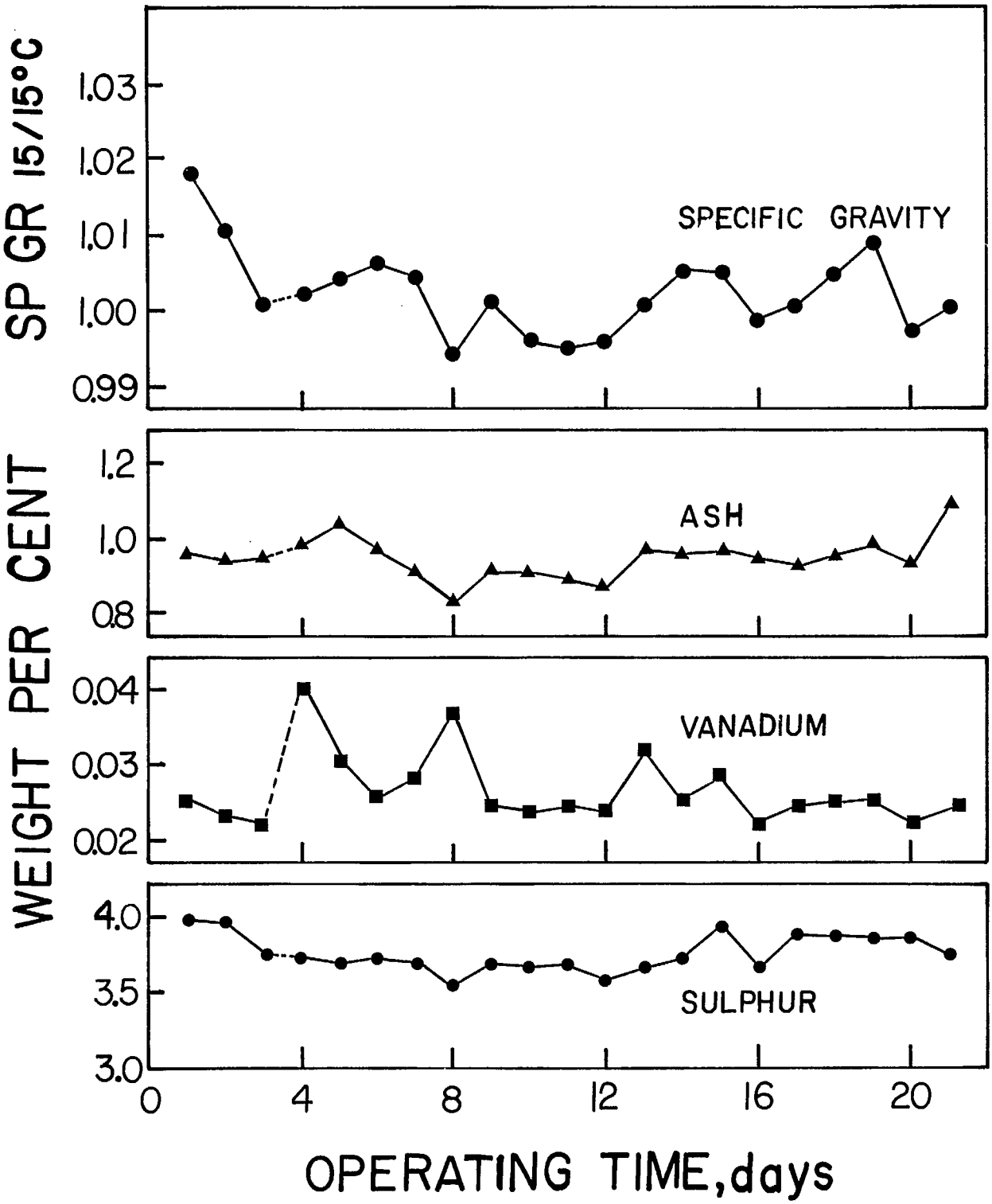

FIGURE 12 - Heavy-oil Properties During Runs CA 450-5 and CA 455

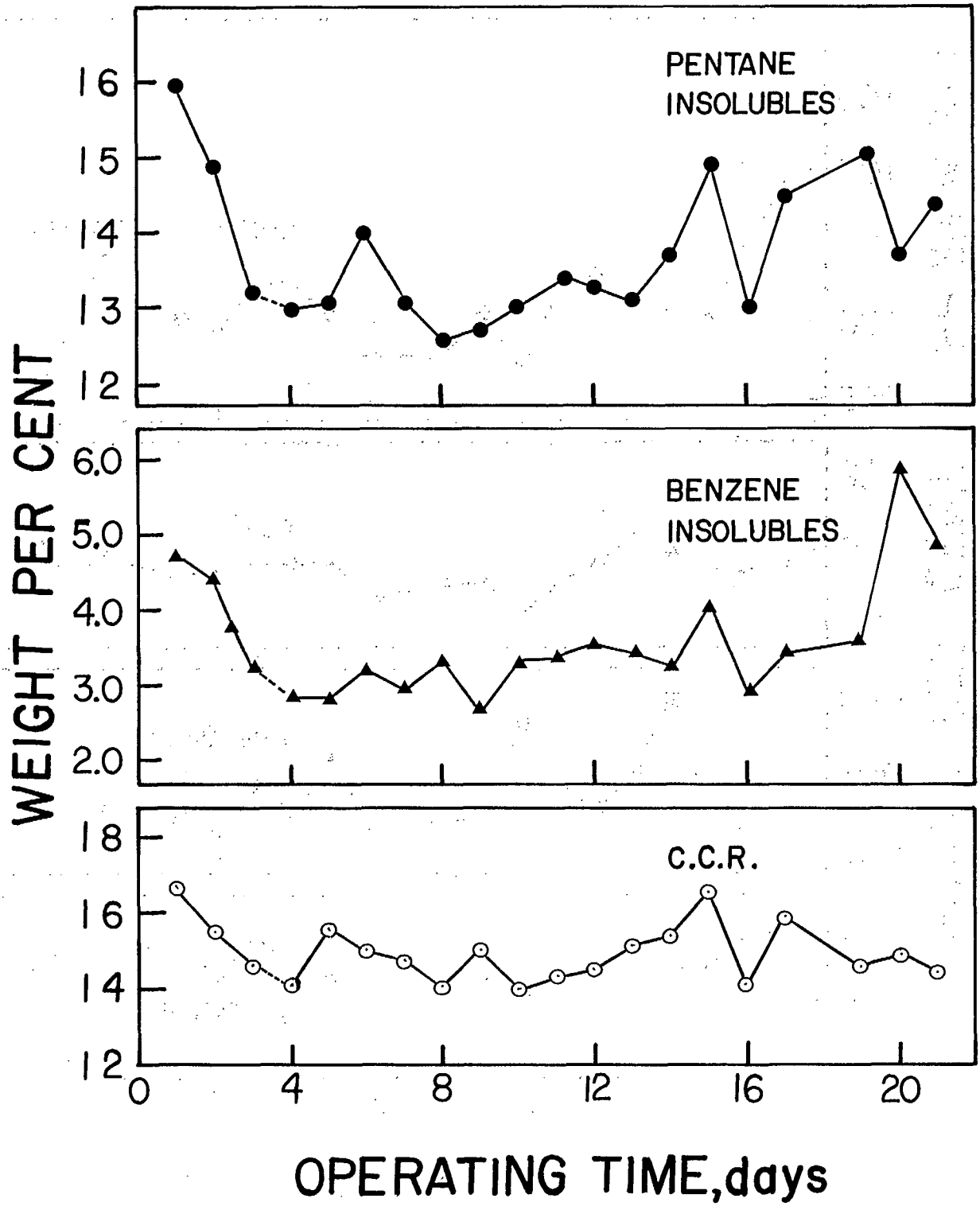

FIGURE 13 - Heavy-oil Properties During Runs CA 450-5 and CA 455

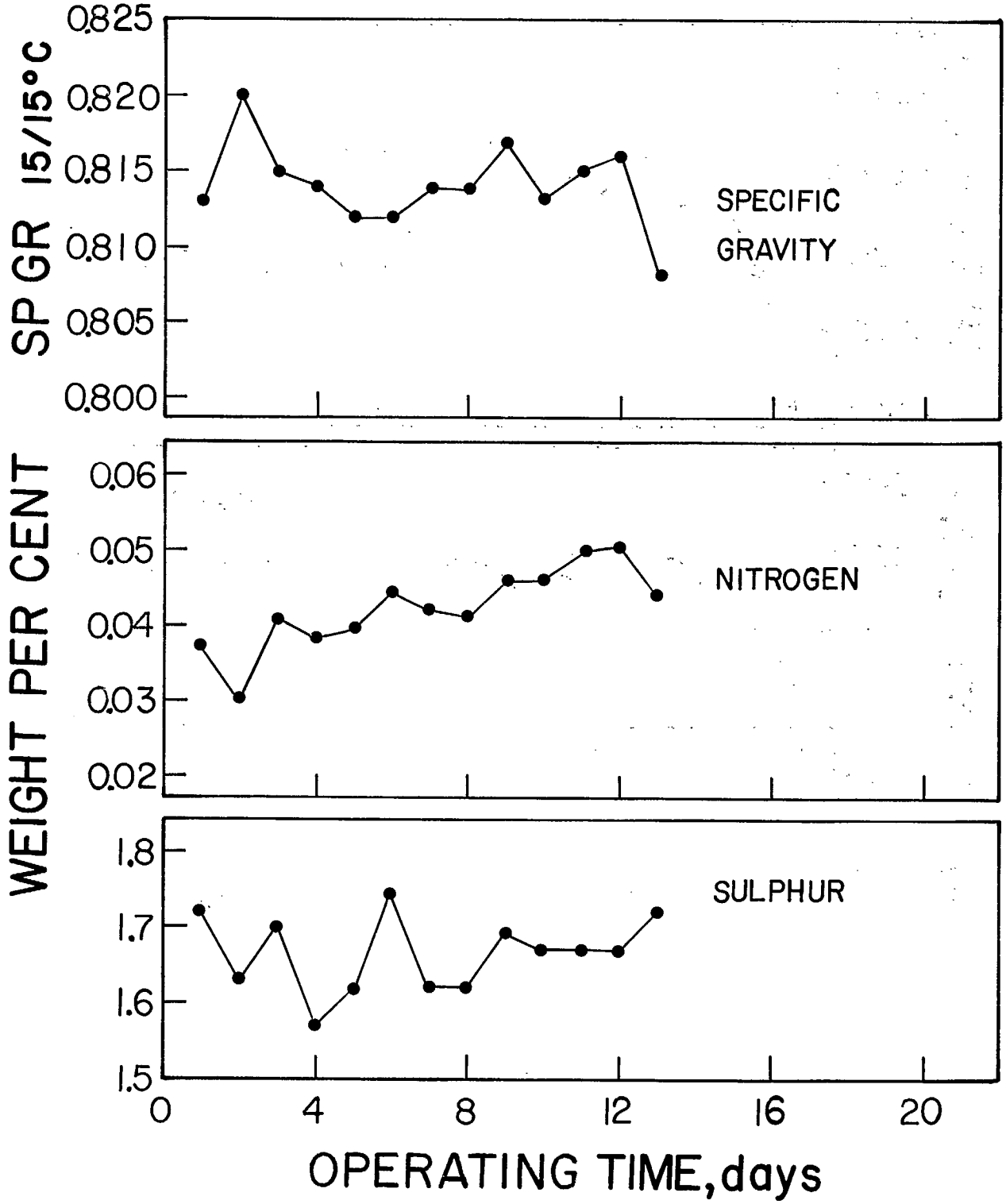

FIGURE 14 - Light-oil Properties During Run NA 450

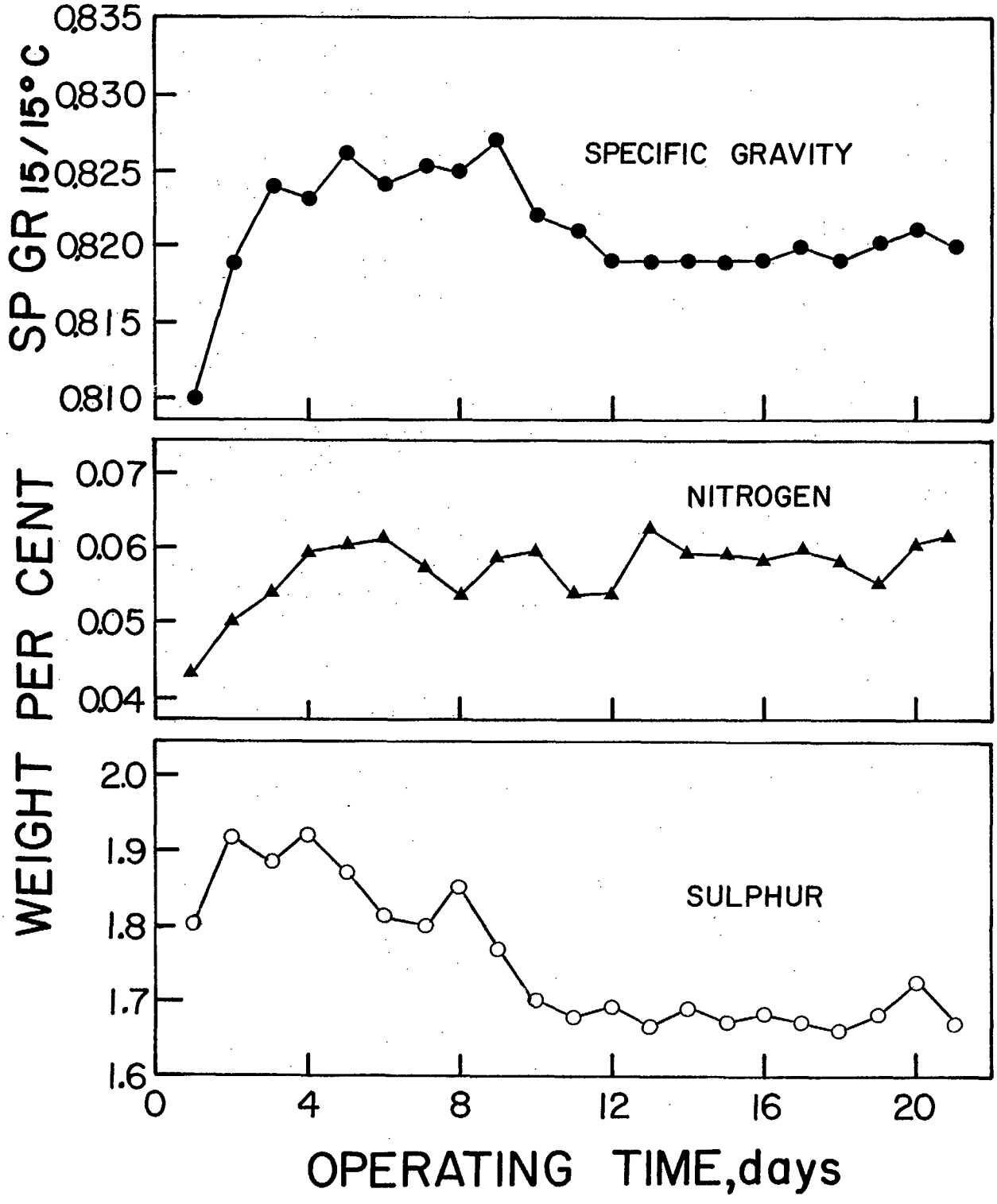

FIGURE 15 - Light-oil Properties During Run CA 450-4

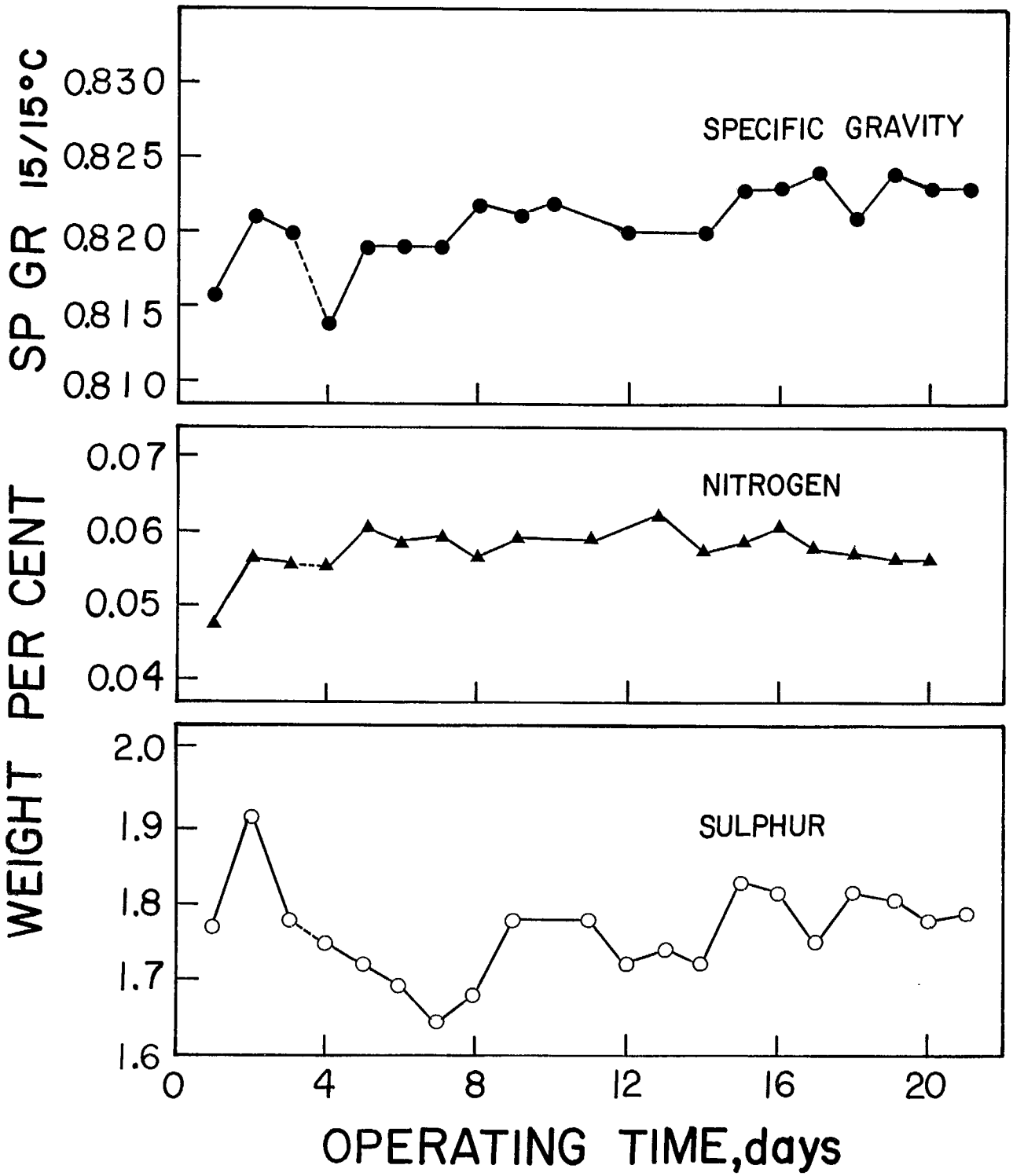

FIGURE 16 - Light-oil Properties During Runs CA 450-5 and CA 455

CANMET REPORTS

Recent CANMET reports presently available or soon to be released through Printing and Publishing Supply and Services, Canada (addresses on inside front cover), or from CANMET Publications Office, 555 Booth Street, Ottawa, Ontario K1A 0G1:

Les récents rapports de CANMET, qui sont présentement disponibles ou qui ce seront bientôt peuvent être obtenus de la direction de l'Imprimerie et de l'Edition, Approvisionnements et Services, Canada (adresses au verso de la page couverture), ou du Bureau de Vente et distribution de CANMET, 555 rue Booth, Ottawa, Ontario, K1A 0G1:

77-14 Pit slope manual - Chapter 7 - Perimeter blasting; P. Calder;
Cat. No. M3B-14/7-1977, ISBN 0-660-01011-9; Price: $2.25 Canada, $2.70 other countries.

77-16 Pit slope manual - Supplement 5-1 - Plane shear analysis; D.F. Coates;
Cat. No. M3B-14/5-1977-1, ISBN 0-660-01008-9; Price: $4.25 Canada, $5.10 other countries.

77-19 Pit slope manual - Supplement 2-2 - Domain analysis programs; D. Cruden and J. Ramsden;
Cat. No. M3B-14/2-1977-2, ISBN 0-660-00990-0; Price: $3.75 Canada, $4.50 other countries.

77-23 Pit slope manual - Supplement 2-4 - Joint mapping by terrestrial photogrammetry; G. Herget;
Cat. No. M3B-14/2-1977-4, ISBN 0-660-00992-7; Price: $2.50 Canada, $3.00 other countries.

77-24 Pit slope manual - Supplement 2-5 - Structural geology case history; G. Herget;
Cat. No. M3B-14/2-1977-5, ISBN 0-660-00993-5; Price: $2.75 Canada, $3.30 other countries.

77-30 Pit slope manual - Supplement 4-1 - Computer manual for seepage analysis; J. Marlon-Lambert;
Cat. No. M3B-14/4-1977-1, ISBN 0-660-01007-0; Price: $3.50 Canada, $4.20 other countries.

77-31 Pit slope manual - Supplement 10-1 - Reclamation by vegetation - Vol. 1 - Mine waste description and case histories; D. Murray;
Cat. No. M3B-14/10-1977-1, ISBN 0-660-01013-5; Price: $3.50 Canada, $4.20 other countries.

77-47 Statistical correlation of ASTM and JIS coke tumbler test results; W.R. Leeder and K.A. Jonasson;
Cat. No. M3B-13/77-47, ISBN 0-660-01705-9; Price: $1.25 Canada, $1.50 other countries.

77-56 Corrosion protection and lateral displacement characteristics of rock anchors; R. Sage;
Cat. No. M3B-13/77-56, ISBN 0-660-01806-3; Price: $1.25 Canada, $1.50 other countries.

77-58 Pit slope manual - Supplement 10-1 - Reclamation by vegetation - Vol. 2 - Mine waste inventory by satellite imagery; D. Murray;
Cat. No. M3B-14/10-1977-1-2, ISBN 0-660-01464-5; Price: $6.00 Canada, $7.20 other countries.

77-60 The high-temperature behaviour of blast furnace coke - A review; D.A. Reeve, J.T. Price and J.F. Gransden;
Cat. No. M3B-13/77-60, ISBN 0-660-01807-1; Price: $1.25 Canada, $1.50 other countries.

77-61 A rapid method for the determination of sulphur and vanadium in petroleum products by non-dispersive x-ray fluorescence; R. Makhija, R.G. Draper and E. Furmisky;
Cat. No. M3B-13/77-61, ISBN 0-660-01848-9; Price: $1.00 Canada, $1.20 other countries.

77-65 Performance of superplasticizers in concrete: Laboratory investigation - Part 1; V.M. Malhotra and D. Malanka;
Cat. No. M3B-13/77-65, ISBN 0-660-01739-3; Price: $1.75 Canada, $2.10 other countries.

78-2 Revision of recommended values for reference ores MP-1 and KC-1; G.H. Faye and W.S. Bowman;
Cat. No. M3B-13/78-2, ISBN 0-660-01712-1; Price: $1.00 Canada, $1.20 other countries.

78-3 Certified and provisional reference materials available from the Canada Centre for Mineral and Energy Technology, 1978; G. Faye;
Cat. No. M3B-13/78-3, ISBN 0-660-01804-7; Price: $1.25 Canada, $1.50 other countries.

76-40F Evaluation des charbons marchands canadiens: Nouvelle-Ecosse et Nouveau Brunswick - 1975; T.E. Tibbetts et W.J. Montgomery;
Cat. No. M3B-13/76-40F, ISBN 0-660-01821-7; Price: $3.00 Canada, $3.60 other countries.