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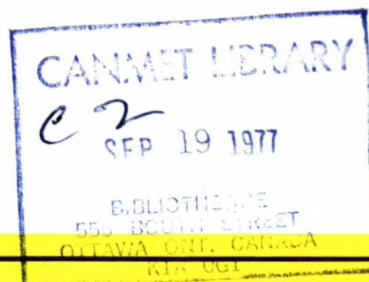
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# THERMAL HYDROCRACKING OF ATHABASCA BITUMEN: EFFECT OF RECYCLE-GAS PURITY ON PRODUCT YIELDS AND QUALITIES

A.M. Shah, B.B. Pruden and J.M. Denis

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THERMAL HYDROCRACKING OF ATHABASCA BITUMEN:  
EFFECT OF RECYCLE-GAS PURITY ON PRODUCT YIELDS AND QUALITIES

by

A.M. Shah\*, B.B. Pruden\*\* and J.M. Denis\*\*\*

ABSTRACT

The thermal hydrocracking of Athabasca bitumen at 13.89 MPa and 450°C has been investigated employing a liquid hourly space velocity of 1 and a recycle-gas rate of 0.0425 m<sup>3</sup>/h at the system pressure and 20°C. The experiments were conducted in a 4.5-l reactor with the recycle-gas purity in the range of 60-96 vol % H<sub>2</sub>. The hydrogen partial pressure in the reactor was in the range of 7-12 MPa.

Liquid products with consistent quality were obtained regardless of the recycle-gas purity. An increased hydrocarbon-gas made with increased recycle-gas purity was obtained at the expense of liquid product yield. The extent of conversion of +525°C pitch to distillable hydrocarbons decreased slightly with increased recycle-gas purity. The sulphur conversion and the hydrogen consumption were constant at 50 wt % and 168.5 m<sup>3</sup>/t respectively, independent of the recycle-gas purity. Data are compared with previously published results.

No operational problems were encountered when running with low recycle-gas purity with increased hydrogen sulphide and hydrocarbon gas contents. No carbonaceous deposits were formed in the reactor, hot separator or preheaters.

The most significant finding of the present investigation is that industrial hydrogen streams with 60-85 vol % H<sub>2</sub> could be used without purification in the preliminary refining of Athabasca bitumen with a reduction in process costs.

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HYDROCRAQUAGE THERMIQUE DE BITUME DE L'ATHABASCA: L'EFFET  
DE LA PURETE DU RECYCLAGE DU GAZ SUR LA PRODUCTION ET LA QUALITE

par

A.M. Shah\*, B.B. Pruden\*\* et J.M. Denis\*\*\*

SOMMAIRE

Les auteurs ont étudié le procédé de l'hydrocraquage thermique de bitume de l'Athabasca sous une pression de 13.89 MPa, à une température de 450°C, avec une vélocité spatiale liquide horaire de 1 et avec un taux de recyclage du gaz de 0.0425 m<sup>3</sup>/heure à la même pression que le système et à 20°C. Les essais ont été entrepris dans un réacteur de 4.5 litres avec un taux de pureté du recyclage du gaz variant entre 60 et 96% d'H<sub>2</sub> par vol. La pression partielle d'hydrogène dans le réacteur s'échelonnait entre 7 et 12 MPa.

De ces essais, on obtint des liquides d'une qualité conforme et ce, indifféremment de la pureté du recyclage du gaz. Une plus grande pureté de recyclage du gaz produisit plus de gaz d'hydrocarbures, au détriment du produit liquide. Le degré de conversion du brai à + 525°C en hydrocarbures distillables a diminué légèrement après une augmentation de la pureté du recyclage du gaz. La conversion du soufre et la consommation d'hydrogène étaient constantes à 50%, en poids, et 168.5 m<sup>3</sup>/t respectivement, indépendamment de la pureté du recyclage du gaz. On compare les présentes données avec les résultats qui ont déjà été publiés.

Aucun problème de fonctionnement n'a été envisagé lorsque la pureté du recyclage du gaz était basse, que l'hydrogène sulfuré était élevé et qu'il y avait du gaz d'hydrocarbures. Aucun dépôt de carbone ne s'est formé dans le réacteur, dans le séparateur à haute température et dans les préchauffeurs.

La découverte la plus significative de la présente étude est que les écoulements d'hydrogène industriel avec 60-85% d'H<sub>2</sub> en vol. peuvent être utilisés sans purification dans le raffinage préliminaire de bitume de l'Athabasca avec une réduction des coûts de traitement.

Droits de la Couronne réservés

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## INTRODUCTION

The refining of Canada's low grade petroleum resources to clean liquid fuels is complicated by the presence of unwanted minerals, metals and sulphur. In addition, the heavy oils or bitumens are hydrogen poor so that they must be coked to remove carbon, or hydrogenated and cracked to produce distillable liquid fuels. In keeping with a policy of maximum utilization of non-renewable resources, CANMET (Canada Centre for Mineral and Energy Technology) has supported research on hydrogenation and cracking (hydrocracking). Earlier work was focussed on catalytic hydrocracking (1-7) but more recently a thermal hydrocracking process has been developed (8).

Thermal hydrocracking offers an excellent alternative method to the coking processes (delayed coking or flexicoking) in the first-stage refining of Athabasca bitumen or heavy oil to distillable hydrocarbons. The yield of distillate oil is 10-15% higher; the amount of pitch (fraction boiling above 525°C) can be varied to suit energy requirements and the pitch can be easily gasified. Earlier work on thermal hydrocracking of Athabasca bitumen established the relationships between the process variables such as temperature, pressure, liquid feed rate, recycle-gas rate and the measured quantities such as pitch conversion, product yields, desulphurization, hydrocarbon-gas make and hydrogen consumption (8,9). This work was carried out with a recycle-gas purity of 85 vol % H<sub>2</sub>. However, no attempt was made in the past to study the reaction with different recycle-gas purities and hence at different hydrogen partial pressures without changing the system pressure.

In the petroleum industry, hydrogen streams are available from different units such as hydrocrackers and catalytic reformers and have purities in the range of 60 to 85 vol % H<sub>2</sub>. Communications with personnel from Great Canadian Oil Sands Ltd. and Syncrude Canada Ltd. indicated a great interest in research results on the thermal hydrocracking of Athabasca bitumen using recycle-gas of low purity. If hydrogen streams having purities in the range of 60 - 85 vol % H<sub>2</sub> could be utilized without purification for upgrading of Athabasca bitumen, the cost of the hydrocracking process could be reduced thereby making the process economically attractive.

The objectives of the present work were to investigate the effect of the recycle-gas purity on product yields and qualities during the thermal

hydrocracking of Athabasca bitumen and to collect a bank of data at different recycle-gas purities to better advise industry personnel. The recycle-gas purity was controlled by regulating the scrubber-oil rate in a packed scrubber at system pressure.

This report is mainly concerned with the effect of recycle-gas purity on product yields and qualities during the thermal hydrocracking of Athabasca bitumen at 13.89 MPa, 450°C, a liquid hourly space velocity (LHSV) of 1 and a recycle-gas rate of 0.0425 m<sup>3</sup>/h at the system pressure and 20°C.

#### LITERATURE SURVEY

Swift et al. (10) while investigating catalytic dehydrogenation of butene found that the introduction of hydrogen sulphide with feed eliminated the formation of carbonaceous deposits on the catalyst. Goudriaan et al. (11) studied the effect of hydrogen sulphide on the catalytic hydrodenitrogenation of pyridine and found that at 350°C and hydrogen sulphide pressure of 2 atmospheres, denitrogenation was complete, while at 300°C the maximum amount of nitrogen removed was 35%, the hydrogen sulphide pressure required being 10 atmospheres. In a U.S. patent, Johnson et al. (12) claimed that, in multiple stage catalytic hydrodesulphurization of residuum, increasing the concentration of hydrogen sulphide in the reactor decreased the reaction rate constant. In another U.S. patent, Helfrey et al. (13) claimed that, in catalytic hydrocracking of petroleum feedstocks, the aromatic content of the product could be controlled by regulating the hydrogen sulphide concentration in the reaction zone, and a product of desired quality could be obtained. Christman et al. (14) claimed that addition of hydrogen sulphide or a hydrogen sulphide precursor to the final stage had a beneficial effect in a multi-stage catalytic hydrodesulphurization process and maintained the catalyst in the sulphided state with a flat aging curve.

In another U.S. Patent, Aristoff et al. (15) claimed that, in the hydrocracking of the distillate fraction (218-232°C) to produce benzene and naphthalene, the addition of saturated hydrocarbon gases such as propane to the reactor zone with the feed accomplished a very marked reduction in the coke formation.

Chervenak et al. (16) and Wolk et al. (17) suggested that, in the preliminary refining of bitumen recovered from tar sands, the recycle-gas



purity must be greater than 60 vol % H<sub>2</sub>. However, most of their work was carried out with a recycle-gas purity of about 80 vol % H<sub>2</sub> or greater. Research on thermal hydrocracking of Athabasca bitumen at CANMET has been conducted with the recycle-gas purity of 85 vol % H<sub>2</sub>. The steady-state effects on product qualities and yields in the absence of recycle-gas scrubbing have been reported elsewhere (18).

## EXPERIMENTAL

### Feedstock

The characteristics of Athabasca bitumen used in the present investigation are given in Table 1.

### Apparatus and Procedure

Detailed descriptions of the pilot-plant and process were given in previous reports (19,8,9). A simplified schematic diagram of the pilot-plant is given in Fig. 1. Experiments were conducted in a 4.5-ℓ reactor at 13.89 MPa, 450°C, an LHSV of 1 and a recycle-gas rate of 0.0425 m<sup>3</sup>/h at the system pressure and 20°C. The temperature of the liquid-phase reactor (3.81 cm ID x 3.91 m long) was controlled by four equally spaced electrical heaters and was measured by five thermocouples inserted through the sides of the reactor wall and located centrally in the vessel. The temperatures of the hot catch pot (hot separator) and cold separator (cold receiver) were maintained at 350° and 20°C respectively.

The recycle-gas was oil-scrubbed before the addition of fresh make-up hydrogen (purity 100%). The recycle-gas purity, measured after the addition of make-up hydrogen, was varied by regulating the scrubber-oil rate. The scrubber vessel was 5.56 cm ID x 2.13 m long with an internal volume of 5.17 ℓ. A screen table 61 cm high was placed in the bottom and the vessel was packed with 0.635 cm Berl saddles to a height of 1.37 m leaving 15.24 cm clear at the top. The recycle-gas from the cold receiver entered through a stand-pipe in the bottom cap 5.84 cm high, passed up through the Berl saddles and exited at the top. Voltesso 35 scrubber-oil from an atmospheric supply tank was pumped in through the top cap using a Model B Duplex Milroyal pump. The scrubber-oil dripped down through Berl saddles contacting the rising

gases and vapours at the operating pressure and was then returned to the supply tank through a control valve. The level in the supply tank was controlled so that there was about 600 ml of oil in the system at all scrubber-oil rates. The level in the bottom of the scrubber vessel was maintained at about 20 cm. The hydrogen sulphide, ammonia and light hydrocarbon gases along with hydrogen were dissolved in the oil at high pressure. These gases came out of solution in the supply tank when pressure was reduced, and the flow of scrubber-oil off-gas was measured by a wet-test meter.

When starting the experiments, the system was pressurized with pure hydrogen (13.89 MPa) and tested for leaks. Then with gas recirculating at the rate of  $0.0425 \text{ m}^3/\text{h}$  computed at the system pressure and  $20^\circ\text{C}$ , the reactor and the hot catch pot (hot receiver) were brought up to  $350^\circ\text{C}$ . At this temperature, the feed flow was started and set at 4500 g/h and the preheaters were switched on. The reactor temperature was rapidly raised to  $430^\circ\text{C}$  at which time the scrubber pump was started to give a scrubber-oil rate of 8.26 g scrubber-oil/g feed. The reactor temperature was then increased by  $5^\circ\text{C}$  every two hours until it reached  $450^\circ\text{C}$ . When temperatures, feed rate, recycle-gas rate and recycle-gas purity were steady, a 4-hour test was carried out. At the end of the 4-hour test period, scrubber-oil rate was changed to another value, keeping other conditions unchanged, and when the recycle-gas purity reached a constant value, another 4-hour test was carried out. In this manner, 4-hour tests were carried out at different scrubber-oil rates. During 4-hour test periods, all gas flows were measured by wet-test meters, and liquid product samples were collected, weighed and analysed. The gas flows were expressed as  $\text{m}^3/\text{t}$  at atmospheric pressure and  $20^\circ\text{C}$  unless specified otherwise. The gas samples were collected half an hour after the start and half an hour before the end of the 4-hour test period, and analysed by a mass spectrometer.

The extent of conversion of the residual material (pitch) to distillable hydrocarbons (up to  $525^\circ\text{C}$  atmospheric equivalent) was determined using a Podbielniak equilibrium flash still (20,21). The product distribution of the distillate oil ( $-525^\circ\text{C}$ ) was determined using a modified U.S. Bureau of Mines Hempel distillation procedure (22). The sulphur contents of the liquid products were determined by X-ray fluorescence.

## RESULTS AND DISCUSSION

Experimental results of the investigation of the thermal hydrocracking of Athabasca bitumen at 13.89 MPa, 450°C, and LHSV of 1, a recycle-gas rate of 0.0425 m<sup>3</sup>/h at the system pressure and 20°C and at different scrubber-oil rates (0-8.26 g scrubber-oil/g feed) are given and discussed in three sections under the headings Gaseous Products, Liquid Products and General Discussion.

Gaseous Products

The composition of the recycle-gas after the addition of fresh make-up hydrogen as a function of scrubber-oil rate is shown in Fig. 2. As the scrubber-oil rate was increased, the hydrogen content was increased and the hydrocarbon gases (C<sub>1</sub>, C<sub>2</sub>, ..C<sub>5</sub>) as well as hydrogen sulphide contents decreased. At high scrubber-oil rates, most of the hydrogen sulphide and a considerable amount of hydrocarbon gases were removed from the recycle-gas. When the recycle-gas was not scrubbed, the recycle-gas after addition of make-up hydrogen contained, by volume, 59.9% H<sub>2</sub>, 1.6% H<sub>2</sub>S, 30.1% C<sub>1</sub>, 6.1% C<sub>2</sub> and 2.3% C<sub>3</sub>, C<sub>4</sub> and C<sub>5</sub>. However, at the scrubber-oil rate of 8.26 g/g, the composition of the recycle-gas was 95.8% H<sub>2</sub>, 0.07% H<sub>2</sub>S, 3.3% C<sub>1</sub>, 0.1% C<sub>2</sub> and 0.7% C<sub>3</sub>, C<sub>4</sub> and C<sub>5</sub>. The recycle-gas purity (Y) expressed as vol % H<sub>2</sub> was best correlated with the scrubber-oil rate (X) by a fourth degree polynomial, in the range of X values 0 to 8.3 g/g,

$$Y = 60.07 + 32.14 X - 13.18 X^2 + 2.30 X^3 - 0.13 X^4$$

The flow of off-gases as a function of scrubber-oil rate is shown in Fig. 3. The heavy-oil off-gas rate was nearly independent of the scrubber-oil rate. The slight variation in the recycle-gas-sample off-gas rate was due to the adjustment of the metering valve and was not related to the scrubber-oil rate. While the production of total off-gas as well as the scrubber-oil off-gas increased with increased scrubber-oil rate, that of the light-oil off-gas decreased. This was expected because, as the scrubber-oil rate was increased, more gases were removed in the scrubber-oil, thus affecting the equilibrium in the cold receiver. The total off-gas

produced was minimum ( $86 \text{ m}^3/\text{t}$ ) when the recycle-gas was not scrubbed. The hydrogen rates (make-up hydrogen and hydrogen consumption) and the off-gas rates (as  $\text{H}_2$ , H-C gases and  $\text{H}_2\text{S}$ ) as functions of scrubber-oil rate are shown in Fig. 4. The hydrogen consumption and the hydrogen sulphide rates were independent of the scrubber-oil rate, being 169 and  $16.8 \text{ m}^3/\text{t}$  respectively. The make-up hydrogen followed the pattern of hydrogen in the off-gases. This is understandable because, as the scrubber-oil rate was increased, more hydrogen was removed in the scrubber-oil off-gas and, since the hydrogen consumption was constant, more make-up hydrogen was required to maintain the system pressure. Operating with high purity recycle gas (i.e., high scrubbing rates) increases the cost of the process because of the greater requirement of fresh make-up hydrogen and the increased frequency of replacing the deteriorated scrubber-oil. The yield of hydrocarbon gases increased from 50 to  $60 \text{ m}^3/\text{t}$  as the scrubber-oil rate was increased from 0 to  $8.26 \text{ g/g}$  at the expense of liquid product yields.

The off-gas composition\* (as  $\text{H}_2$ ,  $\text{H}_2\text{S}$  and H-C gases) and the composite analysis\*\* of hydrocarbon gases as functions of scrubber-oil rate are shown in Fig. 5. The composite analysis of  $\text{H}_2$  and  $\text{H}_2\text{S}$ -free hydrocarbon gas was nearly constant with an average molecular weight of about 30-31. The average analysis by volume of this hydrocarbon gas was as follows:

Constituents	Vol. %
$\text{C}_1$ (Methane)	39.6
$\text{C}_2$ (Ethane & Ethene)	30.4
$\text{C}_3$ (Propane & Propene)	18.5
$\text{C}_4$ (Butanes & Butenes)	7.9
$\text{C}_5$ (Pentanes & Pentenes)	3.6

The off-gas composition depended on the scrubbing rate, with H-C gas and  $\text{H}_2\text{S}$  decreasing as more hydrogen was removed (Fig. 5).

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\* The off-gas composition is that obtained if all off-gases were mixed.

\*\* The composite analysis is the analysis which would be obtained if all off-gases were mixed and the  $\text{H}_2$  and  $\text{H}_2\text{S}$  removed.

### Liquid Products

The liquid product yields as functions of the recycle-gas purity are shown in Fig. 6. The pitch yield slightly increased with increased recycle-gas purity. The total liquid product yield as well as  $-525^{\circ}\text{C}$  distillate oil yield showed a slight but measurable variation. The decrease in the recycle-gas purity appeared to increase liquid yields while suppressing the formation of hydrocarbon gas. While the total liquid product yield was 90.9 wt % at the recycle-gas purity of 95.8 vol %  $\text{H}_2$ , it was 92.8 wt % at 59.9 vol %  $\text{H}_2$  purity (no scrubbing). This increase in total liquid yield corresponds to 85.5 g/h. The decrease in the hydrocarbon gas make from 66 to  $50 \text{ m}^3/\text{t}$  (Fig. 4) corresponds to a decrease of 89.8 g/h, which is consistent with the increase in the total liquid product yield.

While the sulphur removal was nearly the same ( $\sim 50$  wt %), the pitch conversion slightly decreased from 81.4 to 79.6 wt % as the recycle-gas purity increased from 59.9 to 95.8 vol %  $\text{H}_2$  (Fig. 7). The recycle-gas purity had very little effect on the densities of liquid products (Fig. 8). The scrubber-oil rate (i.e., the recycle-gas purity) had no effect on the sulphur and nitrogen contents of the liquid products (Fig. 9).

The asphaltene, coke and ash contents of the total liquid product as functions of the recycle-gas purity are shown in Fig. 10. The ash content was independent of the recycle-gas purity. The asphaltene content increased with purity while the coke content decreased. Operating with high purity recycle-gas (i.e., high scrubber-oil rate) could decrease the coke content of the total liquid product.

The effects of scrubber-oil rate on the liquid product distributions and saturate, olefin and aromatic contents of the distillate fractions (IBP- $200^{\circ}\text{C}$  and  $200$ - $250^{\circ}\text{C}$ ) are shown in Figures 11 and 12 respectively. The variations in Fraction 3 ( $250$ - $330^{\circ}\text{C}$ ) were opposite to the variations in Fraction 5 ( $418$ - $525^{\circ}\text{C}$ ) indicating that these fractions may be interrelated. Fractions 1, 2, 4 and 6 showed no variations with scrubber-oil rate (Fig. 11). The saturate, olefin and aromatic contents of the distillate fractions were not affected by the scrubber-oil rates (Fig. 12). These figures (11 and 12) indicate that liquid products of consistent quality could be obtained independent of the recycle-gas purity maintained during the operation.

Other properties related to +525°C pitch are shown in Fig. 13. Metal contents (nickel and vanadium) and Conradson carbon residue (CCR) decreased with increased scrubber-oil rate. While asphaltene and ash contents remained nearly constant at about 52 and 5.8 wt % respectively, the coke content of the pitch decreased slightly from 18.3 wt % at no scrubbing to 14.8 wt % at a scrubber-oil rate of 8.26 g/g.

During the course of the investigation, no operational problems were encountered. Inspection after 3 days of operation revealed that the reactor, hot separator, preheaters and all lines were clean and no coke deposits were found.

### General Discussion

The results of the present investigation are compared in Table 2 with previously published results at similar operating conditions. In the absence of scrubbing, the recycle-gas purity was 59.9 vol % H<sub>2</sub> in the present work, compared to 62.6 vol % H<sub>2</sub> as observed in a 60-hour run (18). The pitch conversion, hydrogen consumption, sulphur removal and production rates of hydrogen sulphide and hydrocarbon gases were quite similar. The liquid product yields and qualities were essentially similar indicating the reproducibility of the results.

When the recycle-gas purity was 85 vol % H<sub>2</sub>, the densities and sulphur contents of the liquid products observed in the present work were similar to those previously reported (8). The amounts of hydrogen sulphide and hydrocarbon gas-make were nearly the same in both cases. While the total liquid product yield and distillate oil yield were the same, the pitch yield was higher in the present work, 10 wt % compared to 7.8 wt %. The lower hydrogen consumption of 168 m<sup>3</sup>/t (6.99 g mol/kg) in the present case, compared to 183 m<sup>3</sup>/t (7.59 g mol/kg) as reported earlier, is explained by the fact that the pitch conversion was also lower, 80.5% compared with 84.4% as reported in reference 8.

The gas rates entering the reactor, hydrogen consumptions and average hydrogen partial pressures in the reactor during the course of the present investigation are summarized in Table 3. The recycle-gas rate is corrected for the compressibility factor at the particular composition and corresponds

to a constant recycle-gas rate of  $0.0425 \text{ m}^3/\text{h}$  at the system pressure and  $20^\circ\text{C}$ . The reported hydrogen rate is obtained by multiplying the recycle-gas rate by the recycle-gas purity and dividing the product by 100. The average hydrogen partial pressure is the average of the hydrogen partial pressures at the bottom and top of the reactor which were computed by taking into consideration the feed and product vaporization, liquid product yield, hydrogen consumption and hydrogen sulphide and hydrocarbon gas-make.

Examining Table 3, it is found that as the recycle-gas purity decreased the ratio of hydrogen input to hydrogen consumption also decreased but was always higher than 4.0. There was always an excess of hydrogen over that required for the reaction and this is the reason for the absence of a large effect on product yields and qualities. The hydrogen partial pressure also decreased as the recycle-gas purity decreased. However, it was always greater than 7 MPa at the system pressure of 13.89 MPa. In U.S. Patents concerning the upgrading of bitumens recovered from tar sands, Chervenak and Wolk (16,17) suggested that the purity of recycle-gas should be greater than 60 vol %  $\text{H}_2$  and the hydrogen partial pressure should be in the range of 1.4-11.0 MPa. However, most of their work was conducted at a hydrogen partial pressure of 5 MPa. Because, in the present investigation, the hydrogen partial pressure and the recycle-gas purity were 7 MPa and 60 vol %  $\text{H}_2$  respectively, or higher, no significant effect on product yields and qualities has been observed.

To find the true effect of hydrogen partial pressure and recycle-gas purity on product yields and qualities, similar experiments at lower system pressures of 7 and 10 MPa should be carried out to cover the hydrogen partial pressure range of 2-7 MPa. At the same time the effect of increased hydrogen sulphide and hydrocarbon gas content at lower recycle-gas purities (< 60 vol %  $\text{H}_2$ ) should be investigated. If the process could be operated at lower pressures with low recycle-gas purities, the capital as well as operating costs could be significantly reduced, thereby making the upgrading of Athabasca bitumen by a thermal hydrocracking process economically more attractive.

## CONCLUSIONS

Liquid products and hydrocarbon gas of consistent quality were obtained independent of recycle-gas purity. The production of hydrocarbon gas increased with increased scrubber-oil rate at the expense of total liquid product yield.

With hydrogen consumption of  $168 \text{ m}^3 \text{ API/t}$ , the average pitch conversion was 81%. The hydrogen partial pressure and recycle-gas purity did not affect the desulphurization reaction which is more dependent on the reaction temperature and liquid feed rate.

Operating with high purity recycle-gas could decrease the coke content of the total liquid product. However this would increase the cost of scrubbing because of a greater requirement of fresh make-up hydrogen.

The most significant finding of the present investigation is that the industrial hydrogen streams with purities of 60-85 vol %  $\text{H}_2$  could be utilized without purification in the upgrading of Athabasca bitumen, thereby reducing the overall process costs.

## ACKNOWLEDGEMENTS

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TABLE 1

Characteristics of Bitumen Feedstock

Density at 15 <sup>o</sup> C	kg/l	1.0084
Sulphur	wt %	4.48
Ash	wt %	0.59
Carbon	wt %	83.36
Hydrogen	wt %	10.52
Nitrogen	wt %	0.43
Kinematic viscosity at 210 <sup>o</sup> F	cst	133.3
Conradson Carbon Residue	wt %	13.3
Pentane insolubles	wt %	15.5
Benzene insolubles	wt %	0.72
Vanadium	ppm by wt	213
Nickel	ppm by wt	67

TABLE 2

Comparison of Results

Operating Conditions: P = 13.89 MPa, T = 450°C, LHSV = 1.0 and recycle-gas rate of 0.0425 m<sup>3</sup>/h at pressure and 20°C. Scrubbing results are for conditions to obtain 85% H<sub>2</sub> in recycle-gas.

	No Scrubbing		Scrubbing	
	This Work	Ref (18)	This Work (by inter- polation)	Ref (8)
Recycle-gas purity, vol % H <sub>2</sub>	59.9	62.6	85.0	85.0
H <sub>2</sub> S formed, m <sup>3</sup> /t	17	18	16	17
H-C gas make, m <sup>3</sup> /t	52	47	54	57
Hydrogen consumed, m <sup>3</sup> /t	167	173	168	183
Hydrogen consumed, g mol/kg	6.93	7.20	6.99	7.59
<u>Liquid Products</u>				
<u>Yields (based on feed)</u>				
	wt %			
Total liquid product	92.84	93.0	92.0	90.7
Distillate oil	83.29	84.0	82.0	82.9
Pitch	9.55	9.0	10.0	7.8
Pitch conversion, wt %	81.4	81.1	80.5	84.4
<u>Density at 15°C</u> kg/ℓ				
Total liquid product	0.9195	0.9230	0.9230	0.9210
Distillate oil	0.8957	0.8980	0.8965	0.8990
Pitch	1.2355	1.2490	1.2250	1.2400
<u>Sulphur Content</u> wt %				
Total liquid product	2.44	2.49	2.45	2.49
Distillate oil	2.27	2.30	2.20	2.27
Pitch	4.55	4.15	4.45	4.95
Sulphur removal, wt %	49.5	51.0	50.0	50.9
<u>Nitrogen Content</u> wt %				
Total liquid product	0.42	0.47	0.40	N/A
Distillate oil	0.28	0.31	0.30	
Pitch	1.66	1.97	1.65	
<u>Content in Total Liquid Product</u> wt %				
Asphaltene	3.89	3.90	4.40	N/A
Coke	2.23	1.65	1.6	
Ash	0.49	0.55	0.5	

TABLE 3

Gas Rates, Hydrogen Consumption and  
Hydrogen Partial Pressures

Operating Conditions: P = 13.89 MPa, T = 450°C, LHSV = 1.0 and  
recycle-gas rate of 0.0425 m<sup>3</sup>/h at pressure and 20°C

Scrubber- oil Rate g/g	Recycle- gas Purity vol % H <sub>2</sub>	Recycle- gas Rate m <sup>3</sup> /t	H <sub>2</sub> Rate m <sup>3</sup> /t	H <sub>2</sub> Consumed m <sup>3</sup> /t	H <sub>2</sub> Rate/ H <sub>2</sub> Consumed	H <sub>2</sub> Partial Pressure MPa
8.26	95.8	1192	1142	175.2	6.53	12.0
4.88	93.7	1204	1128	168.7	6.67	11.8
2.35	88.6	1207	1069	168.0	6.36	11.1
1.11	82.2	1233	1013	163.0	6.21	10.3
0.40	71.3	1283	915	169.5	5.40	8.8
0.0	59.9	1297	777	166.7	4.66	7.2

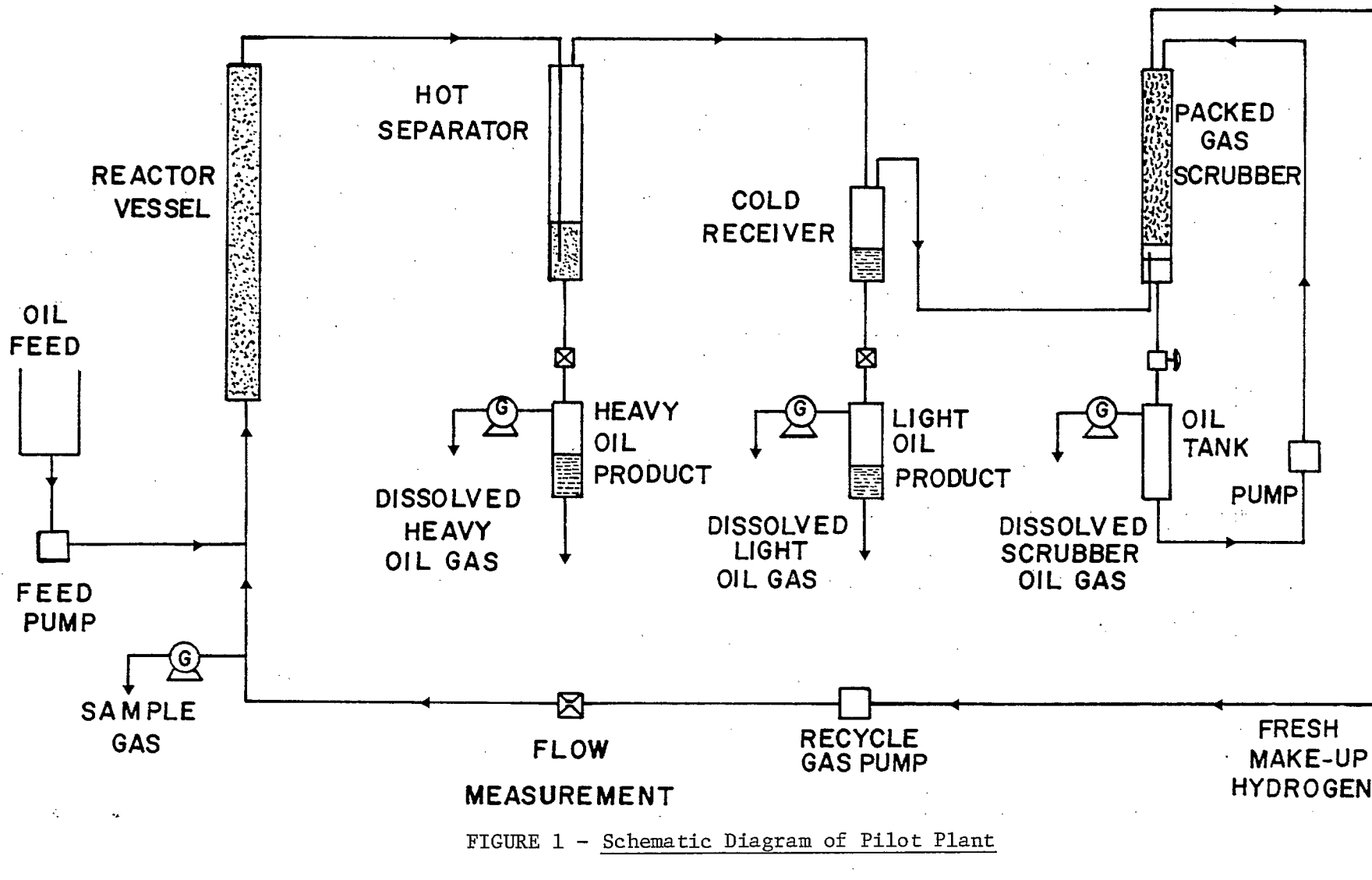


FIGURE 1 - Schematic Diagram of Pilot Plant

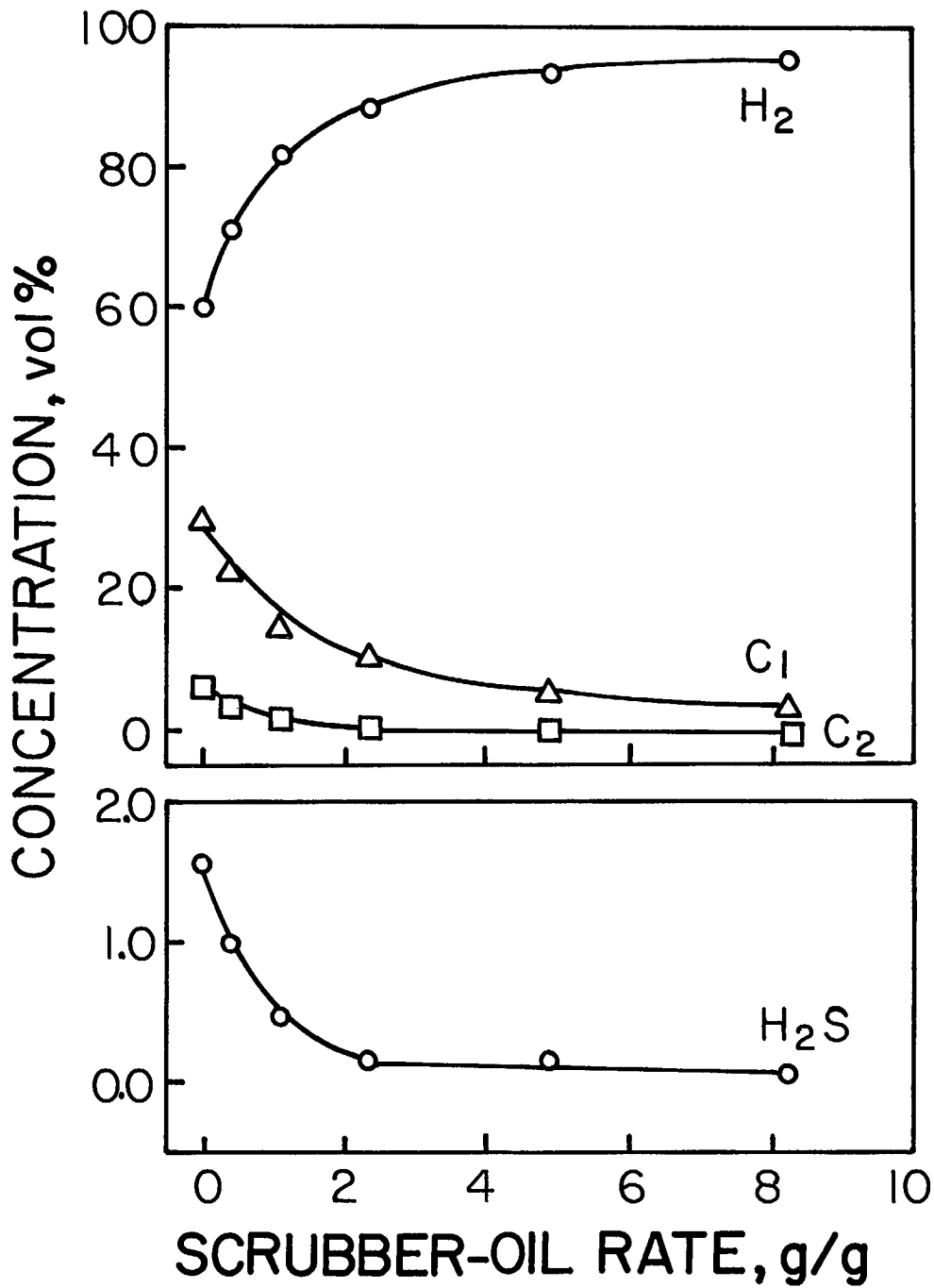


FIGURE 2 - Recycle-gas Composition as a Function of Scrubber-oil Rate

C<sub>1</sub> = methane, C<sub>2</sub> = ethane + ethene

Balance (not shown) consisted of C<sub>3</sub> = propane + propene, C<sub>4</sub> = butanes + butenes and C<sub>5</sub> = pentanes + pentenes

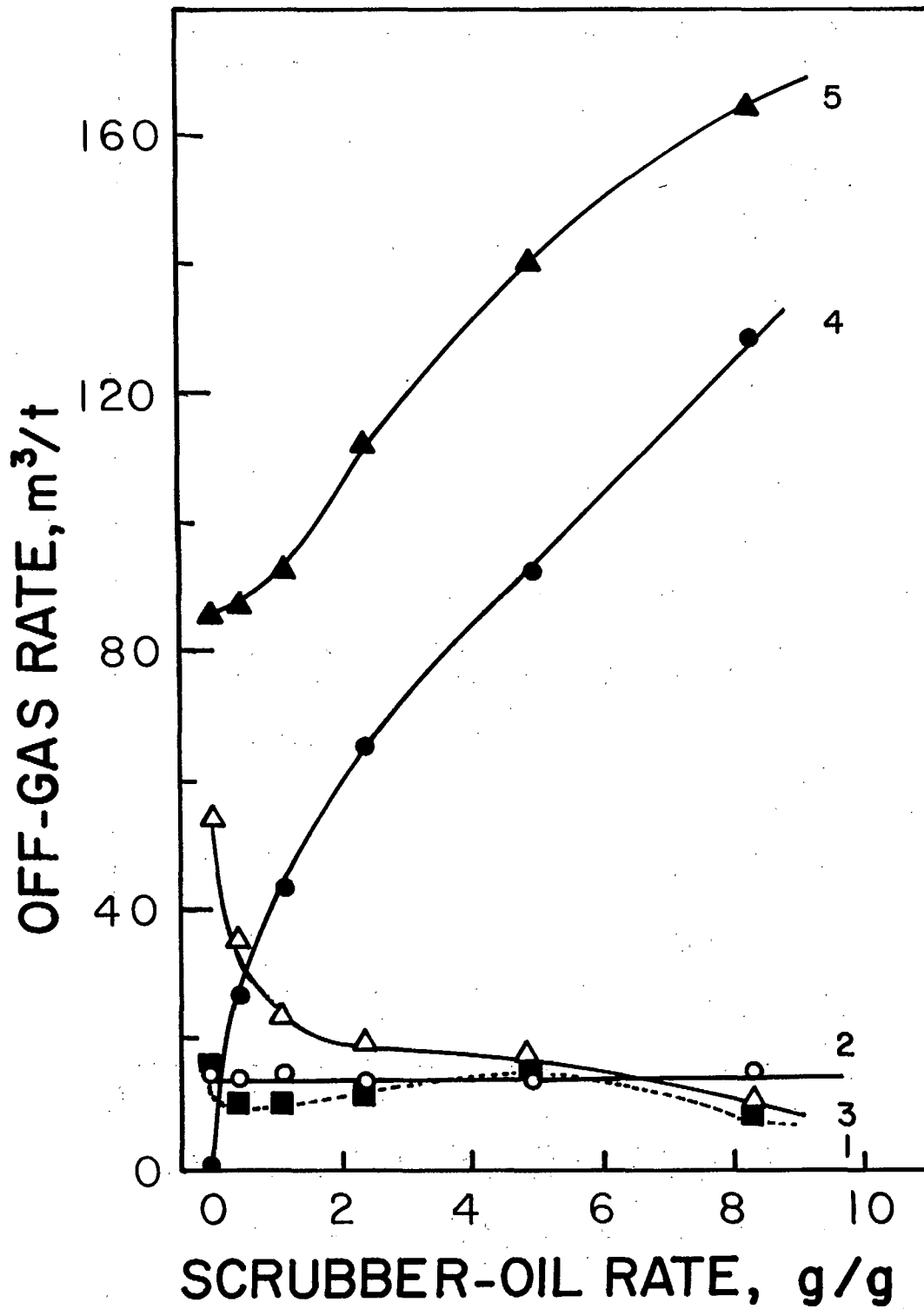


FIGURE 3 - Off-gas Rates as Functions of Scrubber-oil Rate  
 1 - Recycle-gas-sample off-gas, 2 - Heavy-oil off-gas,  
 3 - Light-oil off-gas, 4 - Scrubber-oil off-gas,  
 5 - Total off-gas (1+2+3+4)



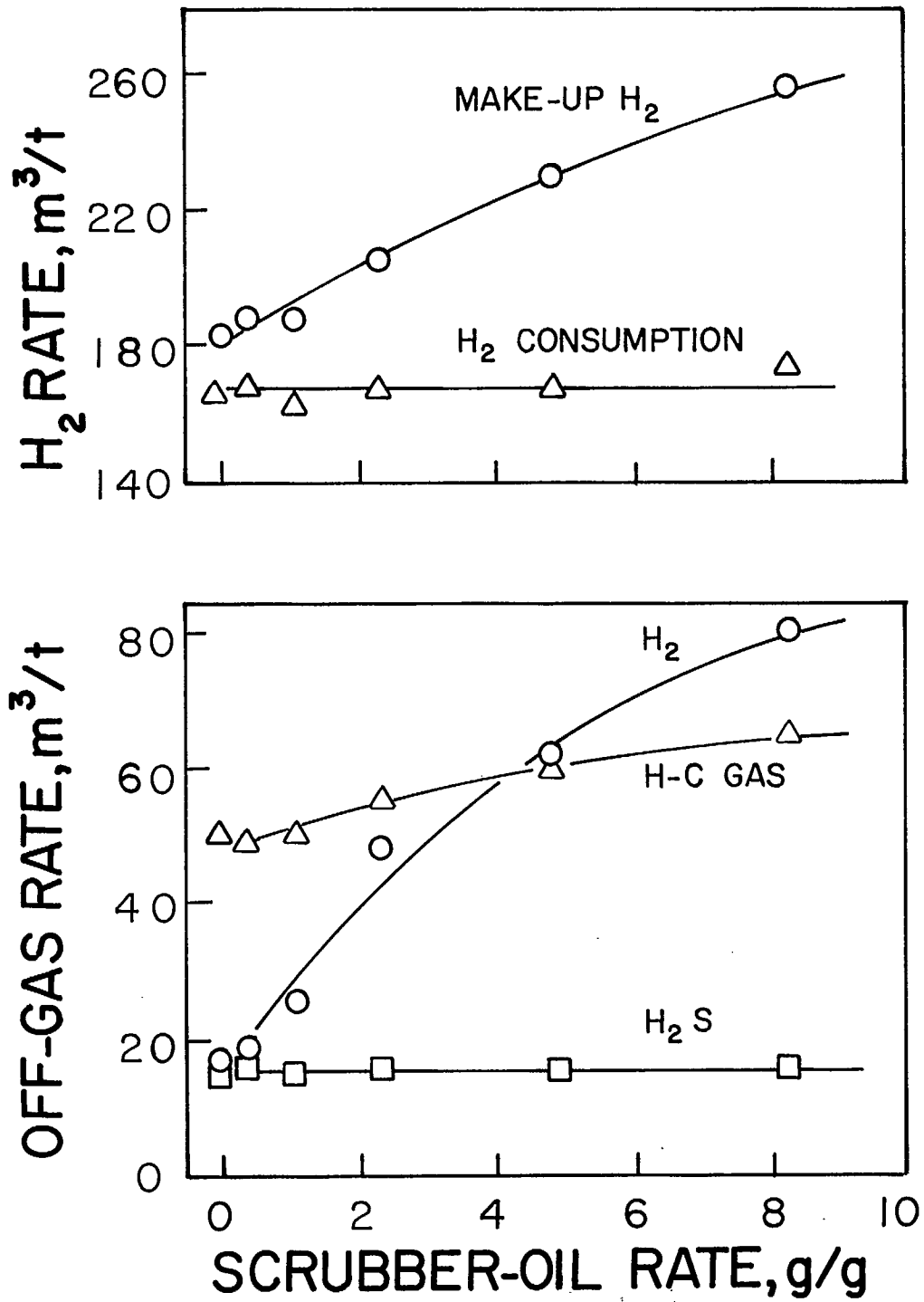


FIGURE 4 - Hydrogen Rates and Off-gas Rates as Functions of Scrubber-oil Rate

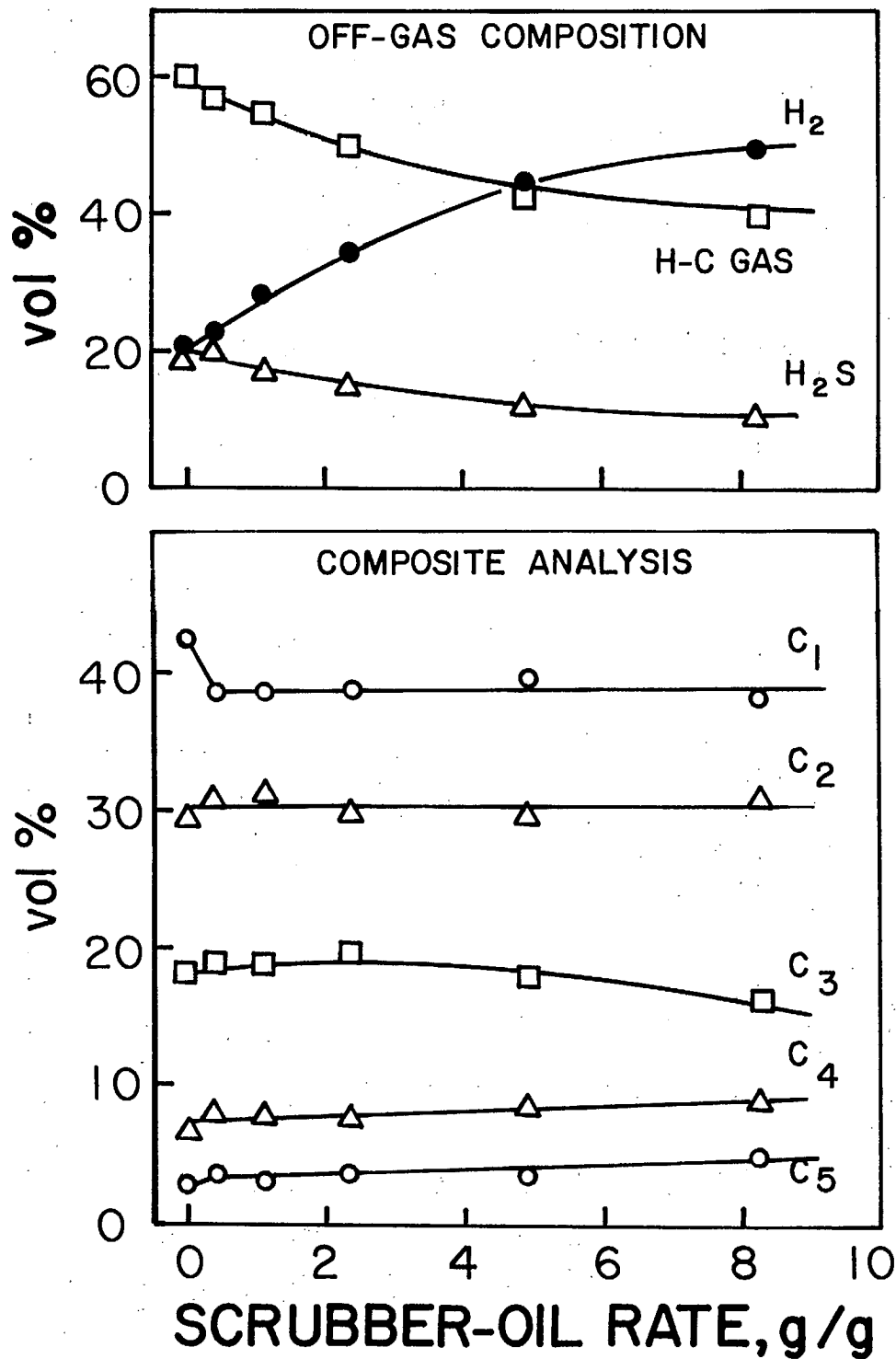


FIGURE 5 - Off-gas Composition and Composite Analysis of Hydrocarbon Gas as Functions of Scrubber-oil Rate

C<sub>1</sub> to C<sub>5</sub> - represent same constituents as in Fig. 2

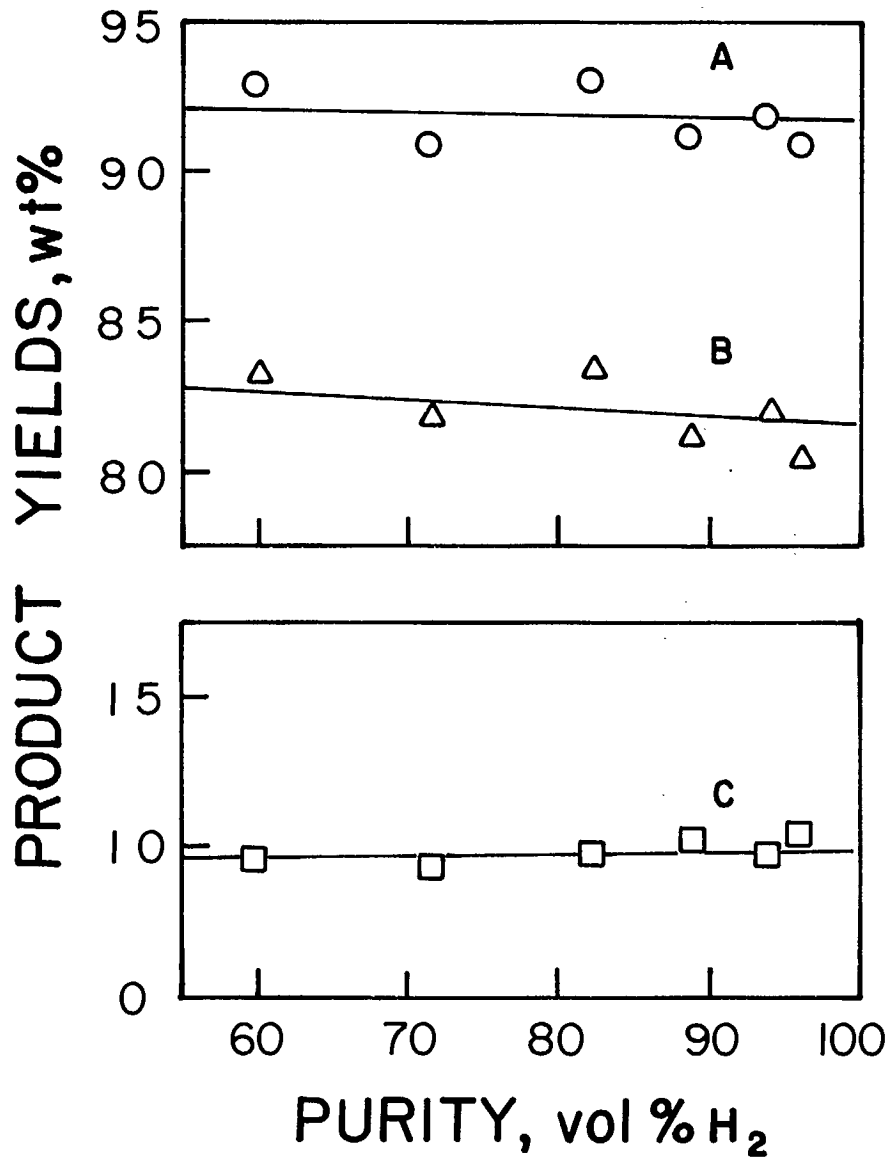


FIGURE 6 - Liquid Product Yields as Functions of Recycle-gas Purity

- A - Total liquid product
- B - Distillate oil
- C - Pitch

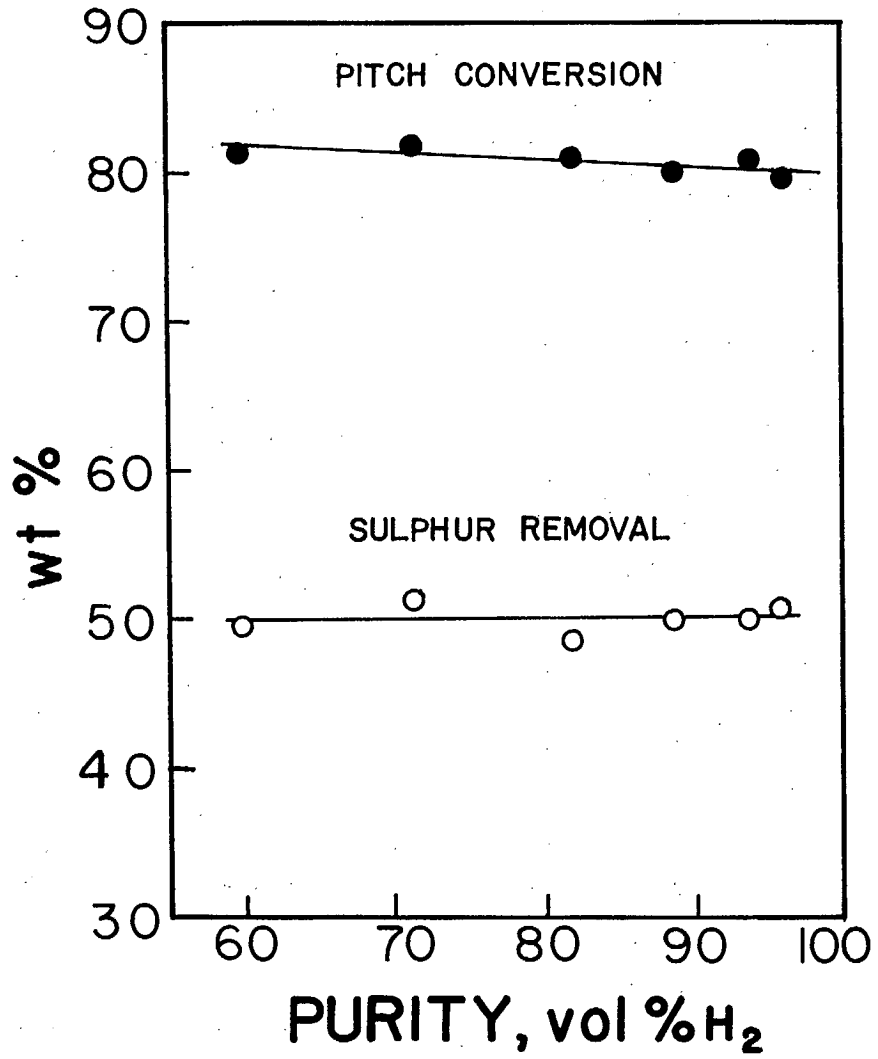


FIGURE 7 - Pitch Conversion and Sulphur Removal as Functions of Recycle-gas Purity

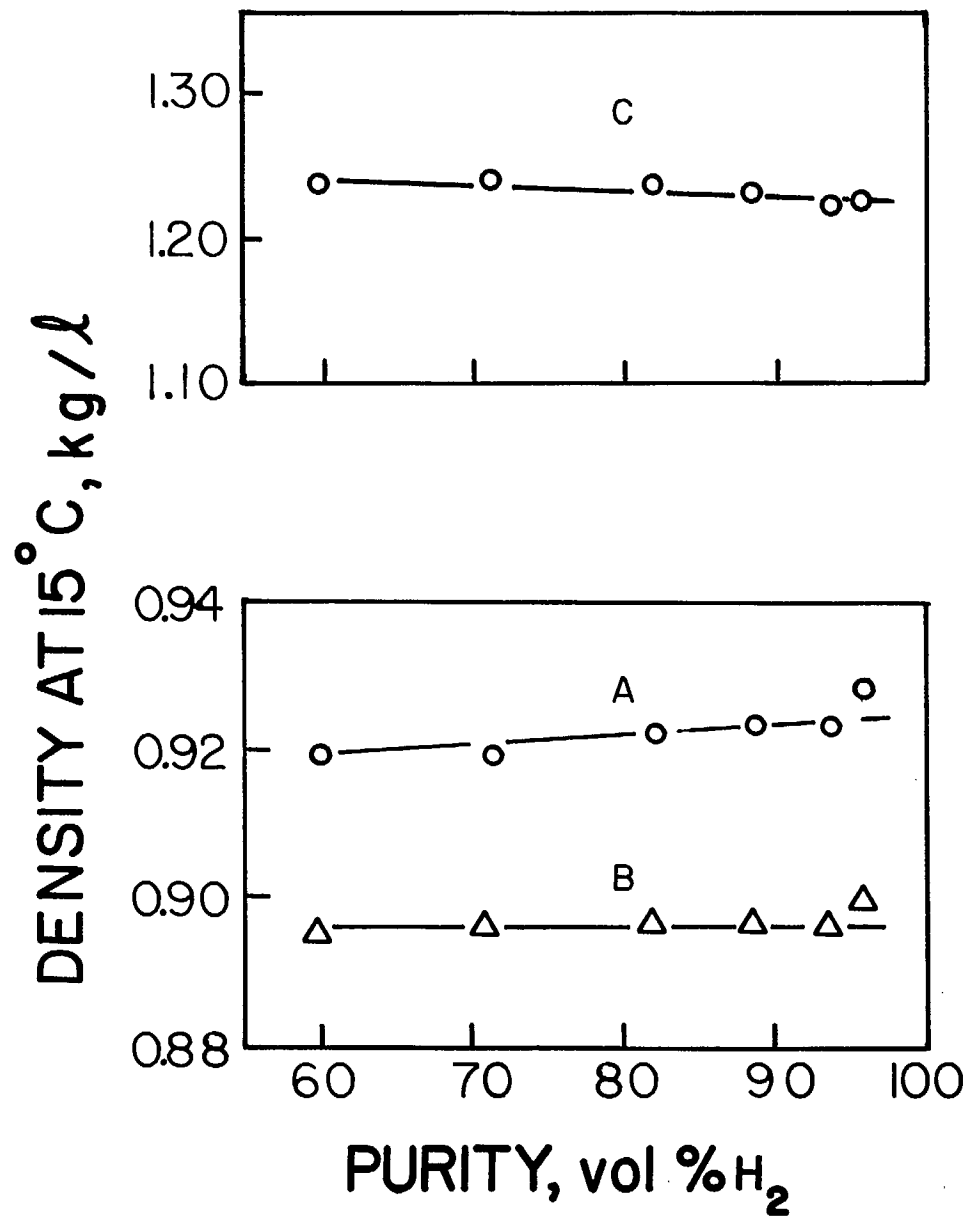


FIGURE 8 - Densities of Liquid Products as Functions of Recycle-gas Purity

- A - Total liquid product
- B - Distillate oil
- C - Pitch

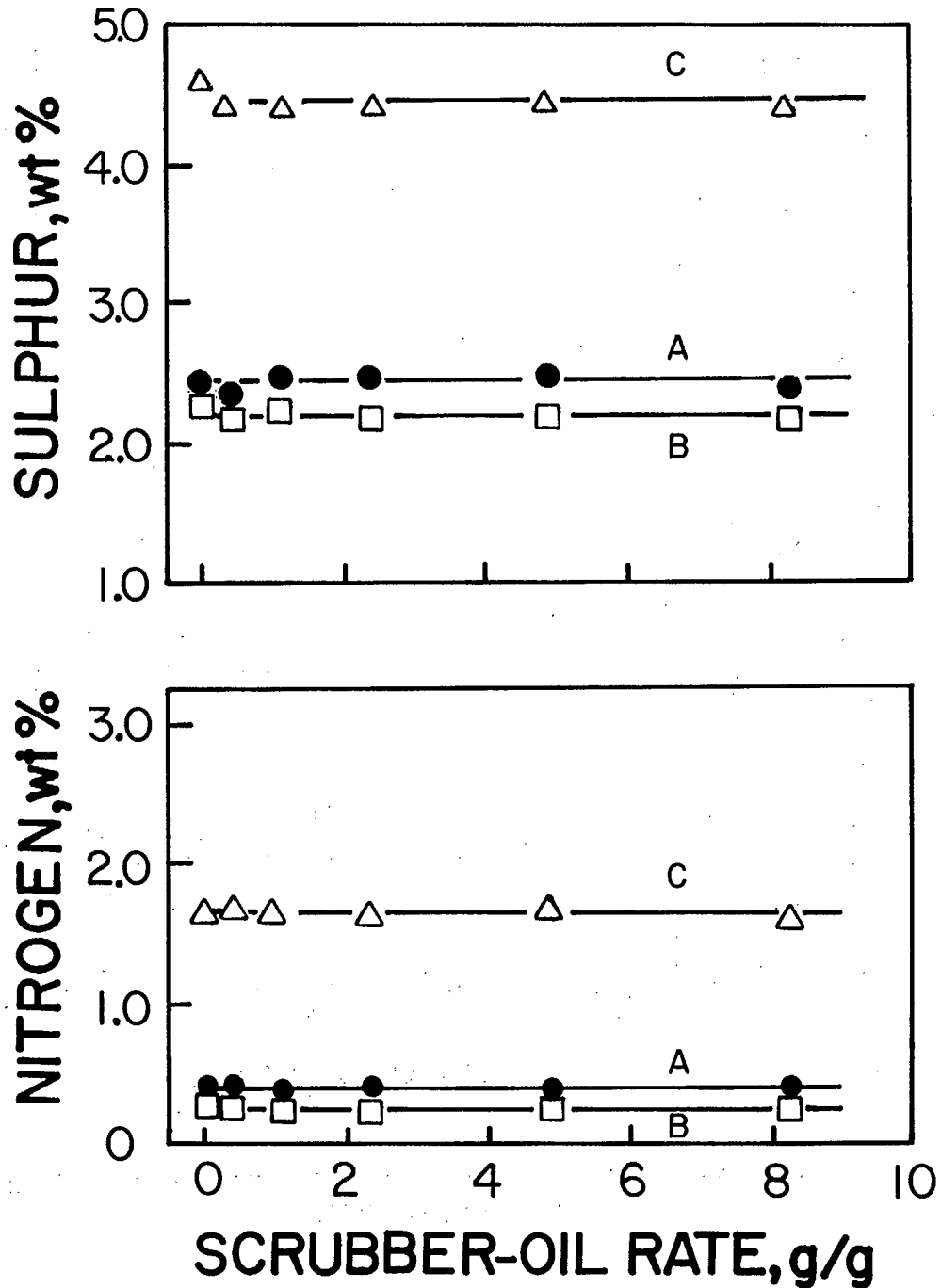


FIGURE 9 - Sulphur and Nitrogen Contents of Liquid Products as Functions of Scrubber-oil Rate

- A - Total liquid product
- B - Distillate oil
- C - Pitch

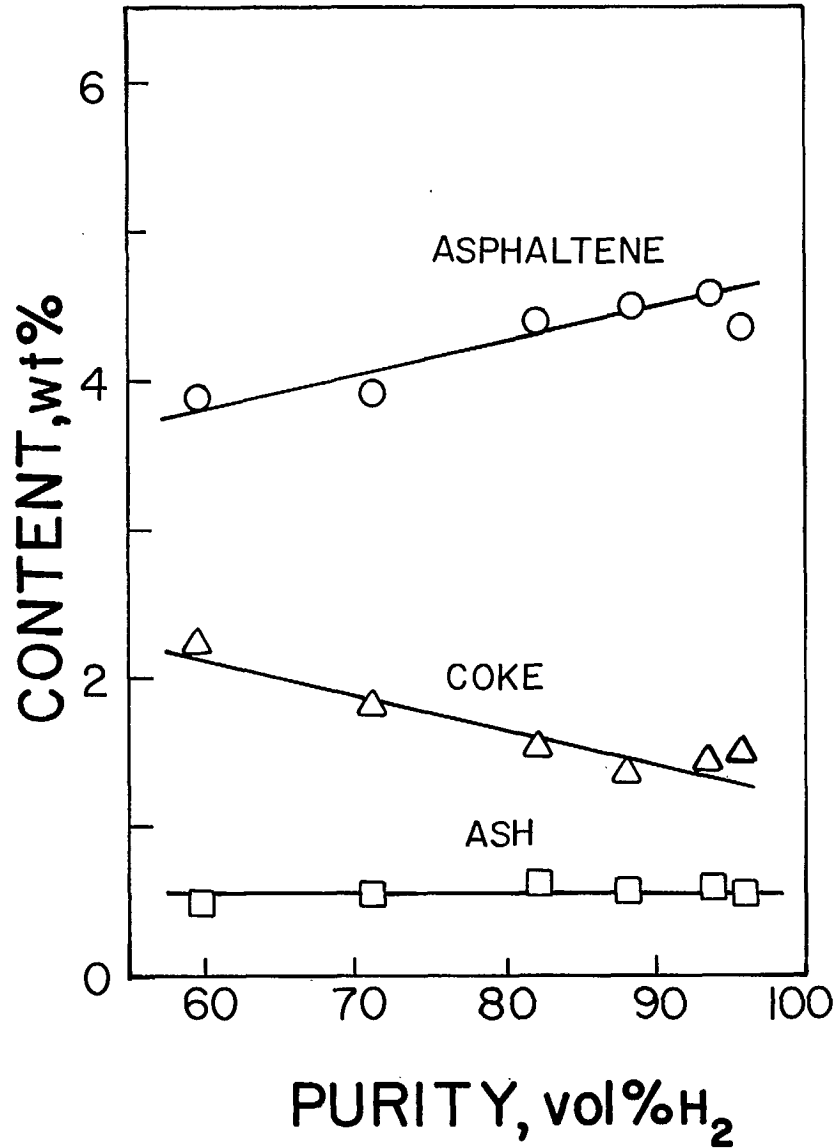


FIGURE 10 - Asphaltene, Coke and Ash Contents of Total Liquid Product as Functions of Recycle-gas Purity

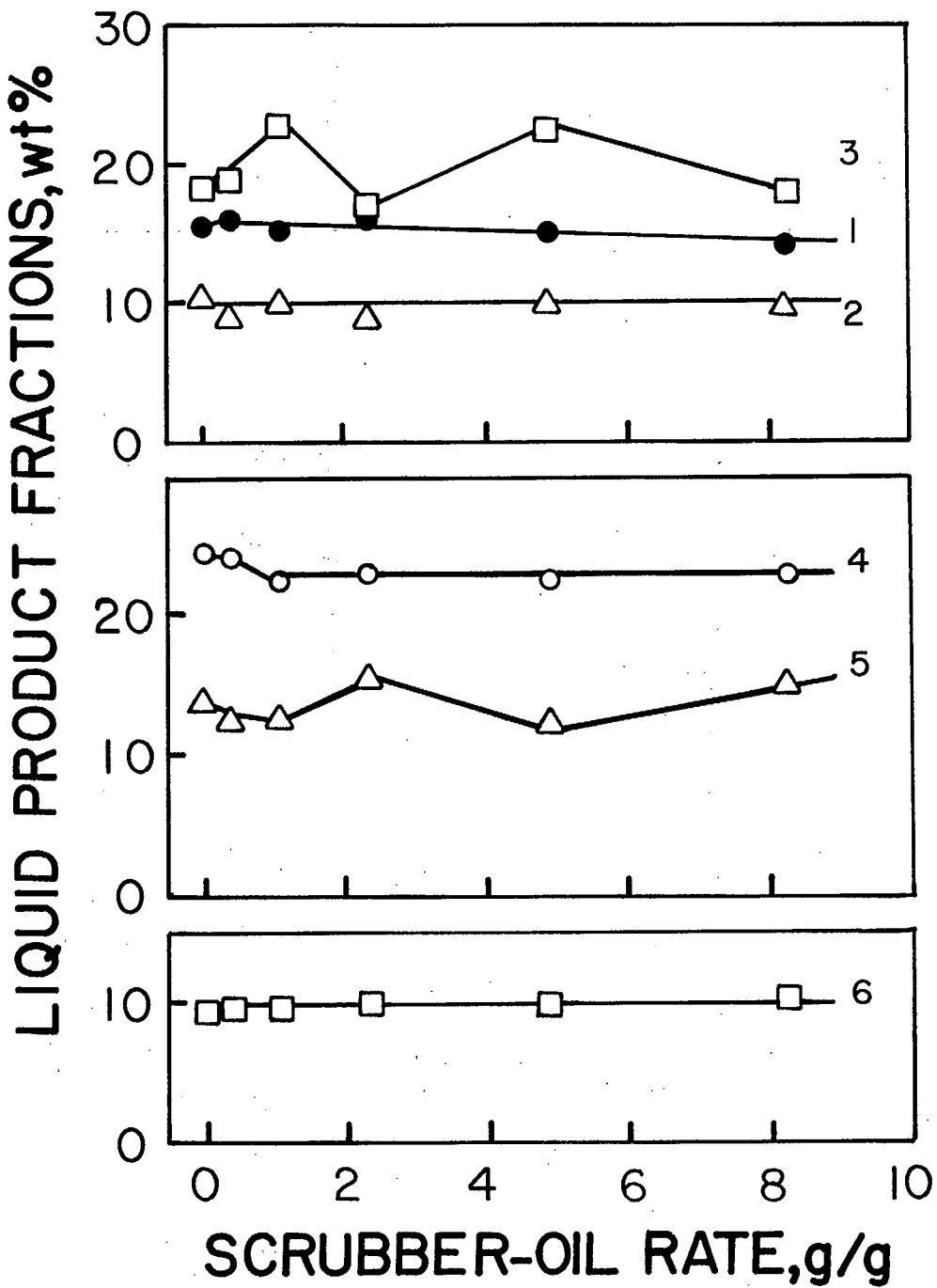


FIGURE 11 - Liquid Product Distributions as Functions of Scrubber-oil Rate

1. IBP-200°C, 2. 200-250°C, 3. 250-333°C,  
 4. 333-418°C, 5. 418-525°C, 6. +525°C



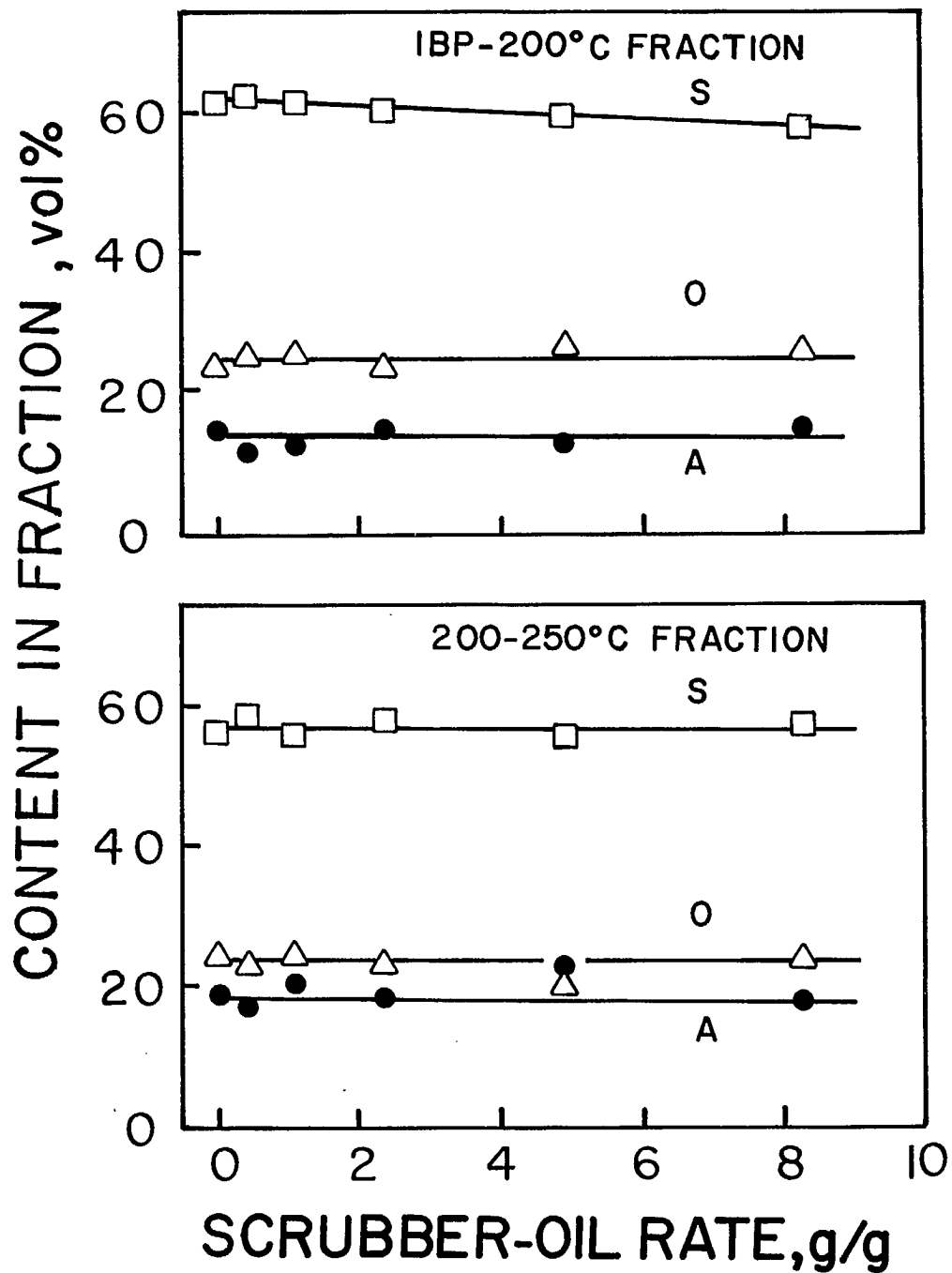


FIGURE 12 - Saturate, Olefin and Aromatic Contents of Distillate Fractions as Functions of Scrubber-oil Rate

S - Saturates, O - Olefins, A - Aromatics

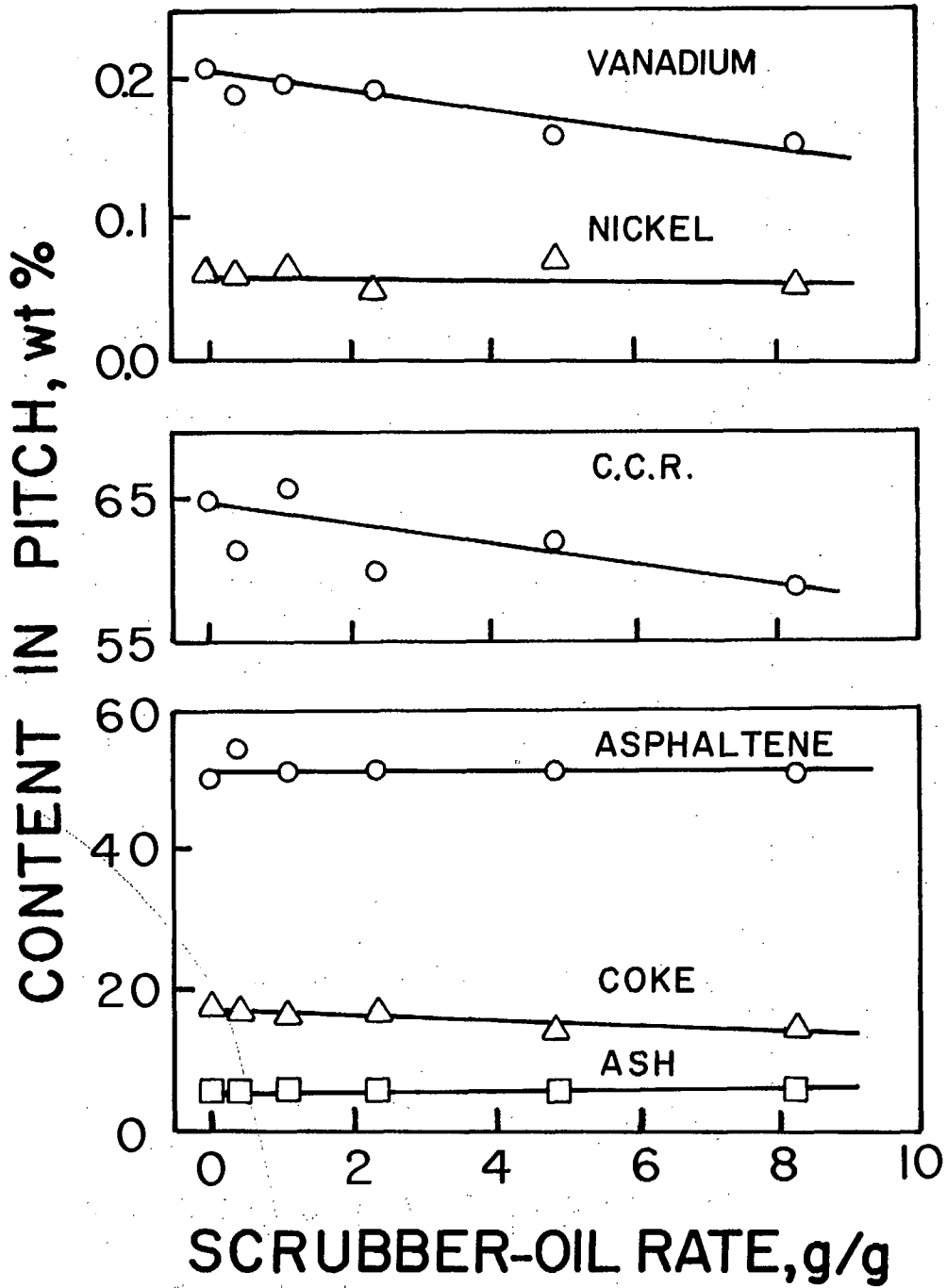


FIGURE 13 - Pitch Properties as Functions of Scrubber-oil Rate

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