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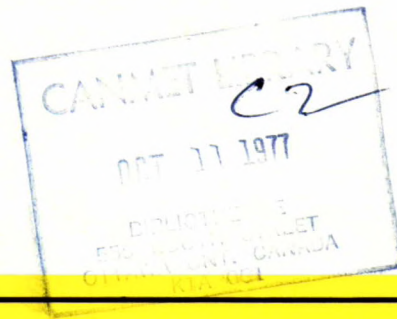
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**ANALYSIS OF REACTOR SAMPLES COLLECTED
DURING THERMAL HYDROCRACKING OF
ATHABASCA BITUMEN**

K. Belinko, B.N. Nandi and J.M. Denis

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THERMAL HYDROCRACKING OF ATHABASCA BITUMEN

by

K. Belinko*, B.N. Nandi* and J.M. Denis**

ABSTRACT

Samples from five reactor levels collected during thermal hydrocracking of Athabasca bitumen were analyzed for asphaltenes, benzene-insolubles and various other components. The data indicated that a conversion of asphaltenes to benzene-insoluble organic matter was occurring during the process. An accumulation of nickel and vanadium at the bottom of the reactor during the run, did not appear to have any significant effect on product quality.

Optical microscopic investigations revealed that the benzene-insoluble matter from all reactor levels consisted primarily of coke derived from the asphaltenes. There was no evidence of coke originating from the heavy aromatic oils.

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* Research Scientists, Coal Resource and Processing Laboratory and ** Manager, Synthetic Fuel Research Laboratory, Energy Research Laboratories, Canada Centre for Mineral and Energy Technology, Dept. of Energy, Mines and Resources, Ottawa, Canada.

ANALYSE D'ECHANTILLONS PRELEVES EN REACTEURS AU
COURS D L'HYDROCRAQUAGE THERMIQUE DES BITUMES DE L'ATHABASCA

par

K. Belinko*, B.N. Nandi* et J.M. Denis**

RESUME

Des échantillons prélevés en réacteur, en cinq différentes étapes, au cours de l'hydrocraquage thermique des bitumes de l'Athabasca, ont été analysés pour dépister les asphaltènes, les composants insolubles dans le benzène et autres composantes. Les données indiquent qu'une conversion des asphaltènes en matière organique insoluble dans le benzène se produit au cours du processus. L'accumulation de nickel et de vanadium au fond du réacteur, pendant le cycle, ne semble pas affecter outre mesure la qualité du produit.

Des études menées à l'aide d'un microscope optique ont révélé que la matière insoluble dans le benzène, extraite à toutes les étapes en réacteurs, se compose en grande partie de coke dérivé des asphaltènes. Rien n'indique que du coke peut provenir des hydrocarbures aromatiques.

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*Chercheur scientifique, Laboratoire de traitement et de l'évaluation des ressources en charbon, et **gestionnaire, Laboratoire de recherche sur les combustibles synthétiques, Laboratoires de recherche énergétique, Centre canadien de la technologie des minéraux et de l'énergie, ministère de l'Energie, des Mines et des Ressources, Ottawa, Canada.

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INTRODUCTION

Bitumen from the Alberta tar sands has recently emerged as a potential source of liquid fuel to supplement Canada's presently diminishing supplies of petroleum. An area of investigation which has received considerable attention at the Energy Research Laboratories has been the hydrocracking of bitumen. Although a great deal of emphasis has generally been placed on catalytic hydrogenation processes (1-3), attention has lately been focussed on non-catalytic hydrogenation or thermal hydrocracking (4,5).

Depending on the operating conditions of the reactor, it is possible to convert up to 95% of the +524°C pitch by thermal hydrocracking. By controlling the extent of pitch conversion, the +524°C residuum fraction can be utilized to supply energy requirements for the mining, separation and refining steps. This flexibility in pitch conversion offers a distinct advantage over other competing processes such as delayed coking, fluid coking and flexicoking.

A primary concern of this program has been to examine conditions which reduce reactor fouling. In a recent report, data were presented and correlated on pitch conversion, hydrogen consumption, gas formation and distillate yield (5). Comparisons were made between short pilot-plant runs, e.g., 4 hours, where there was no appreciable coke formation in the reactor, and long runs of three-weeks duration, where formation of coke in the reactor was evident. In some runs, the deposition of coke on the reactor wall was sufficient to reduce reactor volume and increase the space velocity; both of these factors contributed to a reduction in pitch and sulphur conversion. The effect of accumulation of mineral matter in catalyzing the cracking and hydrogenation processes and in reducing coke formation was also discussed.

In the present report, the distribution of mineral matter, and of coke and coke-precursors, i.e., benzene-insolubles, in the thermal hydrocracking reactor is examined. Data for samples collected along five levels of the reactor are presented. The runs considered were chosen to exemplify successful runs, during which little coke was detected on the reactor wall after three weeks of operation. These studies are complemented by the results of optical microscopic investigations of the benzene-insoluble matter present in the reactor during the thermal hydrocracking process.

EXPERIMENTAL

The operational details of the thermal hydrocracking pilot plant have been described in previous reports (4,5). The data presented in this report correspond to 21-day runs to be referred to as runs 84-1-1 and 84-2-1. Some properties of the bitumen feedstock used in these studies are given in Table 1. Experimental conditions for these runs are summarized in Table 2.

Samples for analysis were collected daily from the 4.5-litre reactor during run 84-1-1 and on an average of every four days during run 84-2-1. Collection of samples was accomplished with the use of 150-cc Kuentzel bombs equally spaced along five levels of the 3.95-m-long reactor.

Levels 1 to 5, counting from bottom to top of the reactor were located at the 0.15-m, 1.1-m, 2.0-m, 2.9-m and 3.8-m positions. The samples were collected by opening a valve between the Kuentzel bomb, kept at ambient pressure, and the reactor, and allowing the bomb to fill, Fig. 1. This valve was then shut, and the sample allowed to drain off the Kuentzel bomb by opening a valve at its bottom. The line between the Kuentzel bomb and the reactor was subsequently cleared by passing high pressure H_2 gas through the valve at the top of the bomb. In general, a reactor sample of approximately 100 gm was taken for each analysis.

The weight per cent of asphaltenes, benzene-insolubles and ash were determined by standard ASTM procedures (6). The data for asphaltenes and benzene-insolubles are reported on an ash-free basis. Ash analyses were carried out by X-ray fluorescence techniques. Samples for petrographic examinations were embedded in epoxy and polished by standard methods (7). Microscopic investigations were performed with a Leitz polarized-light microscope.

RESULTS

The experimental conditions for runs 84-1-1 and 84-2-1 are given in Table 2. The major difference between these two runs was in the operating temperature of the reactor: 460°C and 470°C, respectively. During the course of run 84-1-1, a total of 105 samples was removed from the reactor. In comparison, only 18 reactor samples were collected during run 84-2-1. Representative sample data collected for run 84-1-1 are tabulated in Table 3. Similar data were obtained for run 84-2-1 and will be presented whenever relevant.

To facilitate interpretation of results, level 1 will be considered separately from the rest of the reactor. The reason for this becomes apparent when examining the temperature conditions given in Table 2. Whereas the temperature at levels 2 to 5 remained constant throughout the entire 21-day period of operation, the temperature at level 1 varied by 30-40°C during the runs. This change in temperature at the first level was accounted for by the build-up of coke in the pre-heater.

(a) Results for Reactor Level 1

The results for level 1 of the reactor are somewhat more amenable to interpretation than for the remaining four levels since they reflect the early stages of the hydrocracking process, where extensive thermal cracking of the various components of the bitumen has not yet occurred. Consequently, trends in results are more easily identifiable.

The benzene-insoluble organic matter, BIOM, formed at the first level of the reactor appeared to originate from the asphaltenes. This is demonstrated in Fig. 2 and 3 which show an increase in concentration of BIOM as a function of operating time, and a commensurate decrease in asphaltene concentration. The sum of the concentrations of these two fractions remained approximately constant during the entire run.

The variation in ash concentration at the first level of the reactor as a function of operating time is shown in Fig. 4. These results indicated that there was an accumulation of inorganic matter in the reactor during the run. The effect of sampling rate is demonstrated in Fig. 4. The rate of accumulation was found to be lower during run 84-1-1 where daily samples were

collected than for run 84-2-1 where samples were removed on an average of only every four days. When sample withdrawal was carried out every second day during run 84-2-1, subsequent to the 17th day of operation, a net decrease in ash content was observed at level 1 of the reactor, Fig. 4.

There was an appreciable increase in the concentration of nickel and vanadium at the bottom level of the reactor, Table 4. An ash analysis for a sample collected at level 1 of the reactor on the 15th day of operation during run 84-1-1 is shown in Table 5 and compared to an analysis of the ash in the feedstock.

Figures 5 and 6 are optical micrographs of the benzene-insoluble matter present in samples at level 1 collected on the 3rd and 16th day of run 84-1-1, respectively. The micrographs revealed the presence of agglomerated particles, generally containing a fragment of coal as a nucleus. Areas on the periphery of the agglomerated mass appeared to be undergoing transformation to coke, and were found to be anisotropic. It was also evident that certain aggregates had transformed completely into coke. The structure of the coke formed was found to be similar to that of the coke derived from asphaltenes (8). There was no evidence of coke formed from the rest of the bitumen. In general, the number and size of the agglomerated particles were significantly greater for reactor samples taken on the 16th day than for those taken on the 3rd day.

(b) Results for Reactor Levels 2 to 5

The results for reactor samples at levels 2 to 5 collected during run 84-1-1 are given in Table 3. Certain trends are readily apparent from the data. For instance, the concentration of BIOM increased and the ash content decreased in going from level 2 to 5 of the reactor during any one day of the run.

The concentration of BIOM was generally observed to decrease at every level of the reactor (levels 2 to 5) during the run, Fig. 7. In contrast, there was no consistent variation in the concentration of asphaltenes at these levels of the reactor during the run, Table 3.

Ash analyses of samples collected at the higher levels of the

reactor were generally found to be similar to those of the feedstock, Table 5. The concentrations of nickel and vanadium were much lower than those observed at the bottom level of the reactor, Table 4.

Optical micrographs of the benzene-insoluble material from reactor samples at levels 2 to 5 showed little evidence of the agglomerated particles detected at the first level. Representative micrographs of samples taken on the 3rd and 16th days of operation for run 84-1-1 are shown in Fig. 8 and 9, respectively. The coke structure shown in these micrographs was again found to correspond to coke derived from asphaltenes (8).

(c) Product Analysis

Analysis of the heavy-oil product collected from the bottom of the hot separator was conducted on a daily basis during runs 84-1-1 and 84-2-1. Representative data for both these runs are given in Table 6. The asphaltene, benzene-insolubles and ash contents were found to vary only slightly during these runs.

Variations in pitch and sulphur conversion with operating time are given in Table 7 for runs 84-1-1 and 84-2-1. In spite of the accumulation of mineral matter in the reactor during the runs, there appeared to be little improvement in product quality as a function of operating time.

DISCUSSION

Interpretation of the results presented in the previous section is complicated by several factors. The variation in the extent of vaporization of the cracked products, at each of the reactor levels, is perhaps the most serious of these factors. In general, the samples collected at the top levels of the reactor have a greater apparent concentration of asphaltenes, benzene-insolubles and ash than those collected at the bottom levels. Consequently, comparison of the data from different levels of the reactor is subject to some degree of ambiguity. Nevertheless, it is possible to assume that the extent of vaporization at each of the levels will not vary signifi-

cantly during the run. This will not be entirely true for level 1 because of the variation in temperature at this particular level of the reactor, Table 2.

The analytical data collected for level 1 of the reactor, Fig. 2 and 3, suggest that a conversion of asphaltenes to benzene-insoluble organic matter is occurring during thermal hydrocracking of Athabasca bitumen. The total concentration of asphaltenes and BIOM at the bottom of the reactor was found to approximate the concentration of asphaltenes present in the feed, corrected for vaporization of liquid products at this first level of the reactor, Fig. 11. The microscopic observation that the spherical agglomerated particles of BIOM increased in size and number during the run is consistent with the analytical results, Fig. 5 and 6.

The gradual increase in concentration of BIOM at the bottom of the reactor as a function of operating time is somewhat difficult to interpret, Fig. 2 and 3. If this effect were simply the result of an accumulation of BIOM in the reactor during the run, the rate of increase in concentration of BIOM would have been much greater than the rate of decrease in asphaltene concentration. However, this is inconsistent with the analytical results. An alternative explanation could be that the increase in concentration of BIOM is partly related to the temperature effect at the pre-heater outlet and at the bottom level of the reactor, Table 2. The decrease in temperature, along with thermal hydrocracking of some of the constituents of the bitumen which help keep the asphaltenes soluble, may have caused the latter to precipitate out of the bitumen. The precipitated asphaltenes would then have deposited on some nucleation site, e.g., on a fragment of coal, thus explaining the presence of agglomerated spheres at the bottom level of the reactor. The precipitated asphaltenes would probably undergo polymerization reactions, and the polymer products would be benzene-insoluble. Dehydrogenation of these asphaltene polymers or coke-precursors would ultimately result in coking, and this is observed in some of the agglomerated particles in Figure 6.

The absence of agglomerated spheres at the higher levels of the reactor may again be interpreted in terms of a temperature effect. The higher temperature at these levels, Table 2, would have caused extensive thermal hydrocracking of the precipitated asphaltenes. The generation of volatile matter by this process probably resulted in the disintegration of

agglomerated particles. Any coke formed would remain in a dispersed form as observed in micrographs of samples collected at levels 2 to 5, Fig. 8 and 9.

The optical micrographs shown in Figures 5 and 6 revealed that a small fragment of carbonaceous material, generally coal, acted as a nucleus for the precipitating asphaltenes. Microscopic examination of the benzene-insoluble residue in the feedstock, Fig. 10, showed that particles of coal were inherently present in the bitumen. A similar effect was recently reported during hydrogenation of bituminous coals (9).

The decrease in concentration of BIOM at reactor levels 2 to 5 as a function of operating time, Fig. 7, may be a reflection on the role of mineral matter in inhibiting coke formation (10,11). The data for ash content in Table 3 show that there is accumulation of mineral matter in the reactor during the run. It is possible that this mineral matter is active in catalyzing hydrogenation reactions, thereby minimizing the need for hydrogen donors which invariably turn out to be the coke precursors. However, this mineral matter did not appear to be active catalytically in cracking reactions and sulphur removal. Pitch and sulphur conversion remained approximately constant during both runs 84-1-1 and 84-2-1, Table 6.

The high concentration of nickel and vanadium at the bottom level of the reactor, relative to the other inorganic constituents, Table 5, suggests that these metals may have originated from organo-metallic complexes. It is not clear in what form these metals are present in the reactor, but their presence may be a contributing factor to the increasing tendency for the BIOM to form at the bottom of the reactor as a function of residence time.

The variation in concentration of BIOM at levels 2 to 5, Fig. 7, may be largely due to differences in the extent of vaporization at these levels. The amount of vaporized product in the reactor will vary with residence time, and consequently, samples at the higher levels of the reactor will show a greater apparent concentration of BIOM. This can be demonstrated, for instance, by comparing data corresponding to the heavy-oil product, Table 3, with those of samples collected at the very top of the reactor, level 5. Differences in these concentrations arise primarily from the presence of vaporized product at level 5 of the reactor, and therefore approximately 75%

of the product must exist in the vapour state at level 5. If a similar correction is made for the concentration of BIOM at levels 2 to 5 in Figure 7, a vaporization profile along the length of the reactor can be plotted, Fig. 11. The value corresponding to level 5 of the reactor was confirmed by computer predictions using pseudo components based on a distillation of the products (12).

If a correction is made for the asphaltene concentration in the reactor based on the vaporization profile shown in Figure 11, a plot of asphaltene concentration versus reactor level can be drawn, Fig. 12. This plot then shows the extent of thermal hydrocracking of the asphaltenes in the reactor.

Finally, some comments should be made regarding the coke structure observed in the benzene-insoluble material present in reactor samples. Generally, for runs carried out under similar conditions but at lower pressures than in this particular case, e.g., 13.4 MPa, there was evidence of coke originating from both the asphaltenes and the heavy aromatic oils (8). In this run, the only coke observed appeared to originate from the asphaltenes, with no evidence of coke from the heavy aromatic oils of the bitumen. Furthermore, the total amount of coke formed in this run was negligible compared with runs carried out at lower hydrogen pressures. It appears, therefore, that hydrogen pressure is a very important factor in preventing coke formation and that the formation of coke from the heavy aromatic oils of the bitumen may be inhibited at high hydrogen pressures.

CONCLUSIONS

The analytical results and microscopic investigations indicate that, during thermal hydrocracking of bitumen, asphaltenes are converted to benzene-insoluble matter, possibly by a series of steps involving precipitation and polymerization or condensation reactions. This benzene-insoluble matter constitutes a coke-precursor which can ultimately transform to coke by undergoing further condensation and dehydrogenation reactions.

The absence of coke originating from the heavy aromatic oils of the bitumen, during the runs described in this report, is presumed to be due to

the high hydrogen pressure in the system. The availability of molecular hydrogen precluded the need for the heavy oils to act as hydrogen-donors, and consequently, dehydrogenation of these oils was not extensive.

There appears to be a decreasing tendency for formation of benzene-insoluble organic matter in the reactor as a function of operating time. This result could be related to the accumulation of mineral matter in the reactor during the run, suggesting that this mineral matter may have some catalytic activity in hydrogenation reactions. The pronounced accumulation of nickel and vanadium at the bottom of the reactor is presumed to be due to the decomposition of porphyrin molecules during thermal hydrocracking. Although it is not clear by what mechanism these metals are settling to the bottom of the reactor, it appears that they have no catalytic activity in either pitch conversion or sulphur removal.

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TABLE 1

Properties of Athabasca Bitumen

| | | |
|--------------------------|---------------------------|-------|
| Specific gravity | 60/60 ^o F | 1.009 |
| Sulphur | wt % | 4.48 |
| Ash | wt % | 0.59 |
| Conradson carbon residue | wt % | 13.3 |
| Pentane-insolubles | wt % | 15.5 |
| Benzene-insolubles | wt % | 0.72 |
| Vanadium content | ppm | 213 |
| Nickel content | ppm | 67 |
| Total acid number | | 2.77 |
| Total base number | | 1.89 |
| Carbon | wt % | 83.36 |
| Hydrogen | wt % | 10.52 |
| Nitrogen | wt % | 0.43 |
| Viscosity | cst at 100 ^o C | 133.3 |

TABLE 2

Thermal Hydrocracking Conditions

| | | <u>Run 84-1-1</u> | <u>Run 84-2-1</u> |
|--|---------------------------|-------------------|-------------------|
| Pressure | MPa | 24.23 | 24.23 |
| LHSV | | 2.0 | 2.0 |
| Recycle gas flow | ℓ/h at 24.23 MPa, 25°C | 9147 | 9147 |
| H ₂ purity | % | 85 | 85 |
| Temperatures | °C | | |
| Reactor (2nd and 3rd subsequent levels) | | 460 | 470 |
| Reactor (1st level) | | | |
| Day 1 | | 440 | 450 |
| Day 21 | | 414 | 410 |
| Preheater outlet | | | |
| Day 1 | | 415 | 430 |
| Day 21 | | 360 | 345 |

TABLE 3

Distribution of Asphaltenes, Benzene-insolubles
and Ash in the Reactor During Thermal Hydrocracking of Athabasca Bitumen
Run 84-1-1

| Sample | | Asphaltenes % | Benzene - insolubles % | Ash % | Sample | | Asphaltenes % | Benzene - insolubles % | Ash % |
|--------|-------|------------------|------------------------------|----------|--------|-------|------------------|------------------------------|----------|
| Day | Level | | | | Day | Level | | | |
| 1 | 1 | 18.00 | 1.41 | 3.59 | 13 | 1 | 16.42 | 2.26 | 8.42 |
| | 2 | 18.49 | 2.91 | 4.10 | | 2 | 20.82 | 2.35 | 3.93 |
| | 3 | 19.02 | 4.08 | 4.10 | | 3 | 18.45 | 3.60 | 3.55 |
| | 4 | 20.80 | 4.79 | 3.71 | | 4 | 20.40 | 4.70 | 3.70 |
| | 5 | 17.74 | 4.68 | 3.48 | | 5 | 17.49 | 4.56 | 2.55 |
| 3 | 1 | 19.19 | 1.41 | 4.30 | 16 | 1 | 15.22 | 3.83 | 10.85 |
| | 2 | 20.48 | 2.81 | 4.11 | | 2 | 19.34 | 2.34 | 4.82 |
| | 3 | 19.40 | 4.17 | 3.73 | | 3 | 18.16 | 3.01 | 4.43 |
| | 4 | 22.73 | 5.08 | 3.79 | | 4 | 19.92 | 3.42 | 4.46 |
| | 5 | 15.93 | 5.61 | 3.96 | | 5 | 18.74 | 4.31 | 2.85 |
| 7 | 1 | 17.49 | 2.38 | 7.83 | 18 | 1 | 14.43 | 4.15 | 13.02 |
| | 2 | 20.72 | 3.06 | 4.12 | | 2 | 18.34 | 1.73 | 5.53 |
| | 3 | 20.90 | 4.29 | 3.61 | | 3 | 15.99 | 2.99 | 4.92 |
| | 4 | 21.64 | 5.34 | 3.42 | | 4 | 15.81 | 3.25 | 3.14 |
| | 5 | 18.37 | 5.32 | 3.01 | | 5 | 17.00 | 3.66 | 2.64 |
| 10 | 1 | 16.80 | 2.11 | 8.19 | 21 | 1 | 13.60 | 5.52 | 14.01 |
| | 2 | 18.73 | 2.36 | 3.71 | | 2 | 14.76 | 2.28 | 7.36 |
| | 3 | 21.41 | 3.70 | 3.59 | | 3 | 15.60 | 2.46 | 5.94 |
| | 4 | 21.74 | 4.64 | 3.61 | | 4 | 15.77 | 3.13 | 5.40 |
| | 5 | 20.44 | 4.73 | 4.43 | | 5 | 16.74 | 3.68 | 2.98 |

TABLE 4

Distribution of Ni, V and S in the
Reactor During Run 84-1-1

| Sample | | Nickel ppm by weight | Vanadium ppm by weight | Sulphur % |
|--------|-------|-------------------------|---------------------------|--------------|
| Day | Level | | | |
| 1 | 1 | 363 | 1131 | 4.42 |
| | 3 | 394 | 1197 | 4.42 |
| | 5 | 336 | 1031 | 3.69 |
| 7 | 1 | 5620 | 10,700 | 6.17 |
| | 3 | 355 | 1088 | 4.31 |
| | 5 | 312 | 917 | 3.86 |
| 13 | 1 | 7471 | 14,500 | 6.65 |
| | 3 | 353 | 1059 | 4.42 |
| | 5 | 243 | 742 | 3.74 |
| 21 | 1 | 17,700 | 33,200 | 9.82 |
| | 3 | 1449 | 4165 | 4.53 |
| | 5 | 210 | 617 | 3.68 |

TABLE 5

Ash Analysis of Feedstock and of Samples
Collected from the Reactor During Run 84-1-1

| Constituent | Athabasca bitumen feedstock % | Day 15, Level 1 Run 84-1-1 % | Day 15, Level 3 Run 84-1-1 % |
|--------------------------------|-------------------------------------|------------------------------------|------------------------------------|
| SiO ₂ | 41.88 | 12.4 | 40.07 |
| Al ₂ O ₃ | 23.06 | 9.0 | 27.76 |
| Fe ₂ O ₃ | 7.74 | 4.9 | 7.07 |
| Mn ₃ O ₄ | 0.19 | 0.07 | 0.14 |
| TiO ₂ | 3.35 | 3.03 | 4.69 |
| P ₂ O ₅ | 0.45 | 0.51 | 0.44 |
| CaO | 3.81 | 1.6 | 1.64 |
| MgO | 0.88 | 2.5 | 0.39 |
| V ₂ O ₅ | 5.7* | 35.9* | 3.1* |
| NiO | 1.4* | 17.5* | 0.75* |
| K ₂ O | 1.82 | 0.68 | 1.71 |
| Na ₂ O | 0.74 | - | - |
| SO ₃ | 3.57 | - | - |

* By X-ray analysis

TABLE 6

Sample Analysis of the Heavy-oil Product
from Runs 84-1-1 and 84-2-1

| Run 84-1-1 | | | | Run 84-2-1 | | | |
|---|--------------|----------------------|-------|---|--------------|----------------------|-------|
| Sample day | Asphaltene % | Benzene-insolubles % | Ash % | Sample day | Asphaltene % | Benzene-insolubles % | Ash % |
| 1 | 7.04 | 1.97 | 0.96 | 1 | 5.48 | 1.80 | 1.01 |
| 3 | 7.12 | 2.26 | 0.98 | 3 | 6.11 | 1.99 | 1.20 |
| 5 | 7.17 | 2.40 | 1.05 | 5 | 5.59 | 2.01 | 1.12 |
| 7 | 7.70 | 2.36 | 0.96 | 7 | 5.67 | 1.94 | 1.26 |
| 9 | 7.71 | 2.36 | 0.98 | 9 | 6.18 | 2.15 | 1.21 |
| 11 | 7.13 | 2.21 | 0.95 | 11 | 6.17 | 1.29 | 0.59 |
| 13 | 7.40 | 2.26 | 0.99 | 13 | 5.32 | 1.96 | 1.10 |
| 15 | 6.50 | 2.10 | 1.08 | 15 | 5.16 | 2.35 | 1.20 |
| 17 | 7.15 | 1.69 | 0.91 | 17 | 5.37 | 1.99 | 1.18 |
| 19 | 6.50 | 1.52 | 0.85 | 19 | 5.72 | 2.38 | 1.29 |
| 21 | 6.73 | 1.95 | 0.77 | 21 | 5.68 | 2.71 | 1.05 |
| Average over total sample (light oil + heavy oil) | 4.6 | 1.4 | 0.62 | Average over total sample (light oil + heavy oil) | 3.04 | 1.1 | 0.59 |

TABLE 7

Pitch and Sulphur Conversion
During Runs 84-1-1 and 84-2-1*

| Day | Run 84-1-1 | | Run 84-2-1 | |
|-----|------------------|--------------------|------------------|--------------------|
| | Pitch conv. % | Sulphur conv. % | Pitch conv. % | Sulphur conv. % |
| 2 | 81.9 | 50 | 88.7 | 57 |
| 7 | 81.2 | 47 | 89.1 | 58 |
| 13 | 81.0 | 48 | 89.5 | 58 |
| 17 | 83.7 | 51 | 89.1 | 60 |
| 22 | 81.9 | 48 | 88.3 | 59 |

* Reference 5

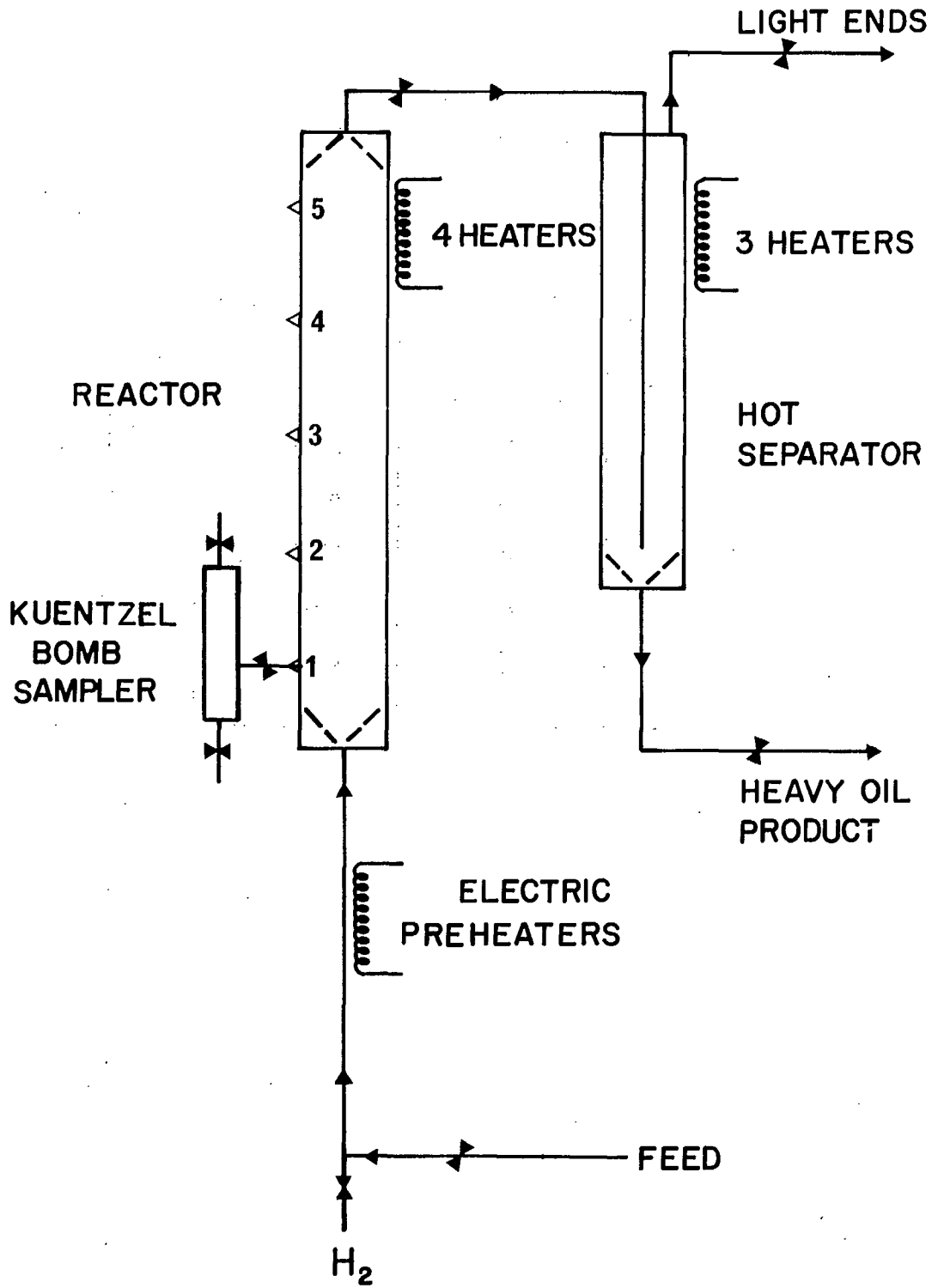


FIGURE 1 - Schematic Diagram of Experimental Apparatus

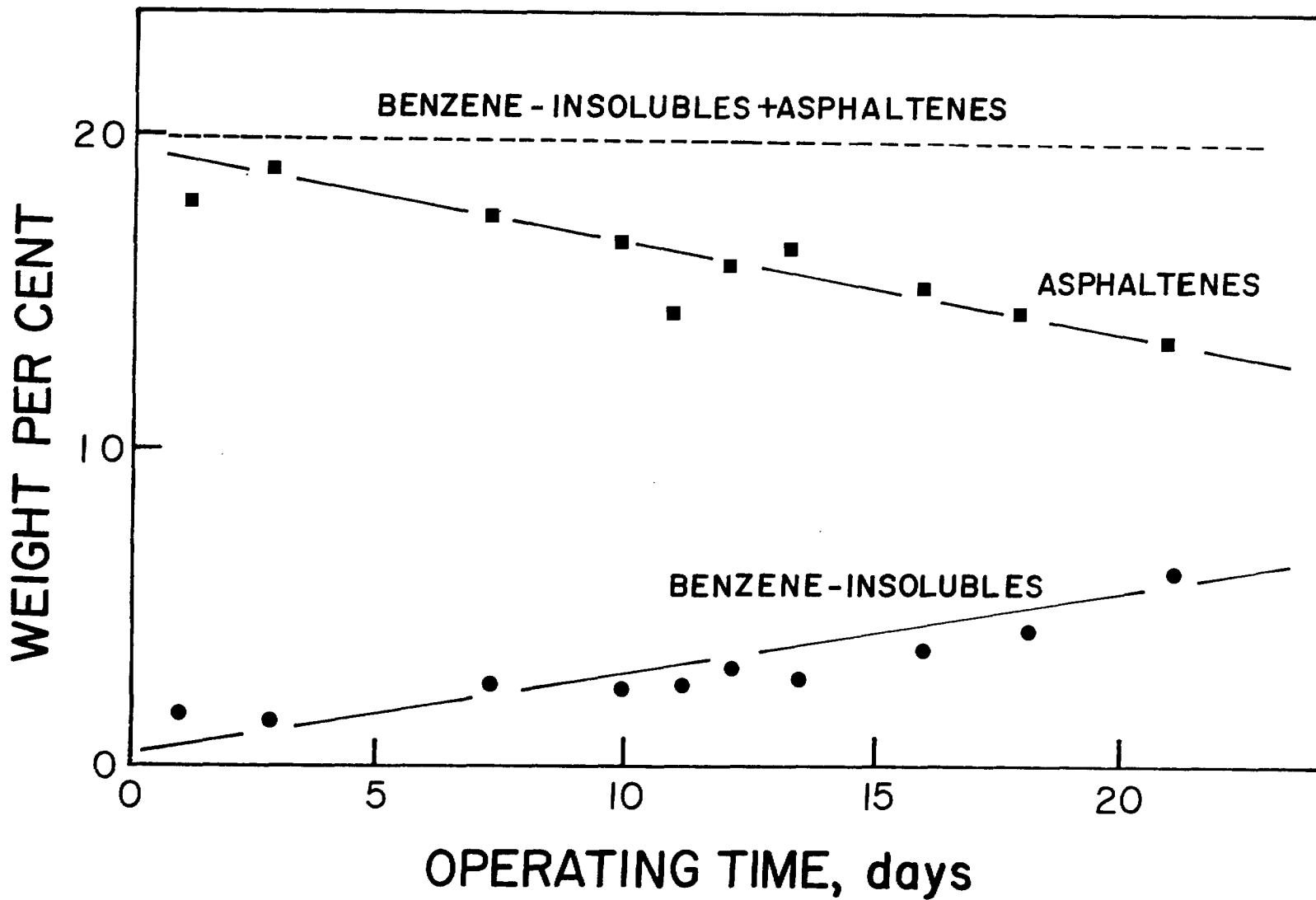


FIGURE 2 - Concentration of Asphaltenes and Benzene-insolubles at the Bottom Level of the Reactor as a Function of Operating Time During Run 84-1-1

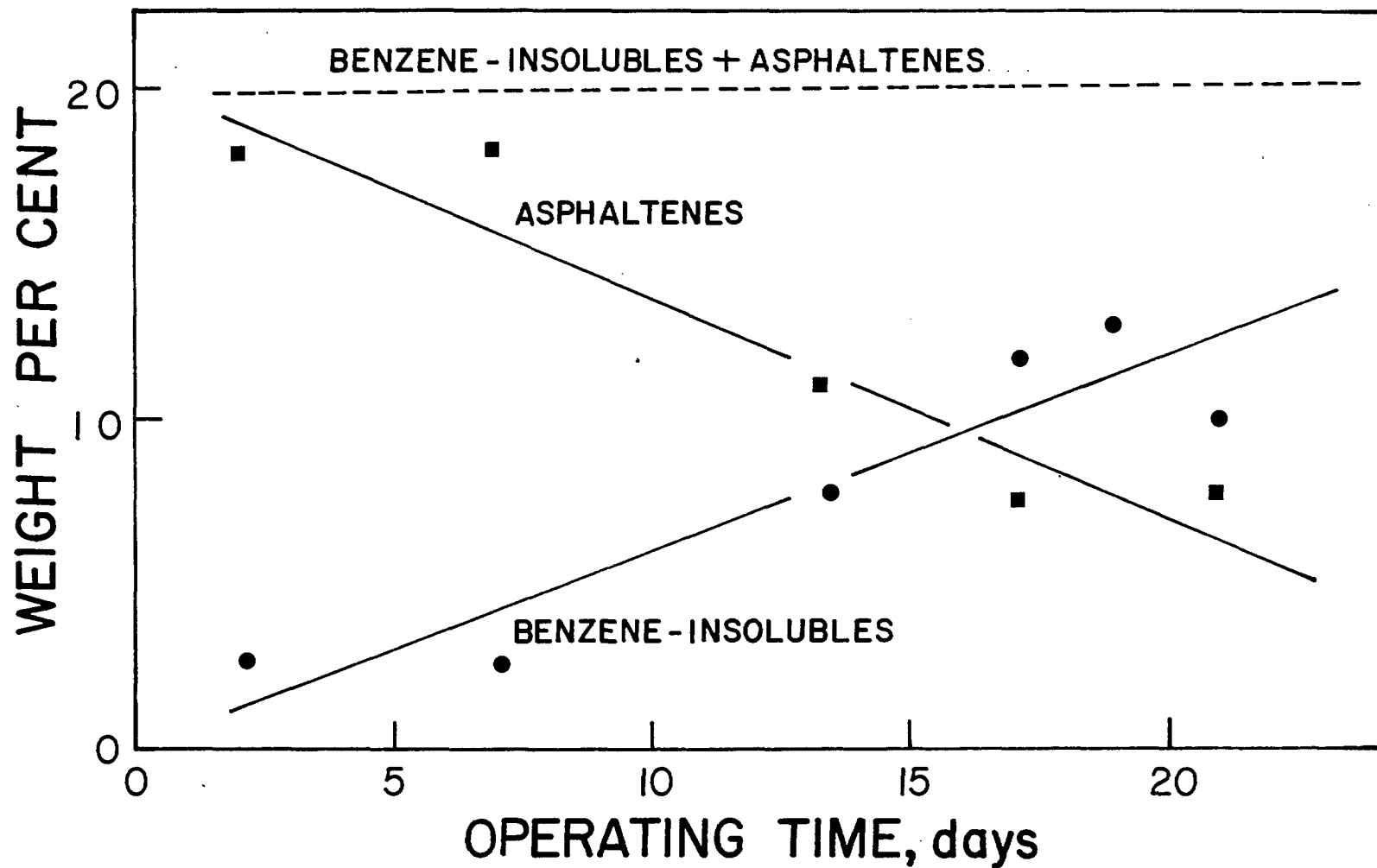


FIGURE 3 - Concentration of Asphaltenes and Benzene-insolubles at the Bottom Level of the Reactor as a Function of Operating Time During Run 84-2-1

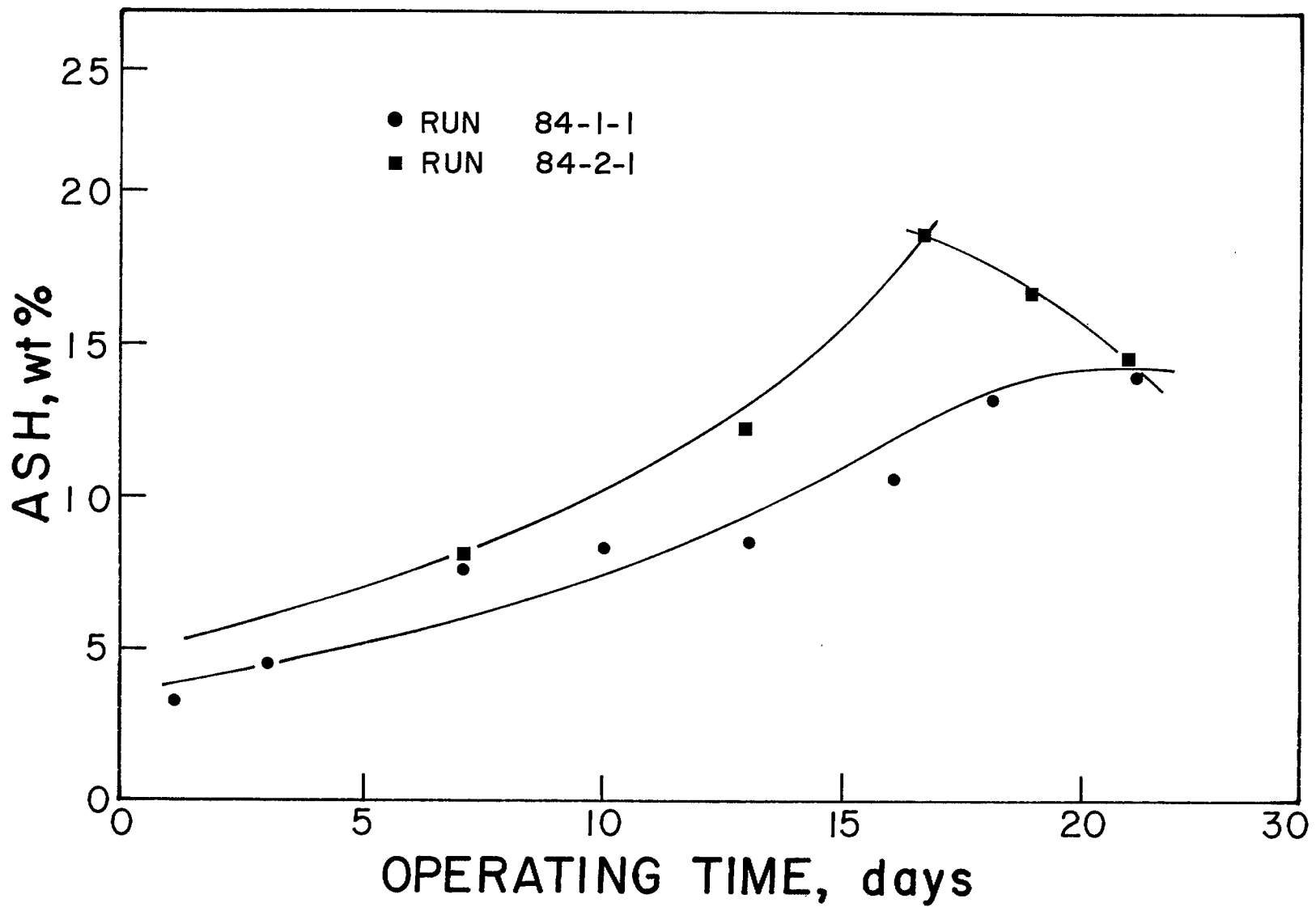


FIGURE 4 - Concentration of Ash at the Bottom of the Reactor as a Function of Operating Time

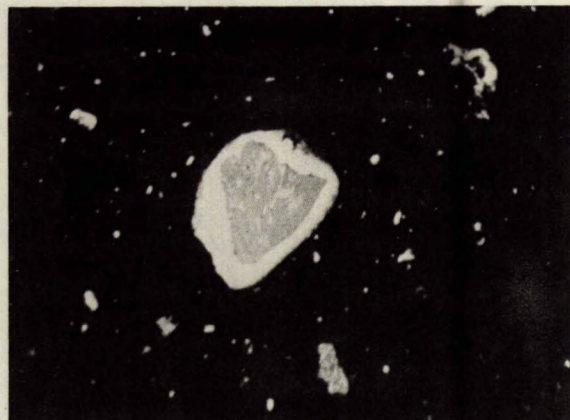
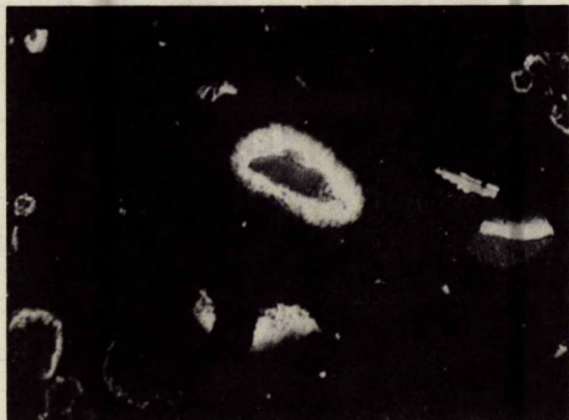


FIGURE 5 - Benzene-insoluble Residue Collected from the Bottom Level of the Reactor on the 3rd Day of Run 84-1-1
(x 600)

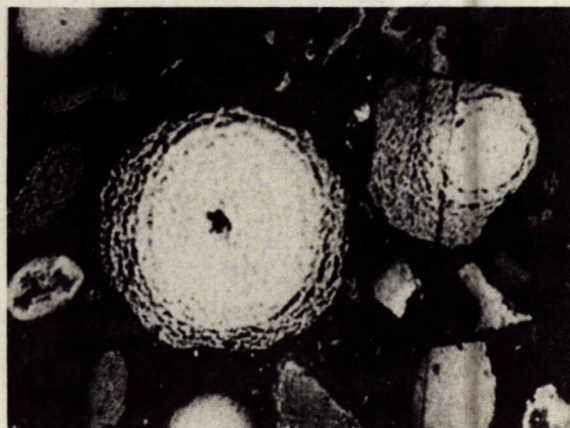
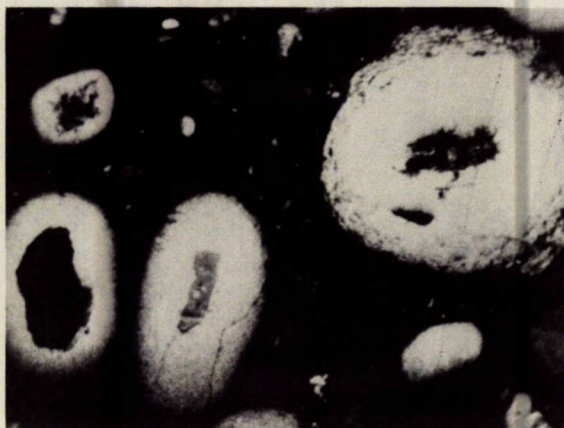


FIGURE 6 - Benzene-insoluble Residue Collected from the Bottom Level of the Reactor on the 16th Day of Run 84-1-1
(x 600)

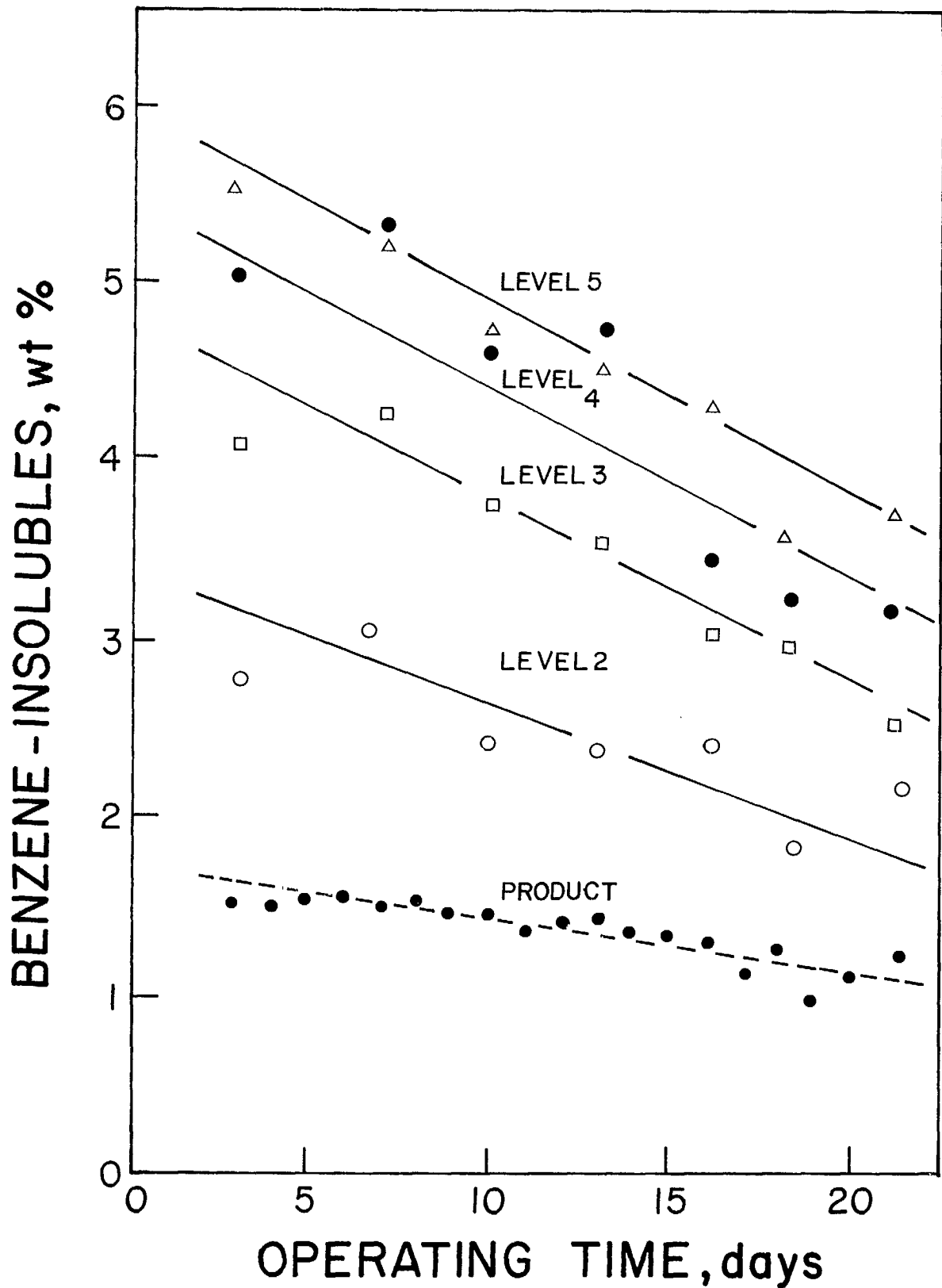


FIGURE 7 - Concentration of Benzene-insolubles versus Operating Time for Reactor Levels 2 to 5 and Product; Run 84-1-1



FIGURE 8 - Benzene-insoluble Residue
Collected from Level 5 of
the Reactor on the 3rd
Day of Run 84-1-1 (x 600)

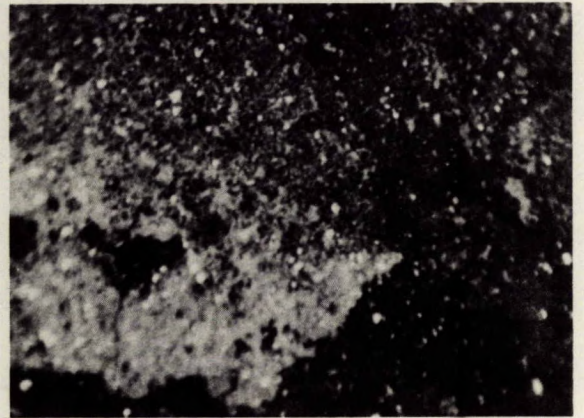


FIGURE 9 - Benzene-insoluble Residue
Collected from Level 5 of
the Reactor on the 16th
day of Run 84-1-1 (x 600)



FIGURE 10 - Benzene-insoluble Residue from the Feedstock
Showing Fragments of Coal Inherently Present
in the Bitumen (x 600)
M - micrinite, V - vitrinite, F - fusinite

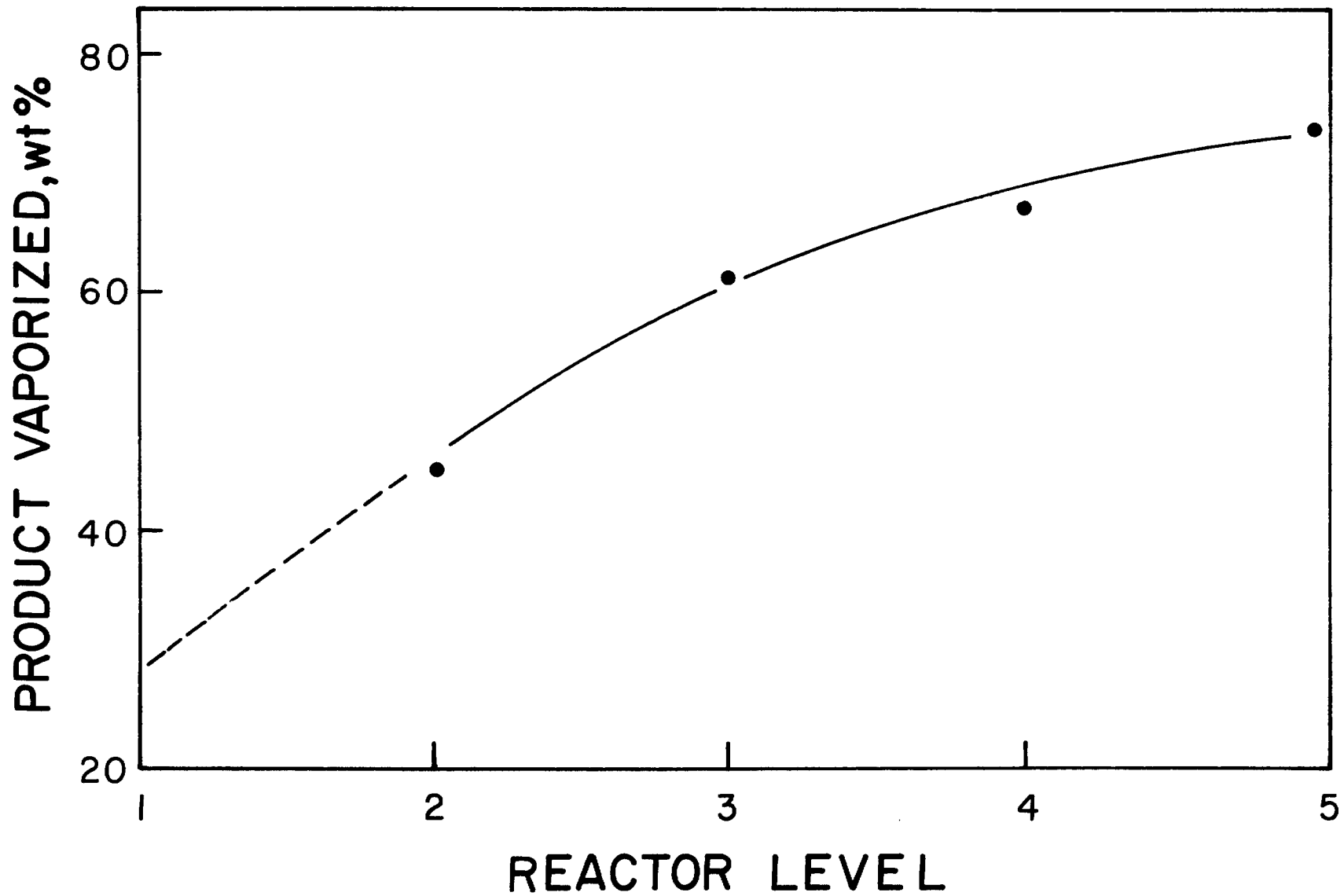


FIGURE 11 - Reactor Profile Showing the Extent of Product Vaporization; Run 84-1-1

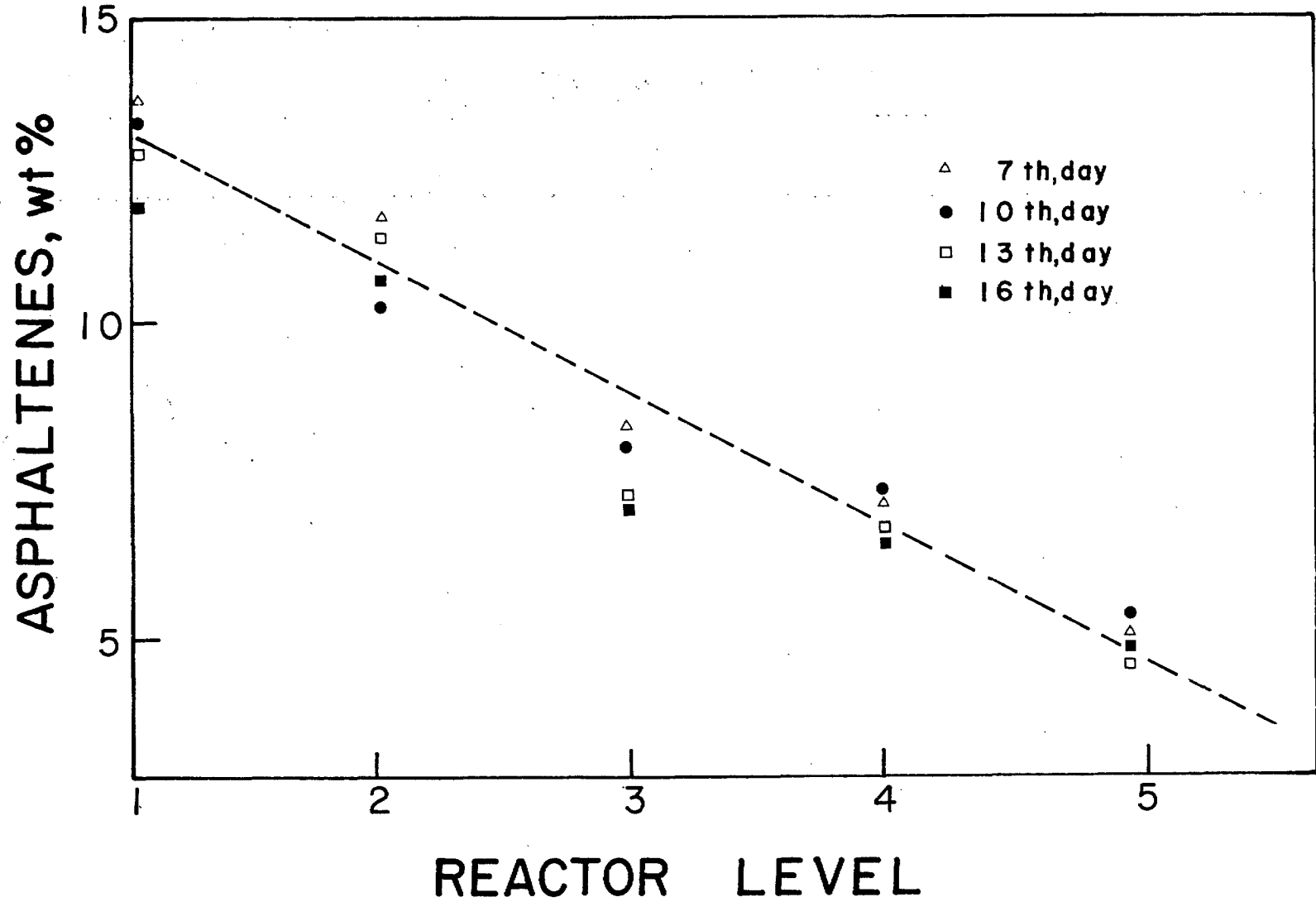


FIGURE 12 - Concentration of Asphaltenes Corrected for Product Vaporization as a Function of Residence Time in the Reactor; Run 84-1-1

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