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A PILOT PLANT INVESTIGATION OF THERMAL HYDROCRACKING OF ATHABASCA BITUMEN: 2. EFFECT OF RECYCLE OF HEAVY OIL ON PRODUCT QUALITY

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

AUGUST 1976

ENERGY RESEARCH PROGRAM
ENERGY RESEARCH LABORATORIES
CANMET REPORT 77-20

Available by mail from:

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

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

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Catalogue No. M38–13/77-20
ISBN 0-660-01072-0

Price: Canada: $1.50
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

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Ottawa, Canada K1A 0G1

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Nº de catalogue M38–13/77-20
ISBN 0-660-01072-0

Prix: Canada: $1.50
Autres pays: $1.80

Prix sujet à changement sans avis préalable.

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by

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

ABSTRACT

Studies were made of thermal hydrocracking in a bottom-feed reactor with recirculation of a portion of the heavy-oil product (recycle run). In this report, a comparison of product quality for runs with and without recycle is made at similar experimental conditions of temperature, pressure, hydrogen gas flow and fresh feed flow.

Compared with once-through runs, the pitch conversion decreased slightly for the recycle run but the hydrogen consumption increased. The sulphur content of the distillate was nearly the same for both cases, but nitrogen content and specific gravity were lower for the recycle run because of a larger fraction boiling between the initial boiling point and 250°C and higher nitrogen removal. The sulphur content of the pitch was nearly the same, but the pitch specific gravity was higher for the recycle run. Sulphur removal was nearly equal but nitrogen removal was about 17 wt % for the recycle run compared with 4% for once-through operation.

Reactor temperature had a marked effect on product yields, pitch conversion and sulphur removal, and a minor effect on product quality.

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UNE ETUDE DE L'HYDROCRAQUAGE THERMIQUE DE BITUME DE L'ATHABASCA SUR UNE INSTALLATION PILOTE
2. L'EFFET DU RECYCLAGE D'HUILE LOURDE SUR LA QUALITE DU PRODUIT

par

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

RESUME

Des études ont été consacrées au recyclage d'une portion du produit d'huile lourde lors du procédé d'hydrocraquage thermique dans un réacteur alimenté par le fond. Ce rapport présente les résultats obtenus de la comparaison qui a été faite entre la qualité du produit provenant d'opérations avec recyclage et celles sans recyclage qui ont eu lieu dans des conditions analogues de température, de pression, d'écoulement d'hydrogène et d'écoulement d'une nouvelle alimentation.

Après avoir comparé ces essais avec recyclage et ceux d'une seule opération, on remarque que la conversion du brai a légèrement diminuée et la consommation d'hydrogène a augmentée. Le contenu en soufre du distillé était presque de la même proportion dans les deux cas; par contre, le contenu en nitrogène et le poids spécifique étaient inférieurs dans le cas de l'opération avec recyclage, et ce, à cause d'une plus grande partie de l'huile lourde qui bouillait entre le point d'ébullition initial et 250°C et à cause d'une plus forte récupération de nitrogène. Le contenu en soufre du brai, lui, était approximativement le même, tandis que le poids spécifique du brai était plus élevé dans les essais avec recyclage. La récupération du soufre s'est faite presqu'au même taux, mais celle du nitrogène a été d'environ 17%, en poids, lors de l'opération avec recyclage et de 4% pour les essais à une seule opération.

La production, la conversion du brai et la récupération du soufre ont fortement été influencés par la température du réacteur, tandis que la qualité du produit ne l'a été que faiblement.

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CONTENTS

TABLES

FIGURES

FIGURES (Cont'd)

INTRODUCTION

Work has been done in accordance with the policy of Energy, Mines and Resources, Canada, of ensuring the effective use of Canada's mineral and energy resources.

One of the objectives of the Energy Research Program of CANMET (Canada Centre for Mineral and Energy Technology) is to develop economical processes to upgrade Canada's low-grade petroleum resources. A thermal-hydrocracking process has been developed at the Energy Research Laboratories for the upgrading of Athabasca bitumen using a once-through reactor (1,2,3). One of the difficulties with the process is reactor fouling when operating at low pressures (4). The process has been found to run successfully for long periods at 24.2 MPa (3500 psi); however, the economics of the process can be considerably improved by operating at pressures of 2000 psi or less. It has been theorized that an increase in liquid velocity in the reactor would aid in keeping mineral matter in suspension and in keeping the walls scoured. This would help in removing wall deposits and in continuously replacing liquid near the wall which could form coke. Accordingly this work was done at 13.9 MPa (2000 psi) using heavy-oil recycle which effectively increased the liquid velocity without increasing the feed rate. An earlier report was written on operational experience with heavy-oil recycle (5). The present report documents the effects of heavy-oil recycle on product quality compared with once-through runs. This includes light-ends gravity, sulphur and nitrogen content for comparison of pilot plant products, and distillate gravity, sulphur, nitrogen, carbon, hydrogen, viscosity and Hempel distillation for evaluation of the distillate as an end product from a commercial plant. This report also includes heavy-oil quality for comparing pilot plant products, and pitch quality for evaluating pitch as a by-product from a commercial plant.

LITERATURE SURVEY

The tar-sand bitumen contains about 51 weight per cent pitch (fraction boiling above 524^{o}C) and about 49 weight per cent distillable hydrocarbons (fraction boiling below 524^{o}C). A considerable amount of research has been conducted on the recovery of the valuable hydrocarbons from the tar-sand bitumen by catalytic (6-8) or non-catalytic (3,8-12) hydroconversion. All of the processes proposed so far have some drawbacks. The bitumen recovered from the tar sand usually contains 0.05 to 4.0 weight per cent clay and metals, such as chemically bound nickel, vanadium and iron, and deposition of these metals on the catalyst makes it extremely difficult to regenerate expensive catalysts. In the hydrocracking of bitumen it is desirable to reduce coke or sludge formation in the system so that fouling of the equipment, resulting in costly shut-downs, can be avoided. It is also desirable to increase the hydrogen content of the pitch to reduce its viscosity and simplify its handling and storage.

Chervenak and Wolk (9) studied the hydroconversion of different bitumen samples with ash contents of 0.7 to 4.1 weight per cent and observed that, with the bitumens containing high ash, the hydrogen content of the pitch was 6.8 wt % and no coke was deposited in the reactor. They found that with Athabasca bitumen, having 2.7 wt % solids and without adding any catalyst in the start-up, a conversion of 75 wt % was obtained at a reactor temperature of 449^{o}C, LHSV of 1.5-1.7 and a hydrogen partial pressure of 1.5-11 MPa (200-1600 psi).

Wolk and Chervenak (10) studied the hydrocracking of three different samples of Athabasca bitumen using a reactor temperature range of 429-449^{o}C, pressures between 5 and 8 MPa (750 and 1200 psi) and separator temperatures of 260-427^{o}C. From their data it seems that the pressure and the liquid velocity in the reactor had no effect on coke formation and pitch conversion. At a reactor temperature of 429^{o}C the pitch conversion was 58 wt %, whereas at a reactor temperature of 449^{o}C the conversion was about 70 wt %. Initial ash content had no effect on pitch conversion.

Layng (11) studied the hydrogenation of tar-sand bitumen in a long reactor (L/D>4.0). In this work reactor effluent was quenched by introducing a part of the heavy ends or a part of the fresh feed to the top of the reactor between the reaction zone and the separator. At conditions of 370-455^{o}C reactor temperature, 3-14 MPa pressure and 0.5-2.0 LHSV, with hydrogen consumption of 4.0-12 g mol/kg (500-1500 scf/bbl) a minimum pitch conversion of 60 wt % was obtained.

Wolk (12) observed that coke formation in the reactor was reduced substantially when the ash content of the reactor fluid was maintained between 4 and 10 wt %. For maintaining the ash content of the reactor fluid, reactor effluent was passed through a low-pressure separator or a liquid hydrocyclone. A part of the liquid underflow with a higher concentration of mineral matter was recycled to the bottom of the reactor. At a reactor temperature of 446°C, a pressure of 8.72 MPa and a liquid space velocity of 1.5 h^{-1} with no heavy-ends recycle, a pitch conversion of 73.9 wt % was obtained; but at a reactor temperature of 454°C, a pressure of 8.37 MPa, a space velocity of 1.0 h^{-1} and a recycle ratio of 0.9 (ash-enriched underflow from the low-pressure separator) the pitch conversion was 87.2 wt %. At a reactor temperature of 449°C, a pressure of 8.37 MPa, a liquid space velocity of 1.5 h^{-1} and with a recycle ratio of 0.09 (ash-enriched underflow from the liquid hydrocyclone containing 10 wt % ash) the pitch conversion was 75 vol %.

Van Driesen et al. (13) have shown that the gas-oil product from H-oil may be advantageously processed in several ways. Often the simplest and least expensive way is to recycle the gas-oil to extinction in the same H-oil reactor used to hydrocrack the residual components of the feed-stock.

Merrill et al. (3) have reported a pilot scale investigation of thermal hydrocracking of Athabasca bitumen without heavy-oil recycle. They showed that at sufficiently high pressures (10 MPa or higher) pitch conversion of 90 wt % or better and distillate yields up to 86 % by weight could be obtained. The sulphur content could be reduced by as much as 60% with an increased hydrogen content in the liquid products.

EXPERIMENTAL

The apparatus and experimental and analytical procedures were the same as described in a previous report (5). The feed-stock used was topped bitumen separated from the Athabasca tar-sand deposits in northern Alberta. The bitumen was supplied by Great Canadian Oil Sands Ltd., Fort McMurray, Alberta. The properties of the bitumen (Lot No. 98) and distillation results are presented in the previous report (5).

RESULTS AND DISCUSSION

For once-through runs (without heavy-oil recycle) at conditions of reactor temperature 440-460^{o}C, LHSV 1.0 and pressure 13.89 MPa, no operating problems were encountered in the initial stages or for short time intervals. However, when the run time was increased, coke deposition in the reactor or in the hot separator made it extremely difficult to operate the plant, although at higher pressures and space velocities, coke deposition was suppressed and life runs of over 3 weeks were possible. Accordingly, the heavy-oil recycle life runs at low space velocity could only be compared with short once-through runs. Table 1 shows the identification numbers and conditions for different runs. Run numbers 53-4-2 and 82-3-1 represent short once-through runs at a reactor temperature of 450^{o}C and run 53-6-2 represents a short once-through run at a reactor temperature of 460^{o}C. R-2-1-1 and R-2-1-2 represent long runs with heavy-oil recycle at a reactor temperature of 450^{o}C and R-2-2-4 represents a long heavy-oil-recycle run at a reactor temperature of 460^{o}C; other conditions were the same except for the hot-separator (hot catch pot, HCP) temperature. For short once-through runs the latter varied between 300 and 370^{o}C and for runs with heavy-oil recycle it was 450^{o}C. In this section of the report comparisons and discussions will be made under the following sub-headings:

1. Light ends (LE)
2. Heavy ends and recycle oil (HE and RO)
3. Distillate
4. Pitch
5. Effect of operating time on product quality and pitch conversion
6. Effect of temperature
7. Comparison between once-through and recycle runs.

The light ends and heavy ends are products of this pilot plant. The distillate and pitch are products which would be obtained by combining and distilling all of the LE and HE or RO together, and would normally be the only products discussed. However, the RO (or HE) and LE in a commercial plant must be pumped and handled, and for this reason all four are discussed.

1. Light Ends (LE)

During recycle runs the hot separator was maintained at a higher temperature than for corresponding once-through runs. More product, therefore, remained in the vapour state, resulting in greater LE yields, specific gravities and sulphur and nitrogen contents as documented in Table 2.

2. Heavy Ends and Recycle Oil (HE and RO)

Heavy-oil products of the recycle runs and of the once-through runs will be differentiated by naming them RO and HE, respectively.

Table 3 shows the weight per cent yields and properties of the two products. Compared with once-through runs, the yields of RO in general decreased as more of the distillable materials stayed in the vapour state in the hot separator and became light ends because of higher hot-separator temperature. This can also be seen from the distillation of HE and RO. The HE for run 82-3-1 contained 16.5 wt % pitch and 83.5 wt % distillable hydrocarbons whereas RO for run R-2-1-2 contained 44.4 wt % pitch and 55.6 wt % distillable hydrocarbons.

At a reactor temperature of 450°C the specific gravity of RO was greater than that of HE. Ash content, metals, asphaltene*, coke** and Conradson Carbon Residue (CCR) of RO were also greater than those of HE. However, based on total liquid product, CCR and ash content were nearly the same. Coke and asphaltene increased for the recycle run (Table 4). The increased amounts of coke and asphaltene may be due to the higher average residence time of RO, which was of the order of 4 hours compared with an HE residence time of about one hour (5).

Figure 1 shows the LE and RO production rate and the ash content in RO for run R-2-2-4. The feed rate was constant at 4500±50 g/h. When the RO production increased, LE production decreased. Since ash holdup in the reactor was small compared with the total ash fed, it is assumed that the ash input was equal to the ash output. Hence, the amount of ash leaving the unit was constant and, by material balance, the ash wt % in RO decreased as the RO production increased.

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* *Asphaltene is defined as pentane insolubles minus benzene insolubles.*
** *Coke is defined as benzene insolubles minus ash.*

3. Distillate

The distillate represents the fraction of the material in the liquid product boiling below 524°C, excluding C_3 and lighter. Table 5 shows some of the distillate properties. For heavy-oil recycle runs, the specific gravity decreased slightly because of an increase in the fraction boiling below 250°C. Sulphur content was nearly the same for once-through and heavy-oil-recycle runs. Nitrogen content decreased slightly for recycle runs. Table 6 shows the Hempel analysis of the distillates. From this table, it is clear that the fraction boiling below 200°C was greater for heavy-oil recycle runs, whereas the fraction boiling between 418 and 524°C was smaller. Table 6 also shows that there are other distinguishable differences when products from once-through and recycle runs are compared but they are small.

4. Pitch

Table 7 shows the properties of pitch, the fraction boiling above 524°C. Sulphur, ash and mineral content of the pitch were nearly the same for all runs, but nitrogen content decreased slightly for heavy-oil recycle runs. The hydrogen content of pitch was less than 6.8 wt % for the recycle runs, but no coke was found in the reactor or hot separator which is contrary to the observations of Chervenak and Wolk (9).

5. Effect of Operating Time on Product Quality and Pitch Conversion

In continuous runs there are several factors such as feed rate, temperature, gas purity, etc., which affect product quality. In this section, variation in liquid product quality with operating time is reported for recycle runs.

Figures 2-10 show the variation in liquid product quality during run R-2-1-2. Figure 2 shows variation in some properties of the light ends. Sulphur content decreased in the latter stages of the run; it seems that as the ash content of the reactor fluid increased, sulphur in the light ends decreased. Nitrogen content was a maximum on the thirteenth day of the run. It seems that nitrogen removal was affected by feed rate, as the feed rate was a minimum on the twelfth and thirteenth days of the run (5).

Figure 3 shows the variation in RO properties. Ash and sulphur content were nearly constant over the run period but coke and asphaltene decreased in the latter stages of the run when the feed rate decreased.

Figure 4 shows the variation in pitch properties. Specific gravity, sulphur, ash content, asphaltene and fixed carbon (from ultimate analysis) were nearly constant but hydrogen content increased steadily from 5.95 wt % to 6.40 wt %. Coke content of the pitch decreased in the later stages of the run. Figure 5 shows that there was only slight variation in the concentration of vanadium and nickel metals in pitch and recycle oil.

Figure 6 shows the variation in distillate properties. Specific gravity, kinematic viscosity, sulphur, nitrogen and hydrogen content were nearly constant for the duration of the run. Carbon varied between 84 and 86 wt %, which is difficult to explain. It could be caused by systematic error in sampling or analysis.

Figure 7 shows the variation in different distillate fractions. Slight variations in initial stages of the run may be due to random analysis error. In the later stages of the run when the fraction boiling between 418 and 524^{o}C decreased from 9 vol % to 6 vol %, other fractions increased slightly. Feed rate also decreased slightly in the later stages of the run (5) resulting in a greater residence time for feed (first pass) and more of the higher boiling material was perhaps converted to lower boiling fractions.

Figures 8-10 show the variation in the properties of different distillate fractions. Specific gravity, sulphur, nitrogen and saturates were nearly constant. Variation in aromatics and olefins may be due to the method of analysis. Because of difficulty in the separation of aromatics and olefins, analysis of these fractions is unreliable. However, analysis for saturates and the sum of olefins and aromatics are reliable. The sum of olefins and aromatics was nearly constant over the run duration.

Figures 11-19 show the variation in the properties of the liquid products for run R-2-2-4. These variations were much less than in run R-2-1-2 because of the method of controlling the hot separator level (5).

Figure 20 shows pitch conversion with respect to operating time. At 460^{o}C (Run R-2-2-4) pitch conversion increased initially following an increase in ash content of reactor fluid, but it decreased after the sixth day of operation. On increasing the feed rate, pitch conversion has been found to decrease (3). For the run at 460^{o}C (R-2-2-4) the actual feed rate increased slightly as shown in Reference 5, and consequently the pitch conversion decreased. At 450°C (Run R-2-1-2) pitch conversion was initially about 81 wt % but it steadily increased to 85.5 wt % during the run time and actual feed rate decreased over the run duration.

In general, there was not much variation in product quality during the operation of recycle runs.

6. Effect of Temperature

An increase in reactor temperature usually affects yields, pitch conversion and liquid product quality. In the present investigation, recycle experiments were conducted at two temperatures, namely 450 and 460°C. Although there was no coke deposition in the reactor or in the hot separator for either run, temperature had a marked effect on yields, pitch conversion, sulphur and nitrogen removal and a minor effect on distillate properties. Yields, hydrogen consumption and product properties on an average basis are presented in Tables 8 and 9.

Total liquid product yields (92.3 and 91.8 wt % at 450 and 460°C respectively) and hydrocarbon gas make (2.03 and 2.05 g mol/kg of feed at 450 and 460°C respectively) were nearly the same for both. Distillate yields, pitch conversion, sulphur removal and hydrogen consumption increased at higher reactor temperature (460°C) from 81.1 to 84.2 wt %, 81.5 to 85.4 wt %, 52.2 to 57.2 wt % and 7.76 to 9.25 g mol/kg feed respectively. Increase in hydrogen consumption was due to higher distillate yields, pitch conversion and sulphur removal. Nitrogen removal was nearly the same for both runs.

Specific gravity and sulphur content of the distillate decreased slightly at higher reactor temperature because of higher sulphur removal, whereas nitrogen content of distillate and pitch was nearly the same.

7. Comparison between Once-through and Recycle Runs

At a reactor temperature of 450°C, total liquid product yields and distillate yields decreased slightly for recycle runs (Table 8) but pitch yields increased from 9.0 to about 11.2 wt %. Hydrocarbon gas make was nearly the same. Pitch conversion decreased from 82.7 to 81.5 wt % but hydrogen consumption increased from 6.73 to 7.76 g mol/kg feed for the recycle run. Distillate specific gravity decreased from about 0.904 to 0.886 and pitch gravity increased from 1.188 to 1.251. Sulphur contents of the distillate and pitch were nearly the same for once-through and recycle runs as was sulphur removal. Pitch conversion for the recycle run decreased but hydrogen consumption increased, resulting in a higher hydrogen content in the distillate.

At a reactor temperature of 460°C (Runs 53-6-2 and R-2-2-4) similar trends in distillate and pitch yields, product quality and sulphur and nitrogen removal were observed (Tables 8 and 9).

Compared with once-through runs, pitch conversion decreased and product quality improved slightly for recycle runs. Sulphur removal was not much affected but nitrogen removal increased from 4.12 to 17.8 wt % for recycle run.

CONCLUSIONS

The fouling of the reactor or the hot separator because of coke or sludge deposits was very much reduced by recirculating a fraction of heavy ends (RO). In general, pitch conversion decreased but there was an improvement in the product quality for the recycle runs. Sulphur removal was only slightly affected but nitrogen removal increased for recycle runs compared with once-through operation. For example, at a reactor temperature of 450°C for recycle runs compared with once-through runs, pitch conversion decreased to about 81.5 wt % from 82.7 wt %. Sulphur removal was nearly constant and nitrogen removal increased to about 17.8 wt % from about 4.12 wt %.

Distillate yields decreased slightly for the recycle runs. The sulphur content of the distillate for once-through and recycle runs was nearly the same. Nitrogen content decreased slightly. The decrease in specific gravity of the distillate for recycle runs was due to an increase in the fraction boiling below 250°C and increased hydrogen content.

The sulphur content of the pitch was nearly the same but nitrogen content decreased slightly for recycle runs. Although the hydrogen content of the pitch was less than 6.8 wt %, no coke was found in the reactor, which is contrary to the findings of Chervenak and Wolk (9). Similar product quality improvements were observed at a reactor temperature of 460°C for recycle runs.

For the recycle runs, reactor temperature had a marked effect on conversion yields and sulphur removal and a minor effect on product quality. Pitch conversion at 460°C reactor temperature as compared with 450°C increased to 85.4 wt % from 81.5 wt %, sulphur removal increased to 57.2 wt % from about 52.2 wt % and distillate yields increased to 84.2 wt % from about 81.1 wt %. The distillate properties for both recycle runs (R-2-1-2 and R-2-2-4) were nearly the same but the fraction boiling below 250°C increased at the higher reactor temperature.

During the course of the run, liquid properties and yields were nearly constant. Slight variation in yields and properties could be due to variation in the feed rate or RO withdrawal rate.

ACKNOWLEDGEMENTS

The authors wish to thank M. Pleet and the staff who operated and maintained the pilot plant. The contribution of R.G. Draper and his laboratory staff for chemical analysis is gratefully acknowledged.

The assistance of G. Noel who performed the calculations, C.W. McNabb who completed the shut-down reports and R. Chagnon who prepared the drawings is much appreciated.

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TABLE 1

Identification and Conditions of Different Runs

Run No.	Duration h	Pressure MPa	Gas Flow g mol/h	H_2 Purity %	LHSV h^{-1}	Reactor Temp °C	HCP Temp °C	Actual Feed Flow g/h	Recycle Oil Flow g/h	Recycle Oil Feed Flow g/h
53-4-2	3	13.89	1.158	85	1.0	450	300	4135	-	0.0
53-6-2	3	13.89	1.158	85	1.0	460	300	4103	-	0.0
82-3-1	4	13.89	1.158	85	1.0	450	370	4475	-	0.0
R-2-1-1	93	13.89	1.158	85	1.0	450	450	4546	-	-
R-2-1-2	477	13.89	1.158	85	1.0	450	450	4535	9060	2.0
R-2-2-4	283	13.89	1.158	85	1.0	460	450	4554	12700	2.8

TABLE 2

Light Ends Yields and Properties

Reactor Temp °C		450			460	
Run Number		82-3-1	R-2-1-1	R-2-1-2	53-6-2	R-2-2-4
HCP Temp	°C	370	450	450	300	450
Yield on feed	wt %	39.4	70.8	69.6	30.2	72.3
Yield on total liquid product	wt %	42.2	76.3	77.1	33.5	81.7
API Gravity		37.8	29.3	30.8	44.1	31.3
Specific Gravity		0.838	0.880	0.872	0.806	0.869
Sulphur	wt %	1.68	2.45	1.98	-	1.77
Nitrogen	ppm	1533	-	2436	-	2132

TABLE 3

Heavy Ends and Recycle Oil Yields and Properties

Reactor Temperature		450°C			460°C	
Run Number		82-3-1	R-2-1-1	R-2-1-2	53-6-2	R-2-2-4
Yield on feed	wt %	53.88	22.00	20.74	59.9	16.43
Yield on total liquid product	wt %	57.77	23.70	22.95	66.53	18.33
API Gravity		10.0	-11.6	-2.3	15.3	-6.2
Specific gravity		1.000	1.180	1.095	0.964	1.129
S	wt %	2.99	4.085	3.68	-	3.59
Ash	wt %	1.22	2.507	2.67	-	3.53
CCR	wt %	11.40	-	30.14	-	36.52
Ni	ppm	97	-	241	-	361
V	ppm	188	-	755	-	1041
PI*	wt %	9.30	-	30.62	-	38.15
BI*	wt %	3.39	-	10.82	-	14.95
A*	wt %	5.91	-	19.82	-	23.2
C*	wt %	2.17	-	8.13	-	11.42
N	ppm	5998	-	8916	-	-
Distillate	wt %	83.5	45.8	55.6	92.7	54.2
Distillate	sp gr	0.962	0.997	0.990	0.942	1.004
Pitch	wt %	16.5	54.2	44.4	7.3	45.8

*PI - Pentane insolubles
BI - Benzene insolubles
A - Asphaltene
C - Coke

TABLE 4

Amounts of Asphaltene, Coke, etc. in Total Liquid Product

Run Number		82-3-1	R-2-1-2
Asphaltene	wt %	3.41	4.55
Coke	wt %	1.25	1.87
CCR	wt %	6.58	6.91
Ash	wt %	0.70	0.60

TABLE 5

Properties of the Distillate

Reactor Temp ^{o}C	450				460	
Run	Once-through		Recycle		Once-through	Recycle
Run Number	53-4-2	82-3-1	R-2-1-1	R-2-1-2	53-6-2	R-2-2-4
Sp Gr $60/60^{o}F$	0.906	0.904	0.893	0.886	0.894	0.884
S wt %	2.28	2.30	2.48	2.16	2.01	2.03
Kinematic viscosity cst	-	4.81	-	3.21	-	2.965
N wt %	-	0.31	-	0.25	-	0.27
C wt %	-	-	-	84.73	-	85.08
H wt %	-	-	-	11.66	-	11.59

TABLE 6

Hempel Analysis of the Distillate

Reactor Temp °C	450				460	
Run	Once-through		Recycle		Once-Through	Recycle
Run Number	53-4-2	82-3-1	R-2-1-1	R-2-1-2	53-6-2	R-2-2-4
IBP to 200°C						
vol %	18.9	21.7	22.2	24.5	22.2	27.1
sp gr	0.759	0.767	0.750	0.760	0.760	0.756
S wt %	1.00	1.00	0.96	0.78	0.73	0.61
N wt %	-	0.09	-	0.06	-	0.07
200 to 250°C						
vol %	12.4	9.5	12.2	13.3	14.0	14.8
sp gr	0.848	0.857	0.848	0.854	0.852	0.857
S wt %	1.79	1.81	1.75	1.61	1.60	1.53
N wt %	-	0.11	-	0.09	-	0.11
250 to 333°C						
vol %	28.3	29.9	26.5	27.1	28.3	26.0
sp gr	0.902	0.910	0.904	0.908	0.905	0.912
S wt %	2.36	2.37	2.35	2.26	2.16	2.16
N wt %	-	0.17	-	0.15	-	0.18
333 to 418°C						
vol %	26.2	24.2	25.1	24.2	24.2	22.1
sp gr	0.959	0.962	0.958	0.969	0.965	0.976
S wt %	2.64	2.65	2.76	2.64	2.31	2.58
N wt %	-	0.33	-	0.38	-	0.45
418 to 524°C						
vol %	12.8	13.4	11.4	8.3	9.5	7.6
sp gr	1.044	1.036	1.040	1.052	1.050	1.073
S wt %	3.43	3.23	3.41	3.46	3.19	3.46
N wt %	-	0.80	-	0.93	-	1.14

TABLE 7

Properties of the Pitch (+524°C Fraction)

Reactor Temp °C	450				460	
Run	Once-through		Recycle		Once-Through	Recycle
Run Number	53-4-2	82-3-1	R-2-1-1	R-2-1-2	53-6-2	R-2-2-4
Specific Gravity	1.246	1.253	1.274	1.251	1.312	1.268
S wt %	4.68	4.06	4.54	4.50	4.06	4.09
CCR wt %	67.0	64.6	-	63.4	77.0	72.6
N wt %		1.87	--	1.67		1.81
Ash wt %		7.13	4.73	6.14		7.37
Ni ppm		637	525	514		650
V ppm		1428	1455	1647		2166
BI * wt %		25.3		24.06		33.90
PI * wt %				84.84		88.55
Asphaltene wt %				60.78		54.65
Coke wt %		18.17		17.92		26.53
C wt %				81.00		80.91
H wt %				6.20		5.59

*BI - Benzene insolubles; PI - Pentane insolubles

TABLE 8

Yields and Pitch Conversion for Different Runs

Run Number	Operating Temperature	Yields: Total Liquid Product		Yields: $-524^{o}C$ Oil Distillate		Yields: $+524^{o}C$ Pitch		Pitch Conversion	Hydro-Carbon Gas Make	H_2S Formation
	^{o}C	wt %*	vol %	wt %*	vol %	wt %*	vol %	wt %	g mol/kg	g mol/kg
82-3-1	450	95.5	102.1	86.5	94.5	9.0	7.6	82.7	1.99	0.718
53-6-2	460	90.9	98.8	86.5	95.6	4.3	3.2	91.6	3.2	0.875
R-2-1-1	450	94.7	100.8	82.6	87.8	12.1	13.0	76.9	1.99	0.645
R-2-1-2	450	92.3	99.7	81.1	92.1	11.2	7.6	81.5	2.03	0.674
R-2-2-4	460	91.8	99.7	84.2	93.7	7.6	6.0	85.4	2.05	0.751

* Sulphur-free basis

TABLE 9

Properties of the Liquid Products

Run Number	Reactor Temp °C	Specific Gravity				Sulphur wt %				Nitrogen wt %				Hydrogen g mol/kg feed		
		LE	HE	Fraction		Total Liq Prod	Fraction		Removal	Total Liq Prod	Fraction		Removal	Fed	Dissolved	Consumed
				−524°C	+524°C		−524°C	+524°C			−524°C	+524°C				
82-3-1	450	0.838	1.000	0.904	1.188	2.44	2.30	4.06	52.3	0.41	0.31	1.87	4.12	8.07	1.34	6.73
53-6-2	460	0.806	0.964	0.890	1.360	2.12	2.04	4.06	59.4	-	-	-	-	-	-	8.55
R-2-1-1	450	0.880	1.118	0.893	1.275	2.75	2.18	4.54	46.0	-	-	-	-	7.62	1.07	6.55
R-2-1-2	450	0.872	1.095	0.886	1.251	2.38	2.16	4.50	52.2	0.39	0.25	1.58	17.8	8.70	.94	7.76
R-2-2-4	460	0.869	1.129	0.884	1.268	2.14	2.03	4.09	57.2	0.40	0.27	1.81	16.8	10.41	1.16	9.25

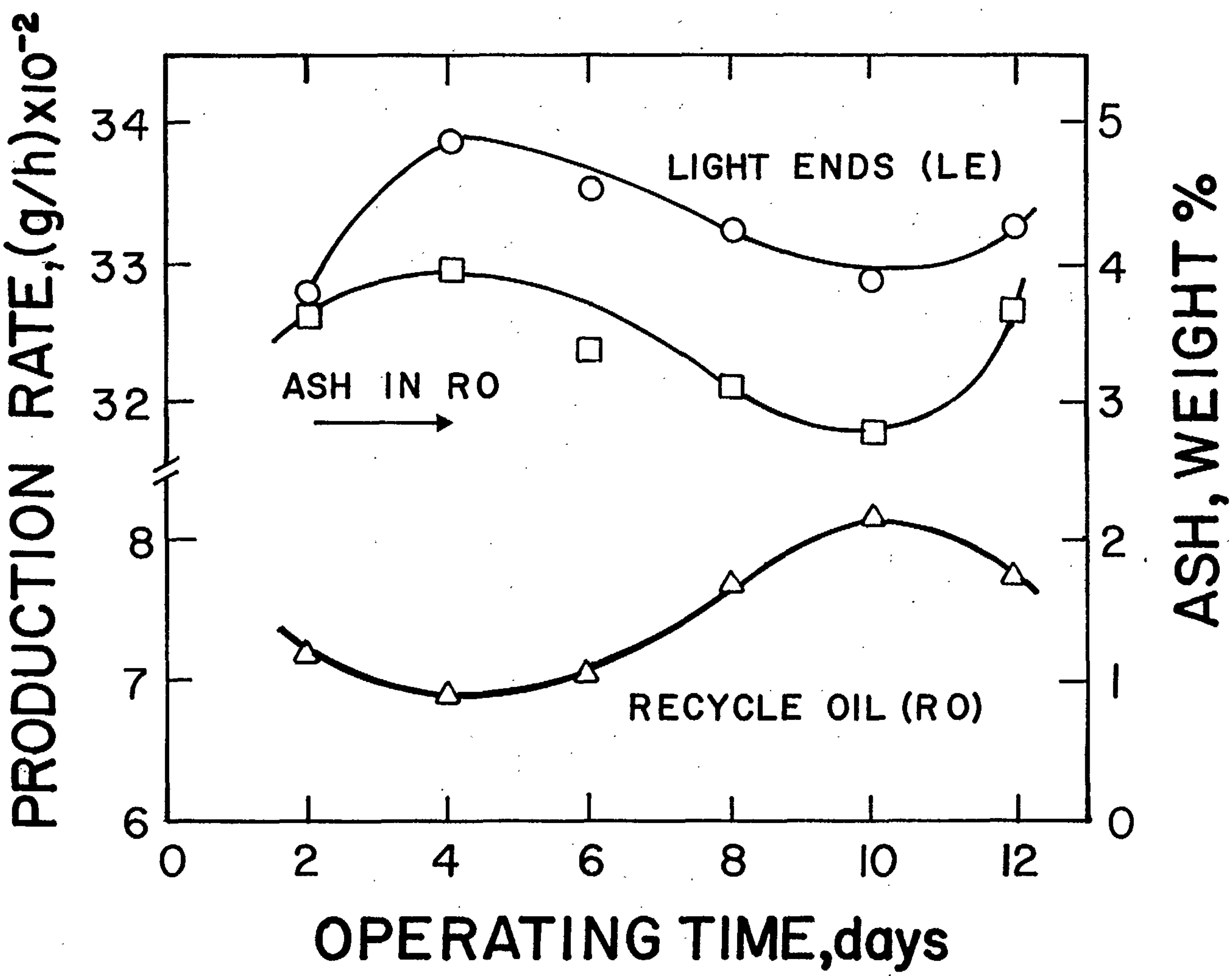

FIGURE 1 - Variation of Recycle Oil and Light Ends Production and Ash Content of the Recycle Oil (R-2-2-4)

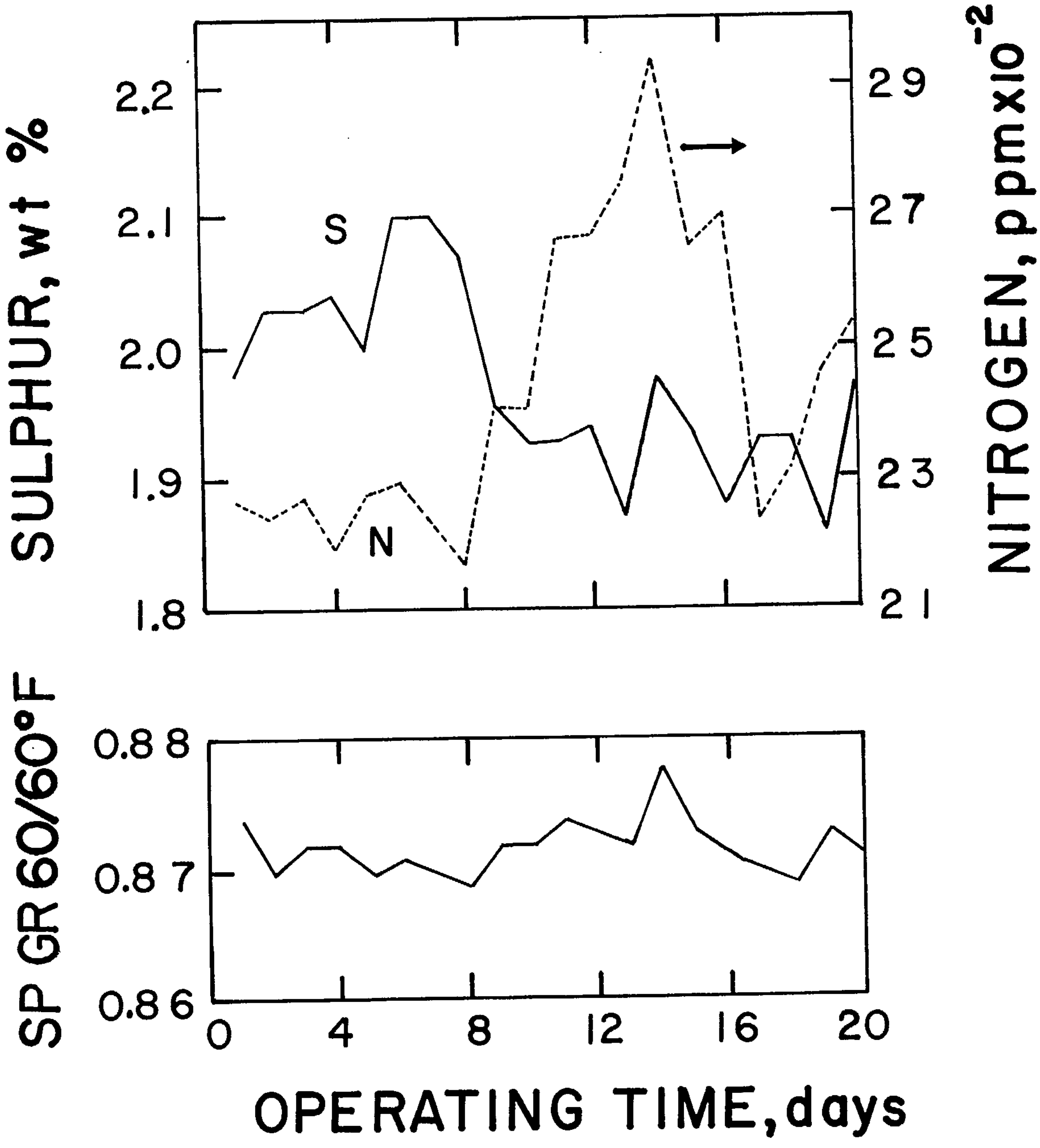

FIGURE 2 - Variation in Light End Properties (R-2-1-2)

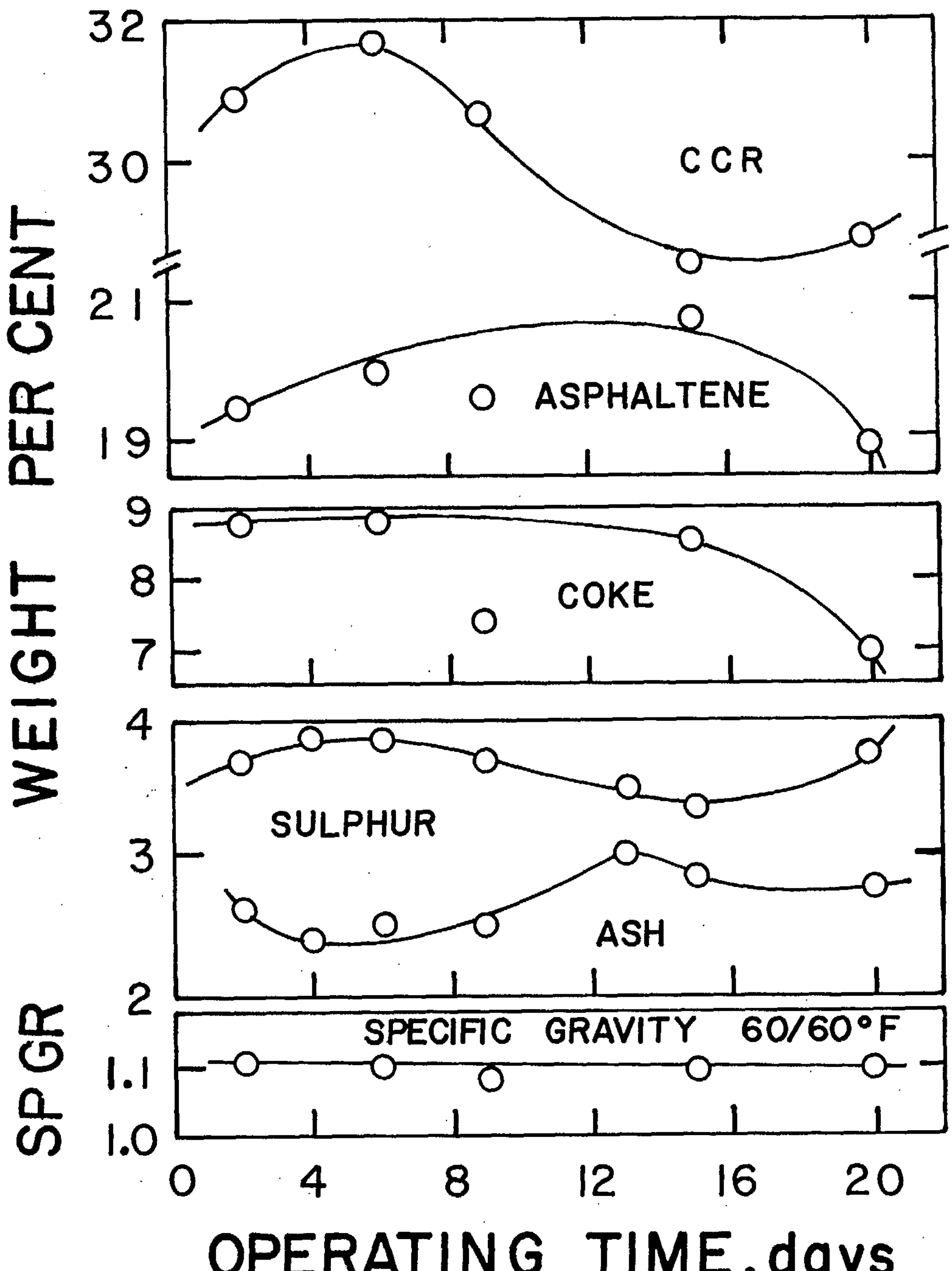

FIGURE 3 - Variation in Recycle Oil (RO) Properties (R-2-1-2)

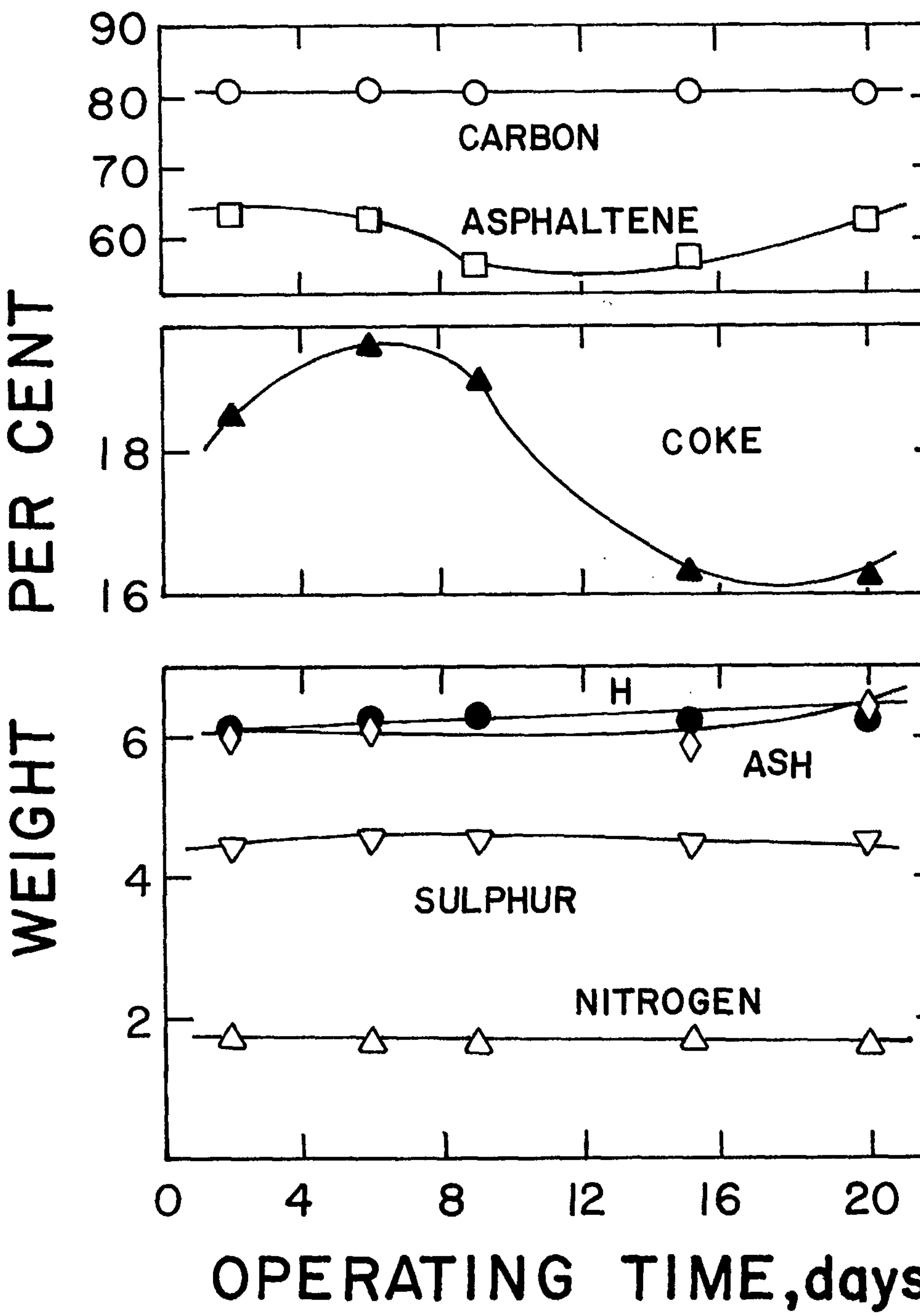

FIGURE 4 - Variation in Pitch Properties (R-2-1-2)

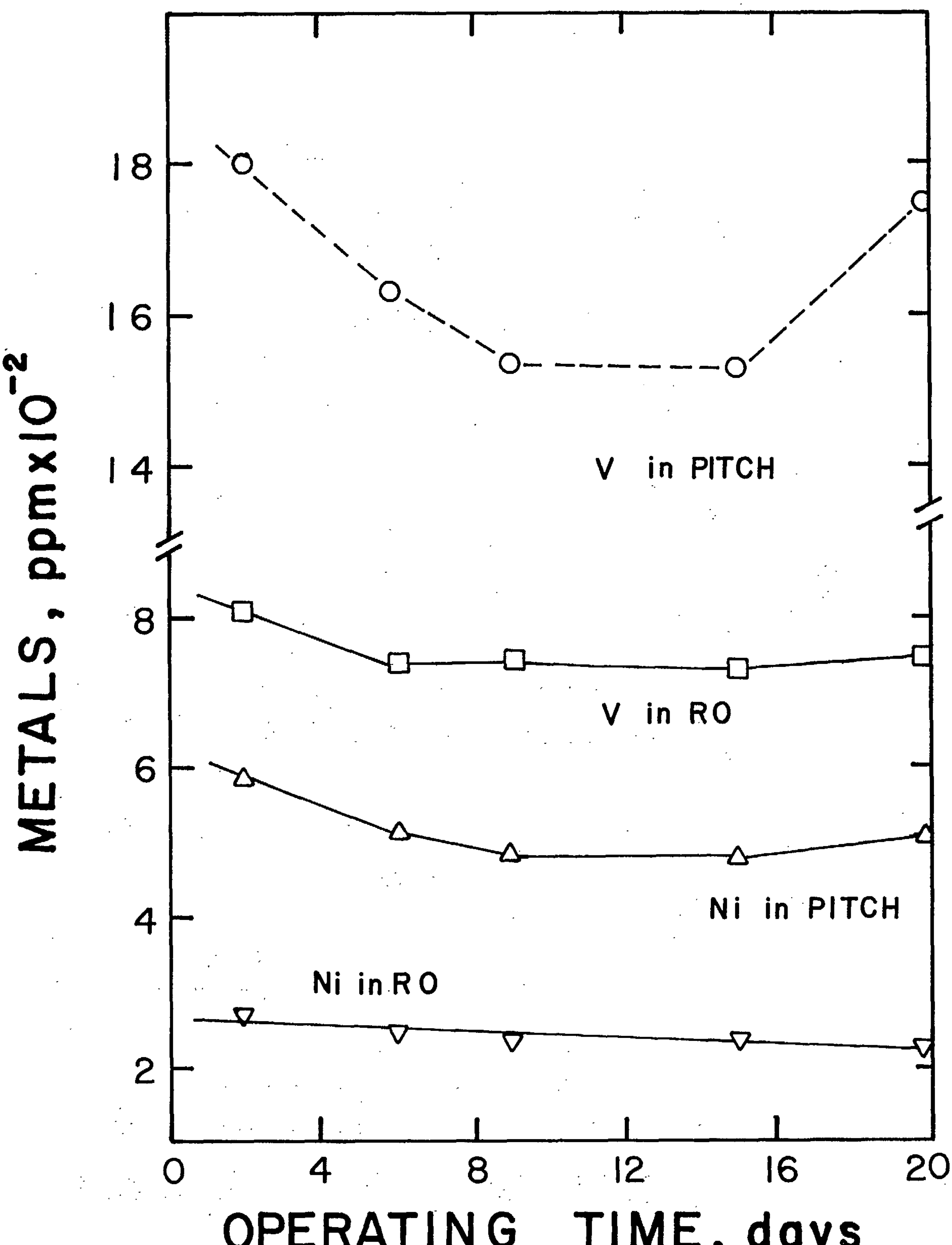

FIGURE 5 - Variation in Metal Contents of Pitch and Recycle Oil (R-2-1-2)

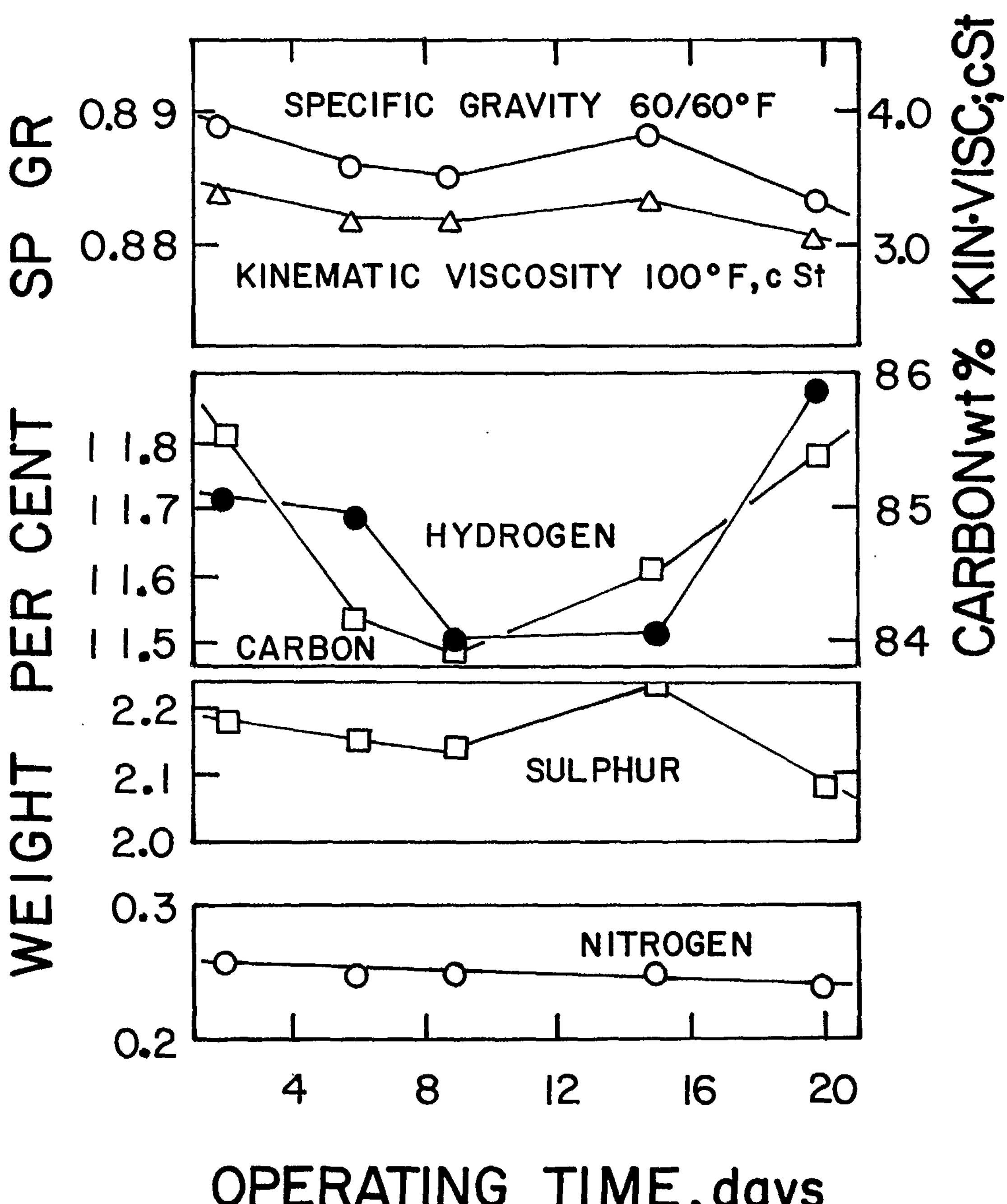

FIGURE 6 - Variation in Distillate Properties (R-2-1-2)

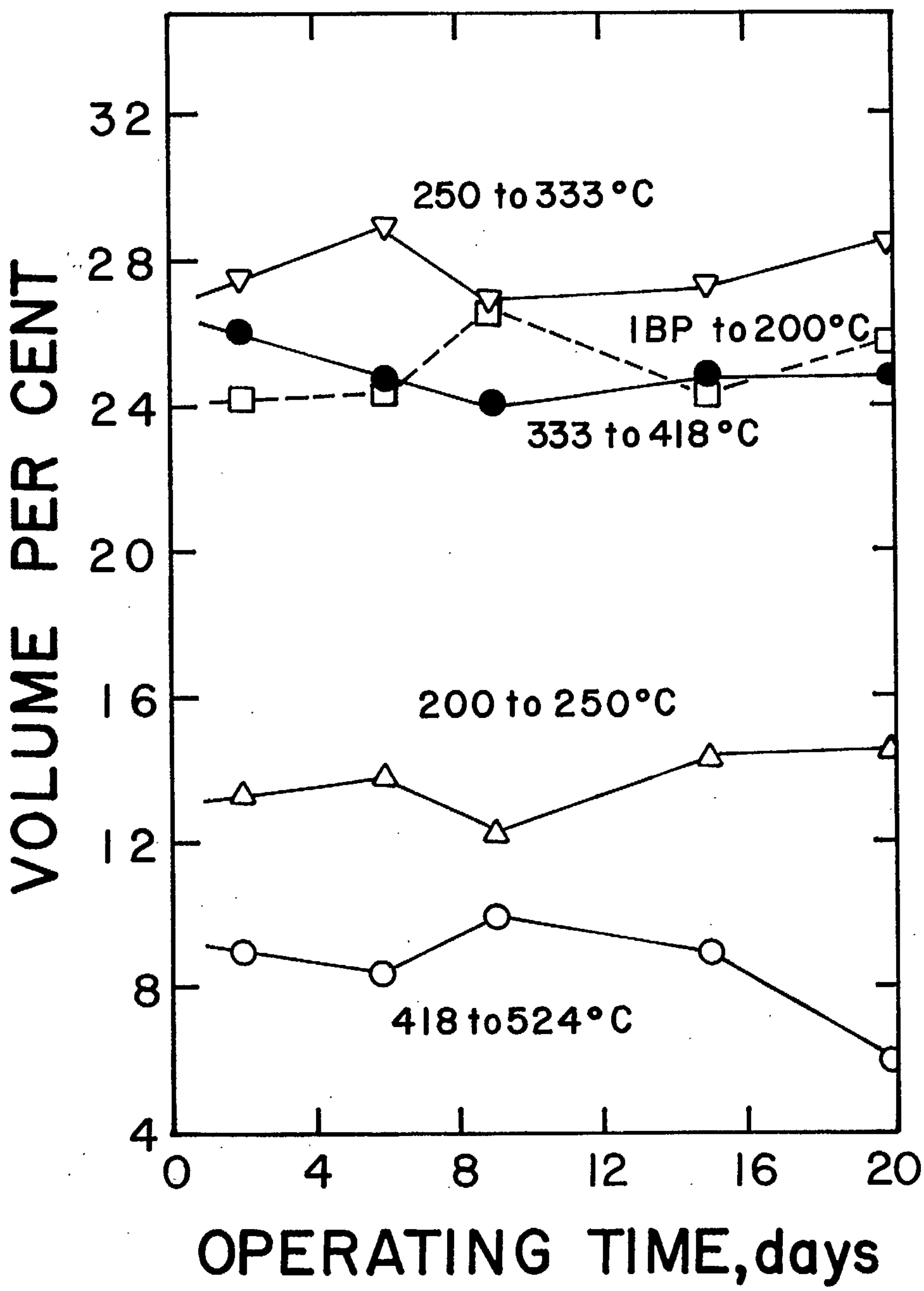

FIGURE 7 - Variation in the Yields of Different Fractions of the Distillate (R-2-1-2)

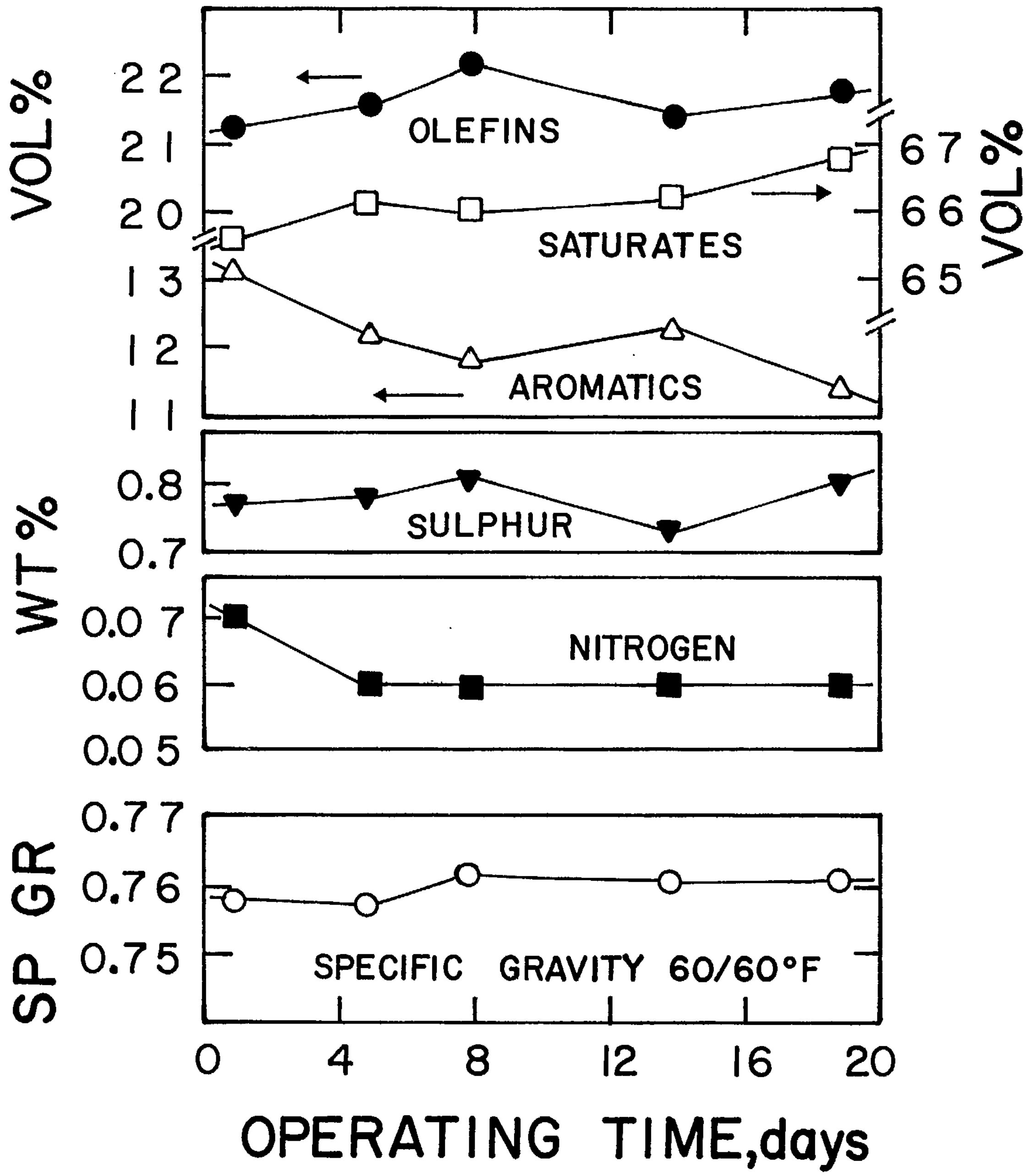

FIGURE 8 - Variation in the Properties of the Distillate Fraction Boiling Between IBP-200^{o}C (R-2-1-2)

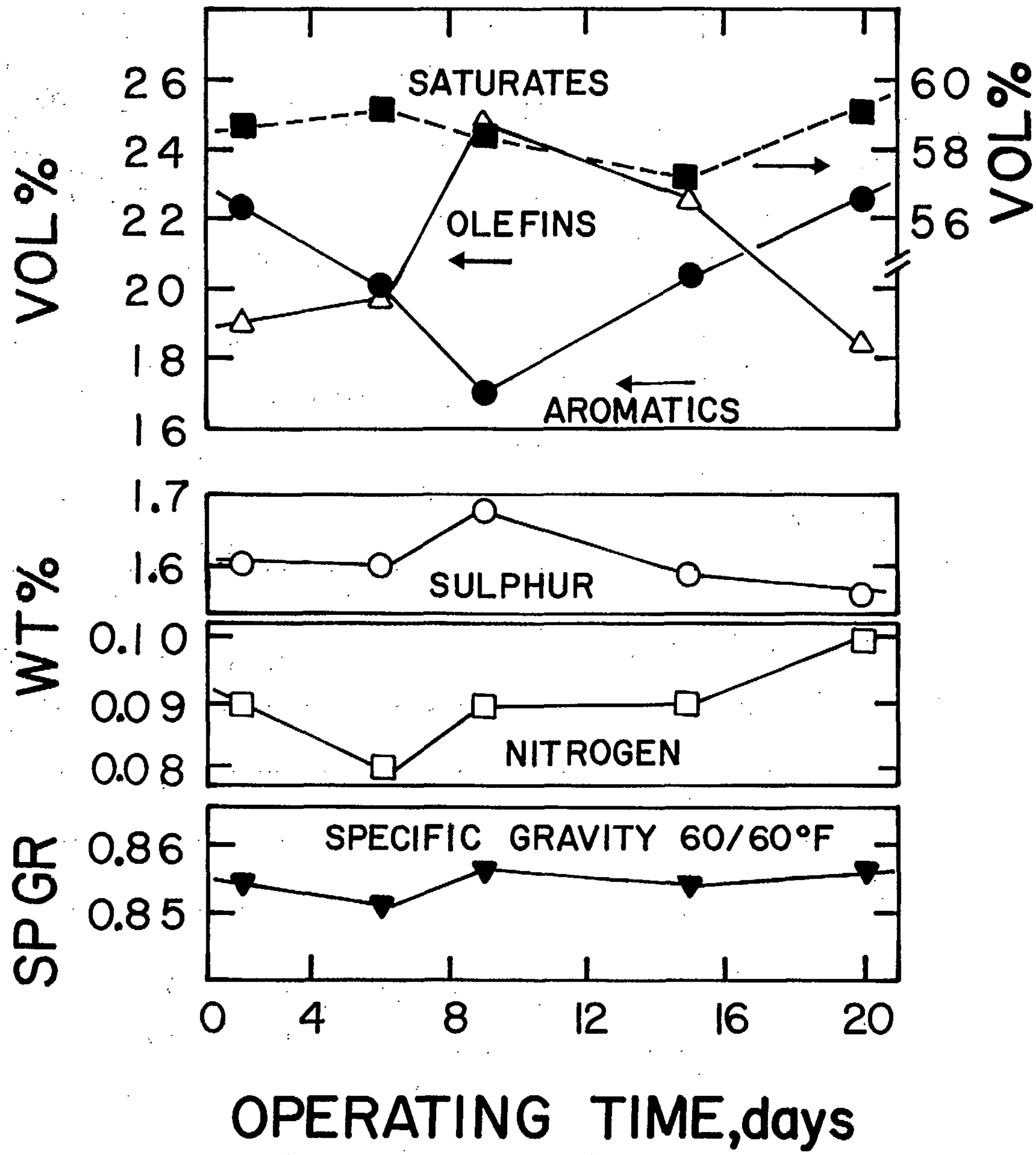

FIGURE 9 - Variation in the Properties of the Distillate Fraction Boiling Between 200-250°C (R-2-1-2)

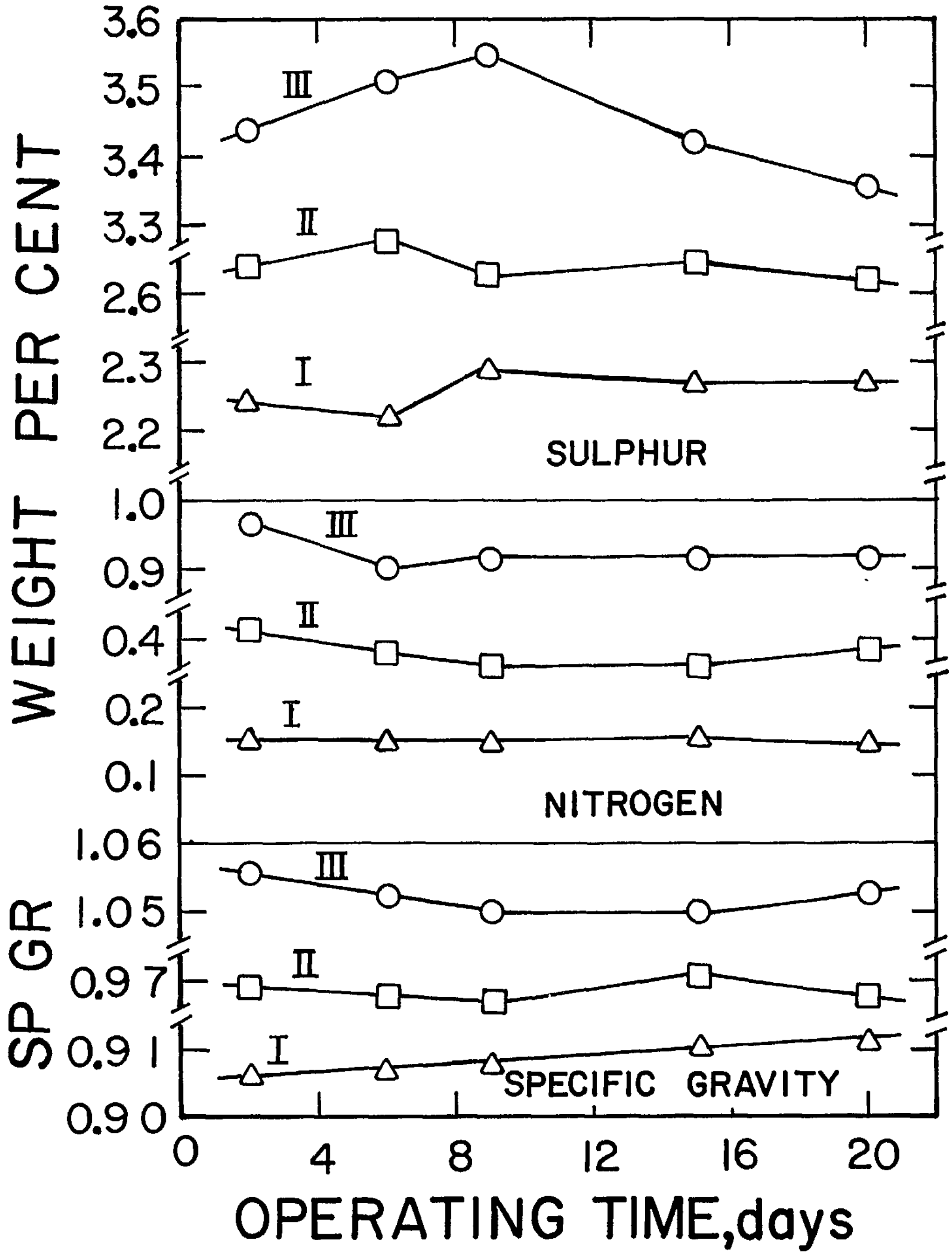

FIGURE 10 - Variation in the Properties of Different Distillate Fractions (R-2-1-2)

I, 250-333°C, II, 333-418°C, III, 418-524°C

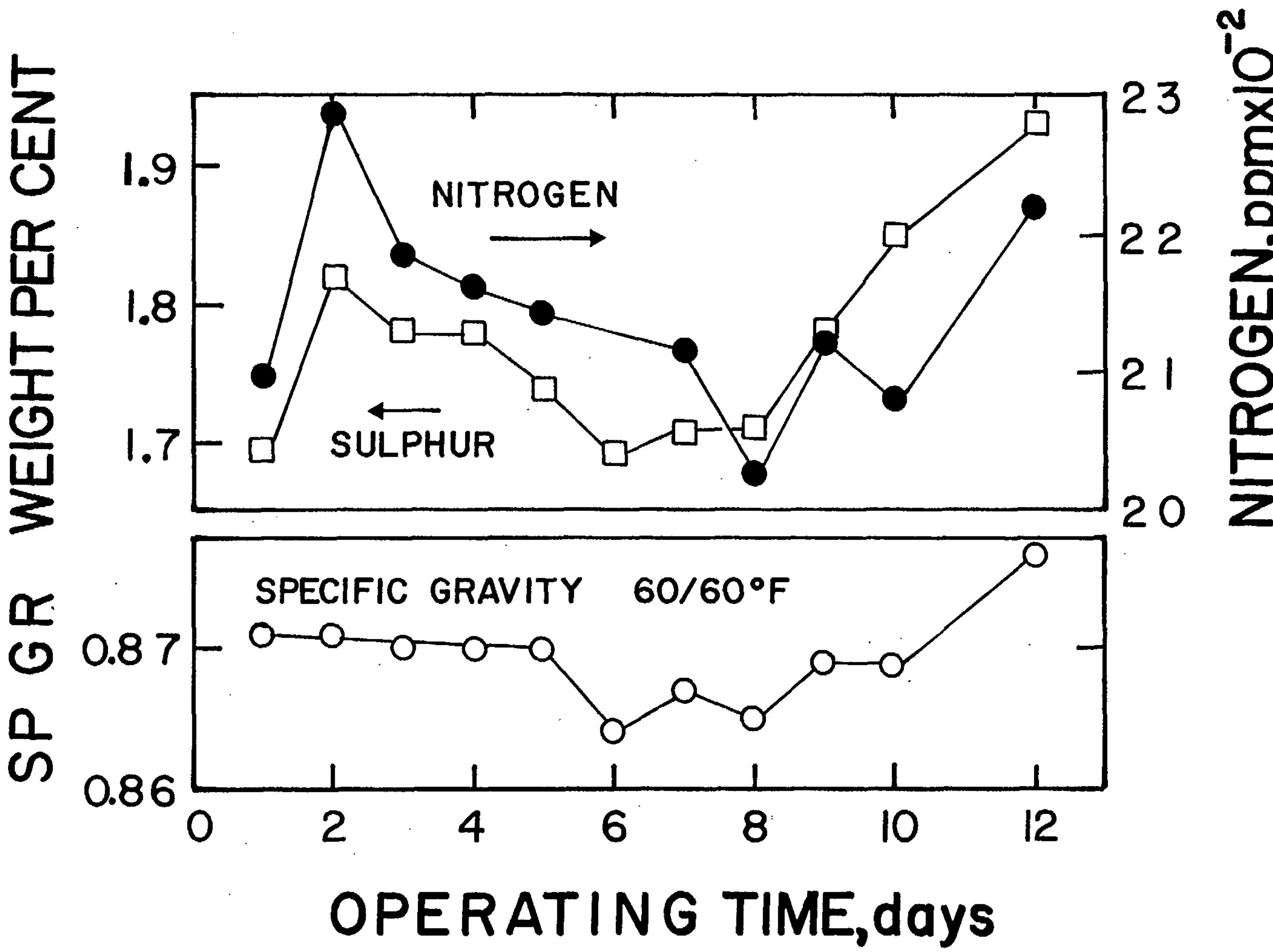

FIGURE 11 - Variation in the Light End Properties (R-2-2-4)

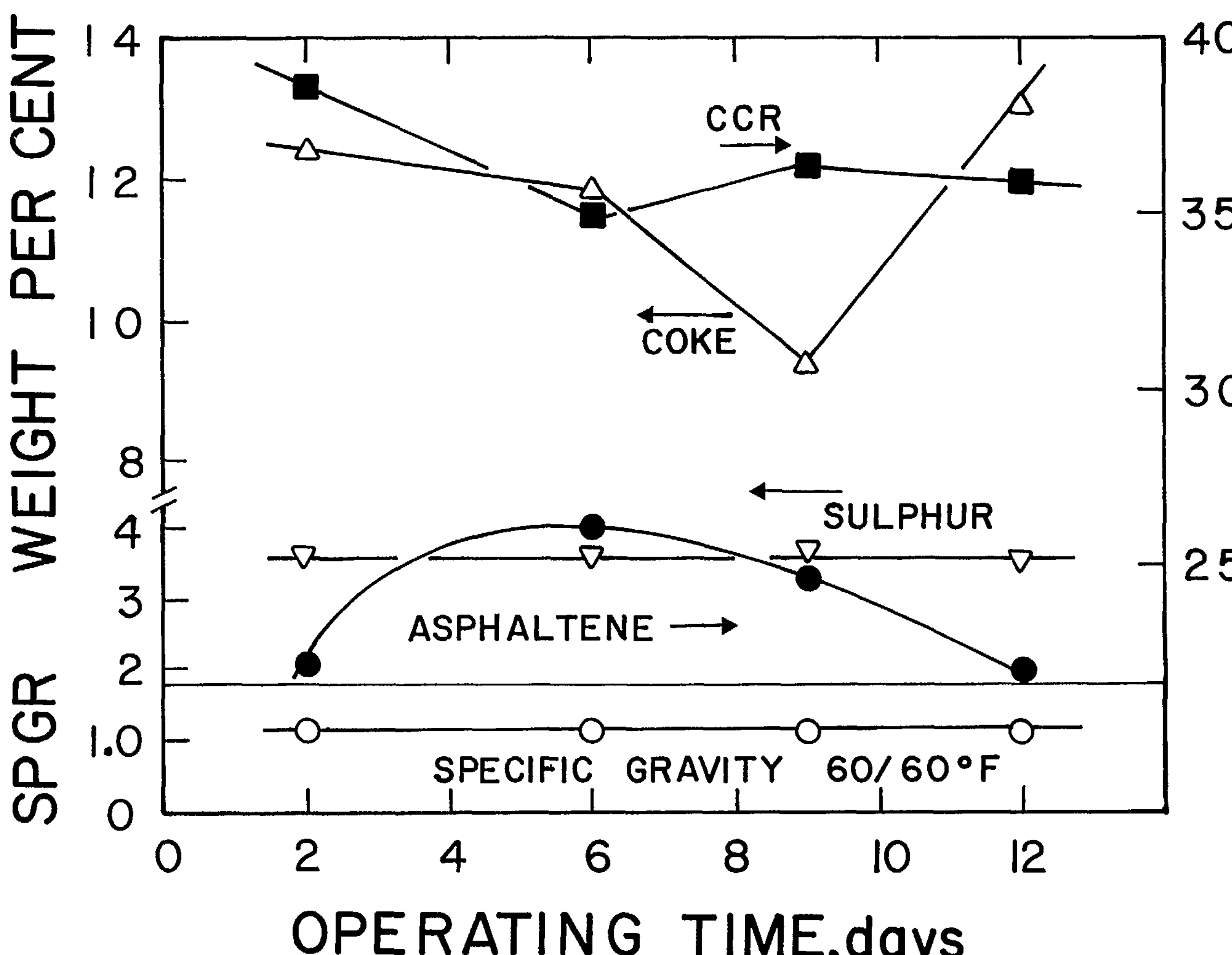

FIGURE 12 - Variation of Recycle Oil Properties (R-2-2-4)

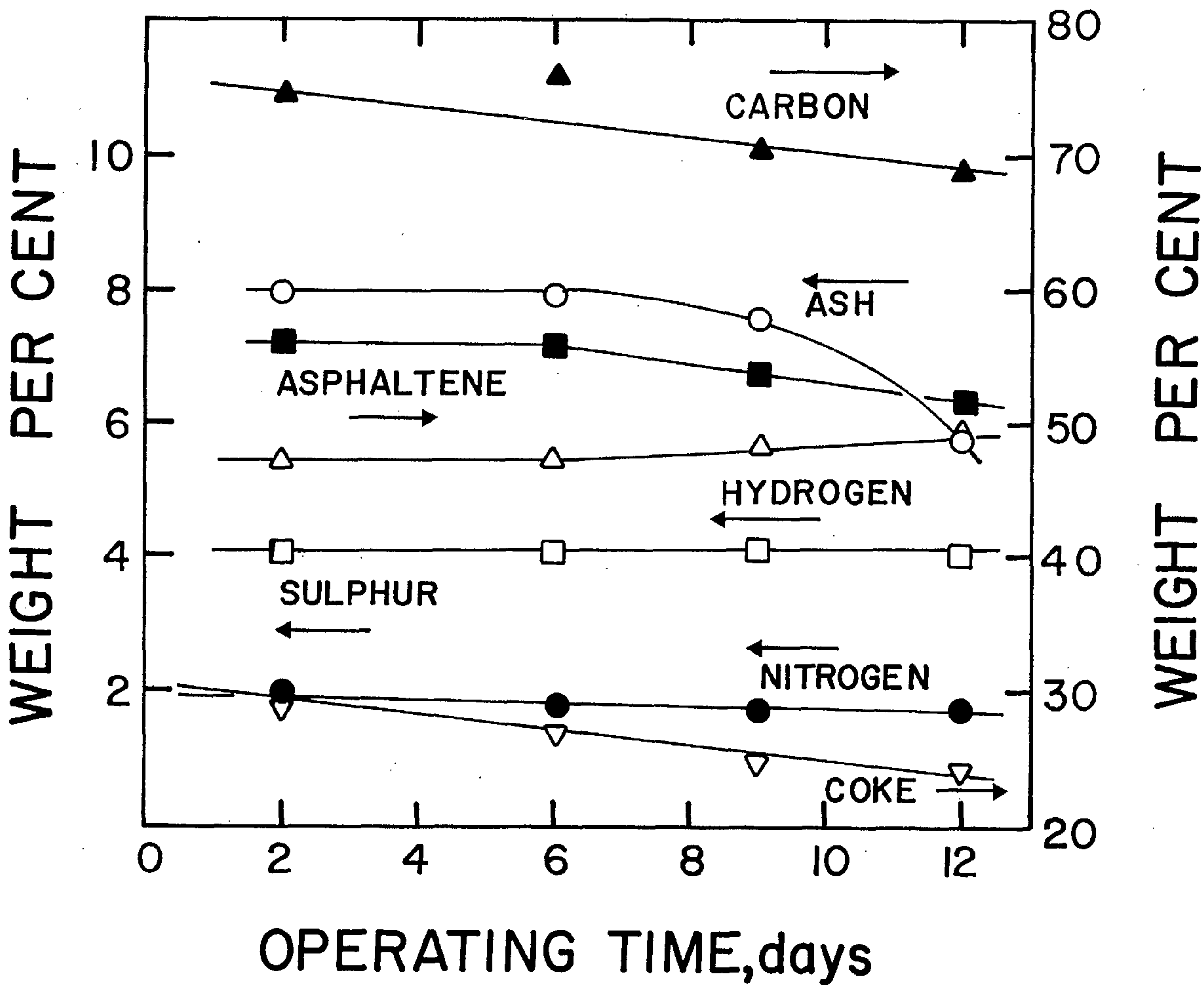

FIGURE 1 - Variation in the Pitch Properties (R-2-2-4)

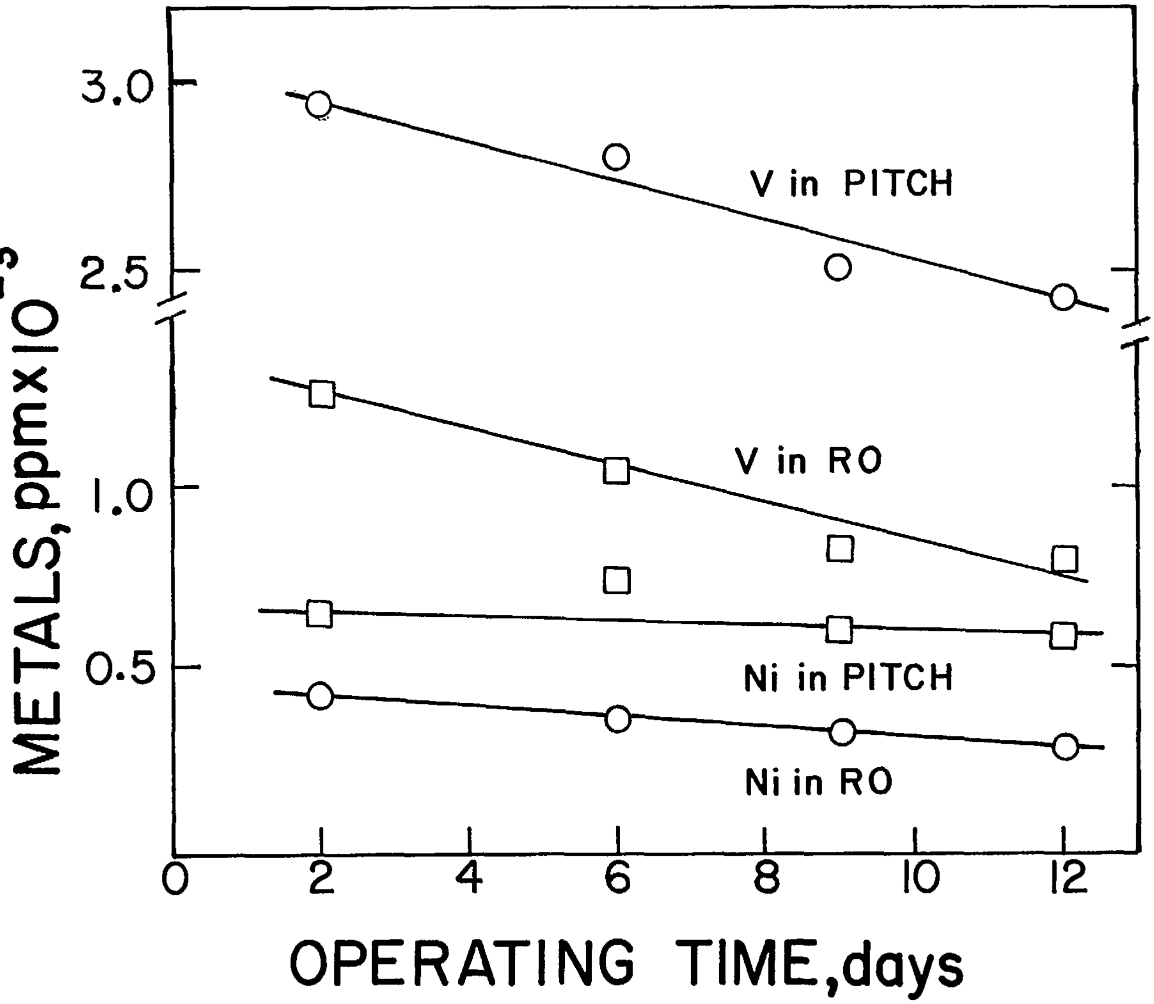

FIGURE 14 - Variation in the Metal Content of Pitch and Recycle Oil (R-2-2-4)

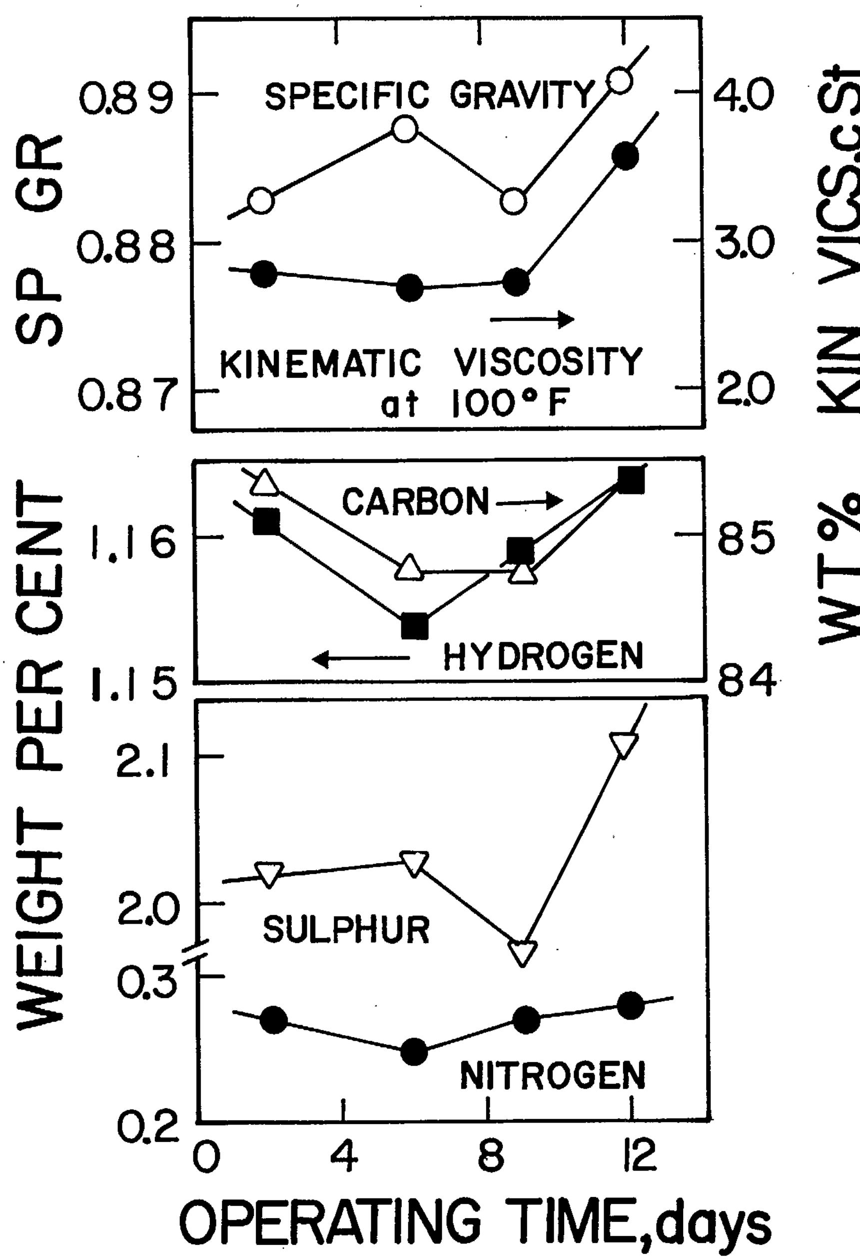

FIGURE 15 - Variation in the Distillate Properties (R-2-2-4)

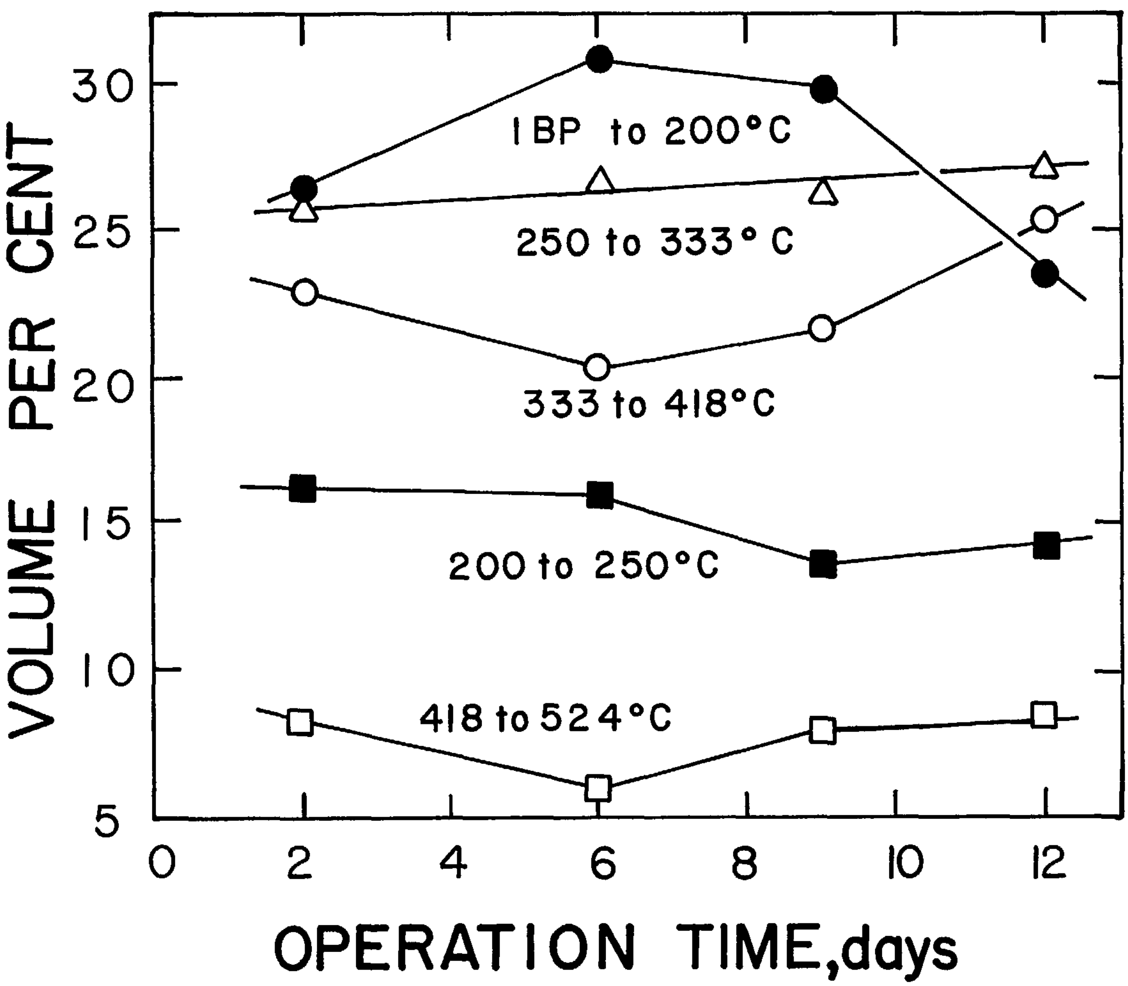

FIGURE 16 - Variation in the Yields of Different Fractions of the Distillate (R-2-2-4)

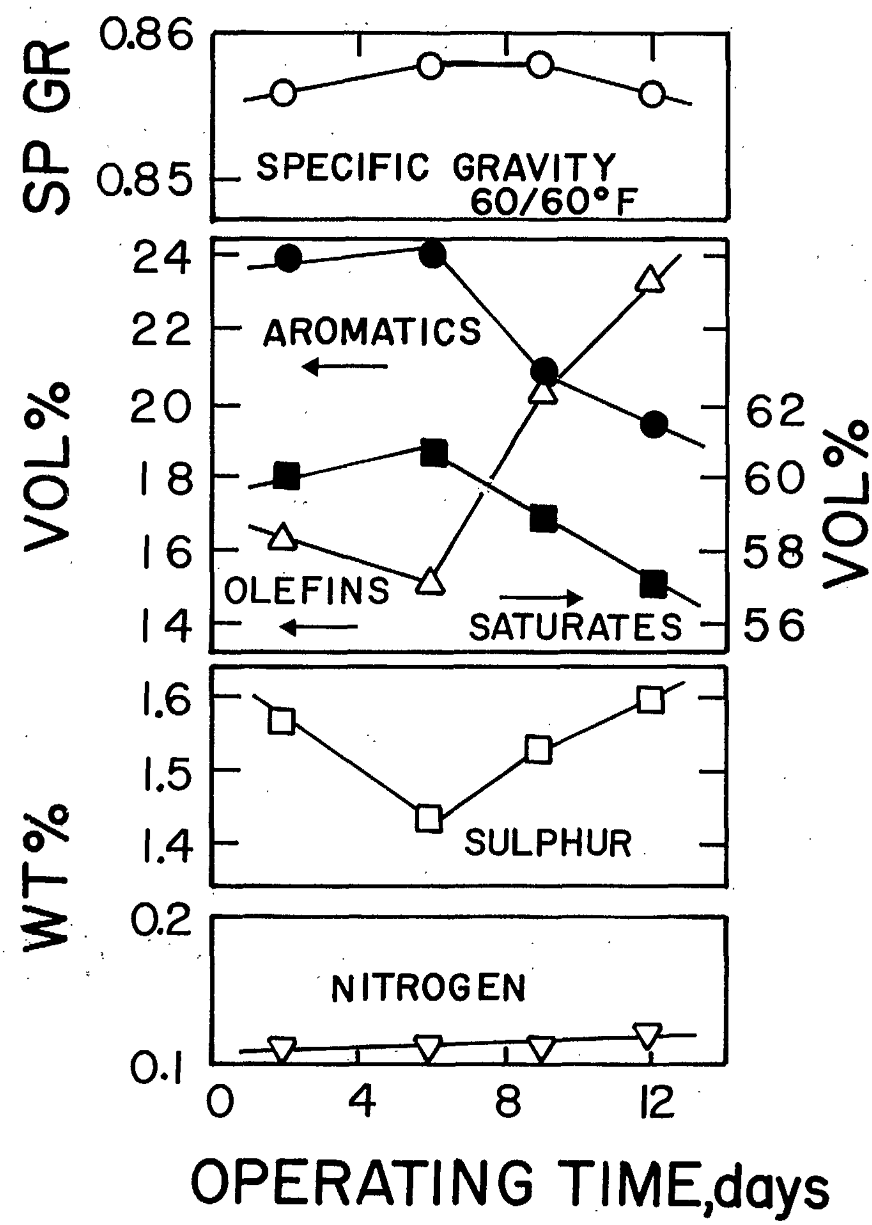

FIGURE 17 - Variation in the Properties of the Distillate Fraction Boiling Between IBP-200°C (R-2-2-4)

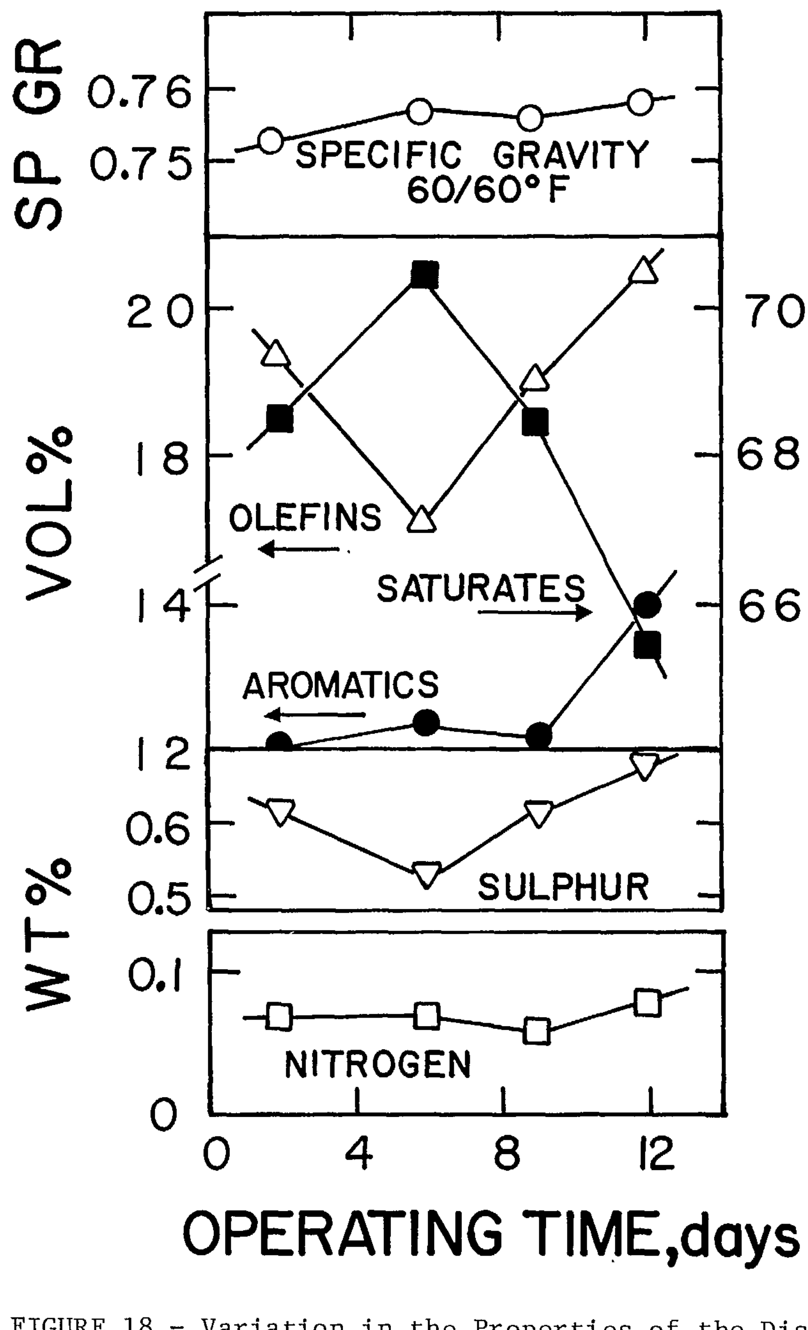

FIGURE 18 - Variation in the Properties of the Distillate Fraction Boiling Between 200-250°C (R-2-2-4)

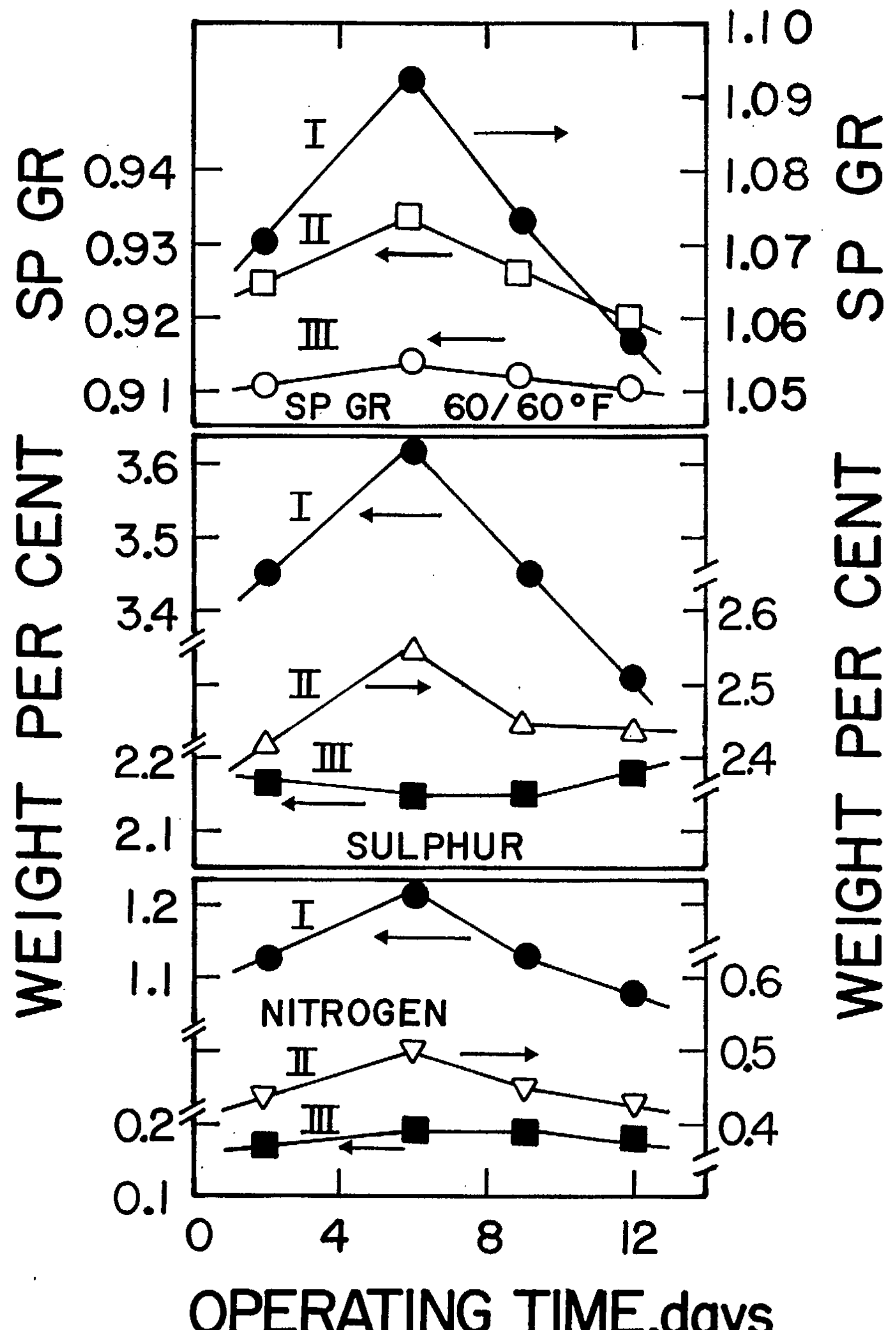

FIGURE 19 - Variation in the Properties of Different Distillate Fractions (R-2-2-4)

I, 418-524°C; II, 333-418°C; III, 250-333°C

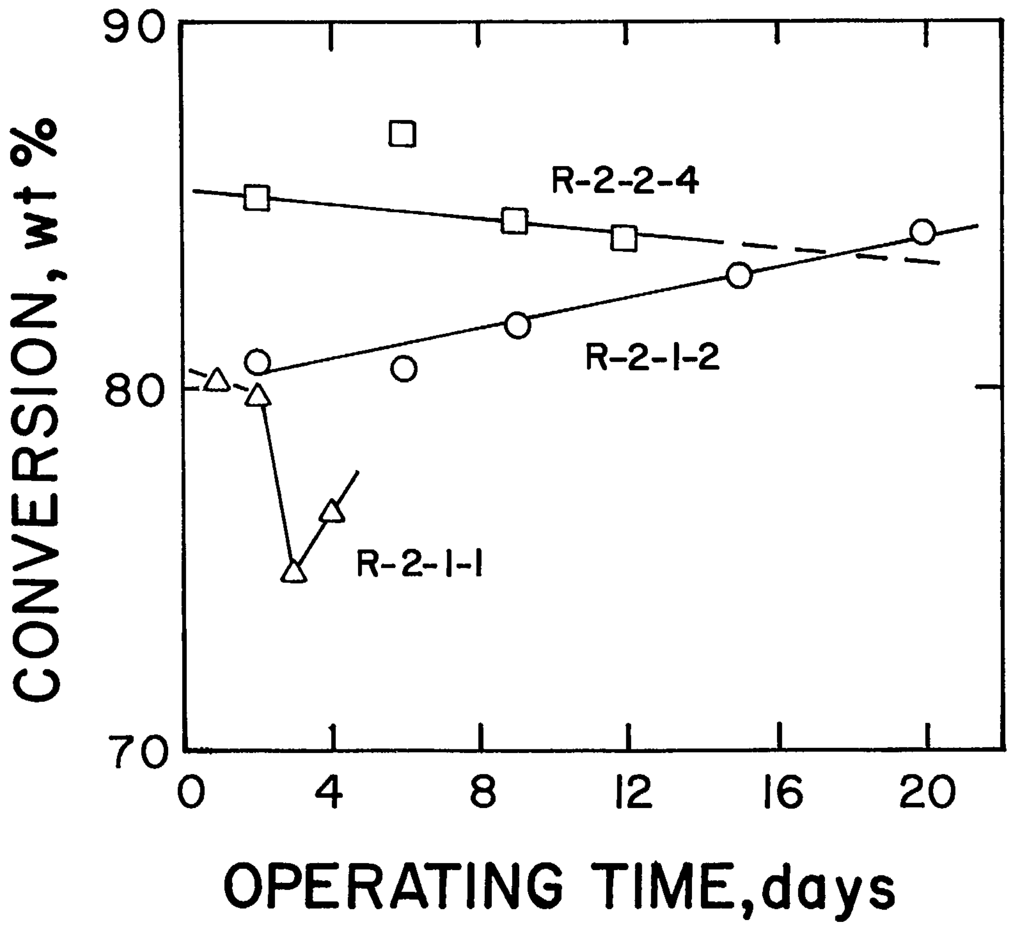

FIGURE 20 - Variation in Pitch Conversion

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