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PRELIMINARY STUDIES ON THE DENITRO- GENATION OF DISTILLATES OBTAINED FROM THERMALLY-HYDROCRACKED BITUMEN

R. Ranganathan, J.M. Denis and B.I. Parsons
Canadian Fossil Fuel Research Laboratory
Process Engineering Section

JUNE 1976



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R. RANGANATHAN, J.M. DENIS AND B.I. PARSONS

CANADIAN FOSSIL FUEL RESEARCH LABORATORY
PROCESS ENGINEERING SECTION

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by

R. Ranganathan*, J.M. Denis** and B.I. Parsons***

ABSTRACT

Distillates of the product obtained from thermal hydrocracking of bitumen contain less sulphur but more nitrogen than distillates from bitumen coking processes. The high nitrogen content of naphtha and gas-oil distillates can be detrimental in the downstream secondary refining processes, especially catalytic cracking. Hence, preliminary studies were carried out to obtain the secondary hydrotreating characteristics of the thermal-hydrocracker (THC) distillates. All hydrotreating studies were carried out in a bench-scale flow reactor using a 3.0 wt % CoO - 12 wt % MoO₃/alumina catalyst.

The denitrogenation studies showed that, in spite of the high nitrogen content of thermal-hydrocracker naphtha distillate, virtually complete removal of nitrogen was possible in a single-stage reactor. The denitrogenation characteristics of two gas-oils containing different amounts of nitrogen are reported. The results indicated that the initial nitrogen content did not have any effect on the per cent nitrogen converted at various reaction conditions. Basic-nitrogen removal was also determined for gas-oil and kerosene distillates. The analysis for diene content in naphtha showed that diene content of THC naphtha feed was low and was completely eliminated during hydrotreating.

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ETUDES PRELIMINAIRES DE LA DENITRIFICATION DE DISTILLATS OBTENUS
APRES L'HYDROCRAQUAGE THERMIQUE DU BITUME

par

R. Ranganathan*, J.M. Denis** et B.I. Parsons***

RESUME

Les distillats du produit résultant de l'hydrocraquage thermique du bitume contenaient moins de soufre et plus d'azote que ceux obtenus par cokéfaction. La haute teneur en azote des distillats de naphte et de gaz-huile peut être néfaste lors des procédés secondaires de raffinage et tout particulièrement lors de craquage catalytique. Par conséquent, les études préliminaires ont été effectuées de façon à obtenir les caractéristiques des distillats obtenus de l'hydrocraquage thermique secondaire (THC). Les expériences de traitement à l'hydrogène ont donc pris place dans un réacteur à écoulement de laboratoire à l'aide d'un catalyseur de 3.0% de CoO, en poids - 12% de MoO₃, en poids/alumine.

Les études de dénitrification démontrèrent que, malgré le fort contenu d'azote dans les distillats de naphte hydrocraqués thermiquement, presque tout l'azote a pu être éliminé à l'aide d'un réacteur à phase simple. Les caractéristiques de deux gas-huiles dénitrifiées contenant différentes quantités d'azote sont décrites. On observa que le contenu initial d'azote n'a eu aucun effet sur le pourcentage d'azote converti dans des conditions diverses de réaction. L'élimination d'azote basique des distillats de kérosène et de gas-huile a aussi été calculée. L'analyse du contenu diénique de la naphte démontra qu'il était faible dans la charge d'alimentation et qu'il a été entièrement éliminé pendant le traitement à l'hydrogène.

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INTRODUCTION

Research on the refining of Athabasca bitumen is essential to develop an economically feasible process for obtaining industrially acceptable synthetic crude. As a part of the Energy Research Program, upgrading of Athabasca bitumen by the thermal hydrocracking process has been studied in the Energy Research Laboratories for several years⁽¹⁾. Thermal hydrocracking pilot-plant studies were carried out with the objective of maximizing distillate-oil yield from bitumen and minimizing coke formation. The thermal hydrocracking process involves hydrogen addition as opposed to carbon removal in the alternative coking upgrading processes. Distillates obtained from thermal-hydrocracker (THC) product would have different properties and hydrotreating characteristics compared with those of coker distillates. Hence, studies on the secondary refining of thermal-hydrocracker product distillates are needed in order to complete the information on the total refining of Athabasca bitumen.

The thermal-hydrocracker product was distilled into three fractions and these were tested for sulphur and nitrogen. The thermal-hydrocracker product contained lower amounts of sulphur and higher amounts of nitrogen compared to delayed- and fluid-coker distillates. For example, the thermal-hydrocracker naphtha distillate (82 to 204°C) contained 1.14 wt % sulphur compared with 2.2 wt % sulphur in the delayed-coker naphtha⁽²⁾. However, the THC naphtha distillate contained 884 ppm nitrogen compared with 150 ppm nitrogen in the delayed-coker naphtha distillates⁽²⁾. Similarly, THC kerosene and THC gas-oil distillates contain more nitrogen than delayed- and fluid-coker distillates. The high nitrogen content of THC distillates might require severe hydrotreating conditions in the secondary refining stage. On the other hand, because the bitumen has been treated by a high-pressure thermal-hydrogenation process, the nitrogen compounds could be less refractory.

For naphtha distillate, as the platinum reformer catalysts are easily poisoned by nitrogen compounds, it is essential that nitrogen content be reduced to below 1 ppm before sending the distillate to the catalytic reformer⁽³⁾. Thomas⁽³⁾ suggested that, for a naphtha feed containing high nitrogen, two-stage treaters with a cobalt-molybdenum catalyst in the first stage and a nickel-molybdenum catalyst in the second stage would be necessary to bring

the nitrogen level down to 1 ppm. Associated Engineering Services Ltd. (AESL), of Alberta, during their discussions with the ERL Process Engineering Group concerning economic evaluation of thermal hydrocracking processes, expressed the opinion that it would be difficult to carry out the denitrogenation of THC naphtha, with 884 ppm N, in a single stage. A two-stage reactor scheme would increase the costs of the overall thermal-hydrocracking process. AESL suggested that hydrodenitrogenation of THC gas-oil might also be difficult because of high nitrogen content. Hence, preliminary studies were carried out to evaluate nitrogen removal from THC naphtha in a single-stage reactor and to study denitrogenation characteristics of THC kerosene and THC gas-oils. Two THC gas-oil distillates with different nitrogen contents were also compared to evaluate the effect of feed nitrogen concentration.

EXPERIMENTAL

Feed Stocks

Properties of the THC naphtha (82-204^oC), THC kerosene (204-279^oC) and THC gas-oils (343 - 524^oC) are listed in Table 1. These materials were obtained by distilling various thermally-hydrocracked bitumen products. THC gas-oil 1 and THC gas-oil 2 were distilled from a blend of thermal-hydrocracker products obtained under different hydrocracking conditions and hence contained different amounts of nitrogen.

Hydrotreating

The hydrotreating experiments were carried out using a bench-scale up-flow reactor described in previous CANMET reports^(4, 5). Cobalt-molybdenum catalyst (3 wt % CoO - 12 wt % MoO₃/alumina, 3.2 mm pellets, Harshaw Chemical Company, catalogue no. CoMo 0603T) was used. The reactor was 2.5 cm in internal diameter, 0.305 m long and was made of 316 stainless steel. The reactor was sequentially filled from the bottom with 42 ml of berl saddles, 100 ml of catalyst pellets and 13 ml of berl saddles. The distillate oil and hydrogen flowed up through the fixed catalyst bed. Gas and liquid reaction products were separated downstream. After steady-state conditions had prevailed for 1 hour, liquid product was collected for 2 hours. Product samples were analysed for specific gravity, sulphur, nitrogen, aromatics, olefins and saturates. Details of the procedures for these analyses are

described elsewhere^(4, 5, 6). Basic-nitrogen contents of distillates and diene values of naphtha were measured using standard UOP tests⁽⁷⁾.

Previous studies⁽⁶⁾ have shown that catalyst pretreatment is important in obtaining stable catalytic activity. Hence, the catalyst bed was pretreated and stabilized before carrying out the reaction runs. The pretreatment conditions depended on the feed stock used. As naphtha distillate contained low sulphur, kerosene was used to stabilize the catalyst bed before hydrotreating naphtha. Pretreatment was carried out with kerosene at reaction conditions of 13.9 MPa (2000 psig), 400°C, space velocity of 2.0 and 890 m³ H₂/m³ oil (5000 scf/bbl) for two hours. In the case of kerosene hydrotreating, pretreatment conditions were the same as above. For gas-oil runs, pretreatment was done at 13.9 MPa, 400°C, space velocity of 1 and 890 m³ H₂/m³ oil for five hours.

After pretreatment, the reaction studies were carried out at various temperatures. The stability of the catalyst after a series of experiments was checked by repeating the first experiment in the series. Hydrotreating studies on THC naphtha and THC kerosene were carried out at several sets of reaction conditions. Hydrotreated gas-oil samples from previous studies conducted by Parsons et al.^(4, 5) were analysed for total- and basic-nitrogen contents. In this report, the denitrogenation characteristics for total nitrogen and basic nitrogen in gas-oil distillates are discussed in addition to the hydrotreating characteristics of THC naphtha and THC kerosene distillates.

RESULTS AND DISCUSSION

Naphtha

Hydrotreating runs were carried out at a space velocity of 1 and a hydrogen flow rate of 890 m³ H₂/m³ oil. Results for 13.9 MPa pressure and 420°C (Table 2) show that nitrogen in the naphtha product is 1 ppm. However, the experiments at 5.6 MPa (800 psig) (Table 2) show that even for reaction at 360°C the nitrogen content is below 1 ppm. A slightly lower activity for desulphurization and denitrogenation at 13.9 MPa (2000 psig) is probably an error introduced by the experimental sequence of runs. The experiments with naphtha at 13.9 MPa were carried out immediately after the stabilization runs with kerosene. Sulphur and nitrogen absorbed on the

catalysts and the kerosene in the catalyst pores were probably entrained with the naphtha product. This would result in higher levels of sulphur and nitrogen in the product. It is interesting to note that the results for the hydrogenation of aromatics were not affected. This is because of similar unsaturates (olefins plus aromatics) in both kerosene and naphtha distillates (Table 1). The weight and volume yields of the product were above 90% at all hydrotreating conditions studied (Table 2).

Denitrogenation and desulphurization results, at 5.6 MPa and at different temperatures, show that there was no apparent decline in activity during the 14 hours of experiments. This was confirmed by running a duplicate 400°C test at the end of the series (Table 2). Runs at higher space velocities for longer periods should be conducted to evaluate the effect of high nitrogen content on the catalyst life.

The maleic anhydride values (MAV) of naphtha distillates and products were determined to obtain a measure of conjugated diolefin content. MAV values close to zero would indicate absence of diolefins. Since dienes are highly unstable and polymerize very easily, a pre-refining step is often required if the diene contents are high. However, it was found that THC naphtha had a MAV of only 4.5 compared with 112 in a pyrolysis naphtha⁽⁸⁾. The low MAV value for THC naphtha suggests that a pre-refining step is not required in upgrading naphtha distillate. Further, as the denitrogenation and desulphurization conversions did not decline during 14 hours of reaction runs, it is apparent that the presence of small amounts of diene did not affect the catalytic activity.

Kerosene

In hydrotreating kerosene, in addition to removing sulphur and nitrogen, aromatics should also be reduced in order to meet fuel-oil specifications. Fuel-oil specifications require an aromatic content of less than 20 vol %⁽⁹⁾. These specifications are met for the THC kerosene processed at 400°C and a space velocity of 1 (Table 3). Hydrogenation of aromatics was found to be more difficult than denitrogenation and desulphurization. For example, at 400°C and a space velocity of 2, 98% of the sulphur and nitrogen compounds were removed. On the other hand, only 43% of unsaturates were removed.

The denitrogenation conversions were less than the desulphurization conversions at temperatures below 380°C (Figure 1). However, at higher temperatures, denitrogenation and desulphurization conversions were the same. The basic-nitrogen to total-nitrogen ratios of the distillates (Table 1) showed that kerosene contained more basic nitrogen than naphtha and gas-oil. After comparing several feed stocks, Richter et al.⁽¹⁰⁾ also reported that some kerosene fractions contained more basic nitrogen than naphtha and gas-oil fractions. The basic-nitrogen determination for kerosene hydrotreated at 320°C indicated that it is removed with greater difficulty than non-basic nitrogen. Basic-nitrogen conversion was found to be only 38% compared with 61% for total-nitrogen conversion.

Gas-oils

The results in Figure 2 are for the denitrogenation of gas-oils having different nitrogen contents. Nitrogen contents could be easily reduced below 2000 ppm at temperatures between 380°C and 420°C and a liquid space velocity of 2. Figure 3 shows that, irrespective of the initial nitrogen concentration, the variations of per cent nitrogen conversions are the same under different reaction conditions. Only one experimental point at low space velocity and temperature (1 and 360°C) did not conform with these results. Hence, this graph can be used as a reasonable first approximation for obtaining the denitrogenation reaction conditions for thermal-hydrocracker gas-oil distillates. However, more studies should be carried out to define the correlation.

Basic-nitrogen compounds are partially responsible for poisoning acidic cracking catalysts. They chemisorb on acid sites and reduce catalytic activity. Even though basic-nitrogen compounds are often only temporary poisons (as they can be removed in the regenerator) it is helpful to remove most of the basic-nitrogen during catalytic hydrotreating before sending the feed for catalytic cracking. Both gas-oil 1 and gas-oil 2 contained 37% basic-nitrogen, but different amounts of total nitrogen (Table 1). In Figure 4, basic-nitrogen removal at various temperatures and space velocities is shown. At temperatures of 420°C, about 68% conversion could be obtained. However, at temperatures below 340°C, basic-nitrogen compounds are not converted.

A comparison of total-nitrogen and basic-nitrogen conversion is shown in Figure 5A. At values less than 50%, basic nitrogen conversions are less than total-nitrogen conversions. However, it is seen from Figure 5B that, at higher values, more basic-nitrogen is transformed since the basic-/total-nitrogen ratio of the product decreased. Denitrogenation results at higher conversions agree with the work reported by Abdou et al.⁽¹¹⁾ who found that basic-nitrogen compounds reacted faster than non-basic nitrogen compounds. However, it was found in our work that at low nitrogen conversions the basic-nitrogen compounds reacted more slowly than non-basic nitrogen.

SUMMARY

The results on denitrogenation of naphtha show that nitrogen can be removed in a single-stage reactor. The catalytic activity remained stable during the 14 hours of experimental studies at different temperatures. Further studies should be carried out to obtain optimum reaction conditions and to determine the effect on the catalyst life of high nitrogen levels in the naphtha distillate. The diene content of naphtha feed was low and was completely eliminated during hydrotreating.

In the case of kerosene distillate, it was found that fuel oil specifications for aromatics were met at reaction conditions of 13.9 MPa, 400°C and space velocity of 1. The hydrogenation of aromatics in kerosene was found to be more difficult than denitrogenation and desulphurization.

The denitrogenation conversions were found to be similar for the two gas-oils containing different nitrogen contents. The basic nitrogen was not converted at temperatures below 340°C. The basic-/total-nitrogen ratio in hydrotreated products decreased with increasing conversion.

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TABLE 1

General Properties of THC Distillates

Property		Naphtha	Kerosene	Gas-oil 1	Gas-oil 2
Boiling range	°C	82-204	204-279	343-524	343-524
Specific gravity, 15/15°C		0.782	0.879	0.975	0.992
Sulphur	wt %	1.14	2.31	3.44	3.59
Nitrogen	ppm	884	1347	2829	3800
Basic Nitrogen	ppm	450	796	1056	1411
Basic/total Nitrogen	ratio	0.509	0.590	0.373	0.371
Aromatics + olefins	vol %	40.3	38.8	-	-
Saturates	vol %	59.7	61.2	-	-

TABLE 2

Product Quality of Hydrotreated THC Naphtha

Reaction Pressure MPa	Reaction Temp °C	Specific Gravity 15/15°C	Nitrogen ppm	Sulphur wt %	Aromatics + olefins vol %	Saturates vol %	Liquid Yield wt %	Liquid Yield vol %
13.9	400	0.764	3.7	0.10	4.3	95.7	94.5	96.7
13.9	420	0.762	1.0	0.12	3.0	97.0	94.2	96.7
13.9	360	0.765	3.1	0.10	8.0	92.0	91.8	93.8
13.9	320	0.768	7.6	0.11	10.1	89.9	95.4	97.1
13.9	400	0.765	1.2	0.08	4.3	95.7	90.7	92.7
5.6	400	0.767	<1.0	0.08	11.0	89.0	94.8	96.7
5.6	420	0.767	<1.0	0.04	13.3	86.7	93.7	95.5
5.6	360	0.768	<1.0	0.03	10.6	89.4	95.1	96.8
5.6	320	0.769	30	0.05	11.0	89.0	90.8	92.3
5.6	400	0.767	<1.0	0.03	9.8	90.2	95.0	96.9

TABLE 3

Product Quality of Hydrotreated THC Kerosene

Reaction Pressure MPa	Reaction Temp °C	Space Velocity hr ⁻¹	Specific Gravity 15/15°C	Nitrogen ppm	Sulphur wt %	Aromatics + olefins vol %	Saturates vol %	Liquid Yield wt %	Liquid Yield vol %
13.9	396	2	0.842	16	0.05	22.9	77.1	98.4	103
13.9	417	2	0.837	5	0.08	21.9	78.1	97.2	102
13.9	358	2	0.849	88	0.10	28.1	71.9	98.0	102
13.9	317	2	0.853	526	0.16	41.0	59.0	98.1	101
13.9	397	2	0.842	15	0.05	23.4	76.6	98.2	103
13.9	397	1	0.838	3	0.15	17.9	82.1	95.7	100

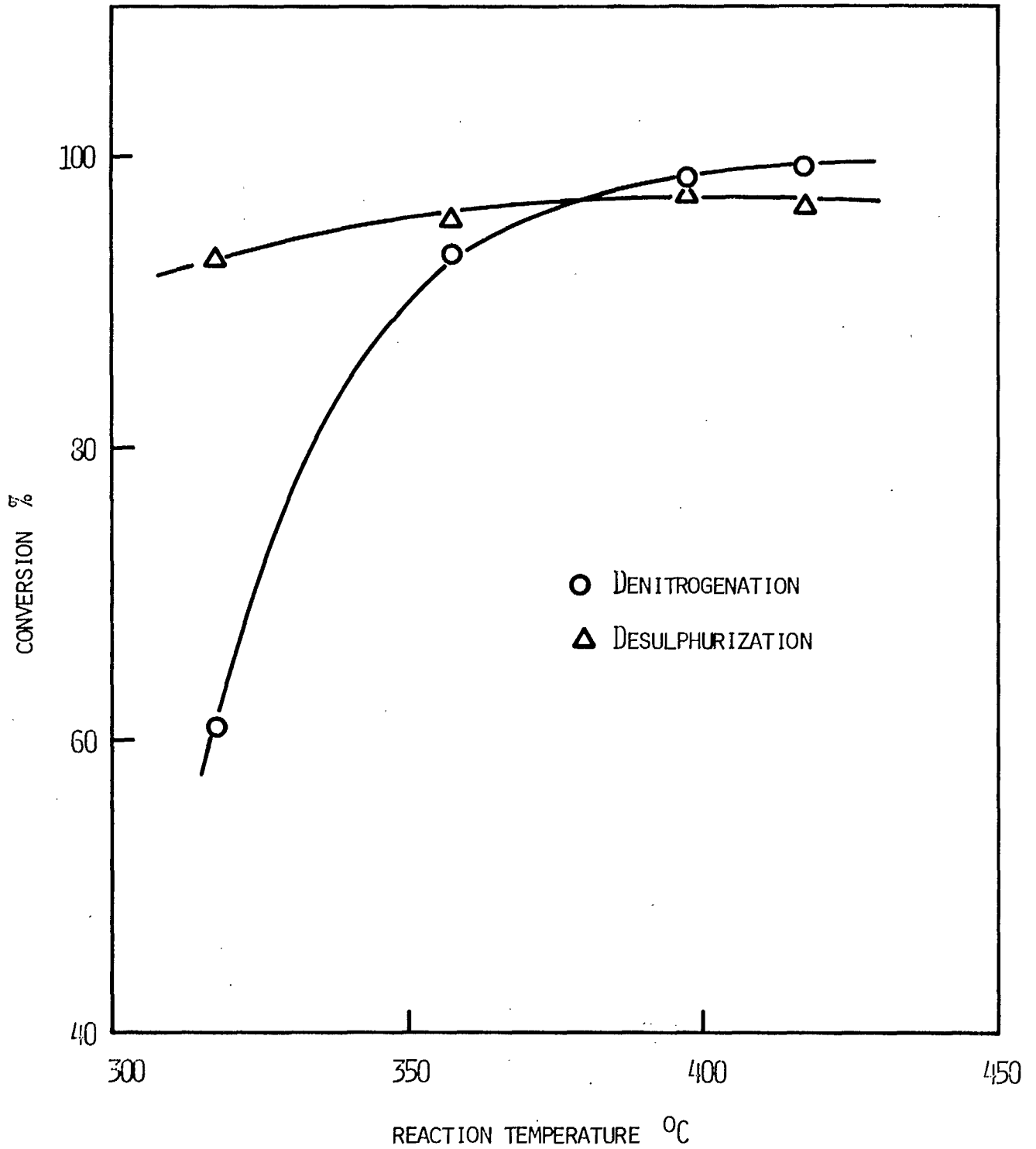


FIGURE 1. DENITROGENATION AND DESULPHURIZATION OF THERMAL-HYDROCRACKER KEROSENE AT 13.9 MPa AND SPACE VELOCITY OF 2.

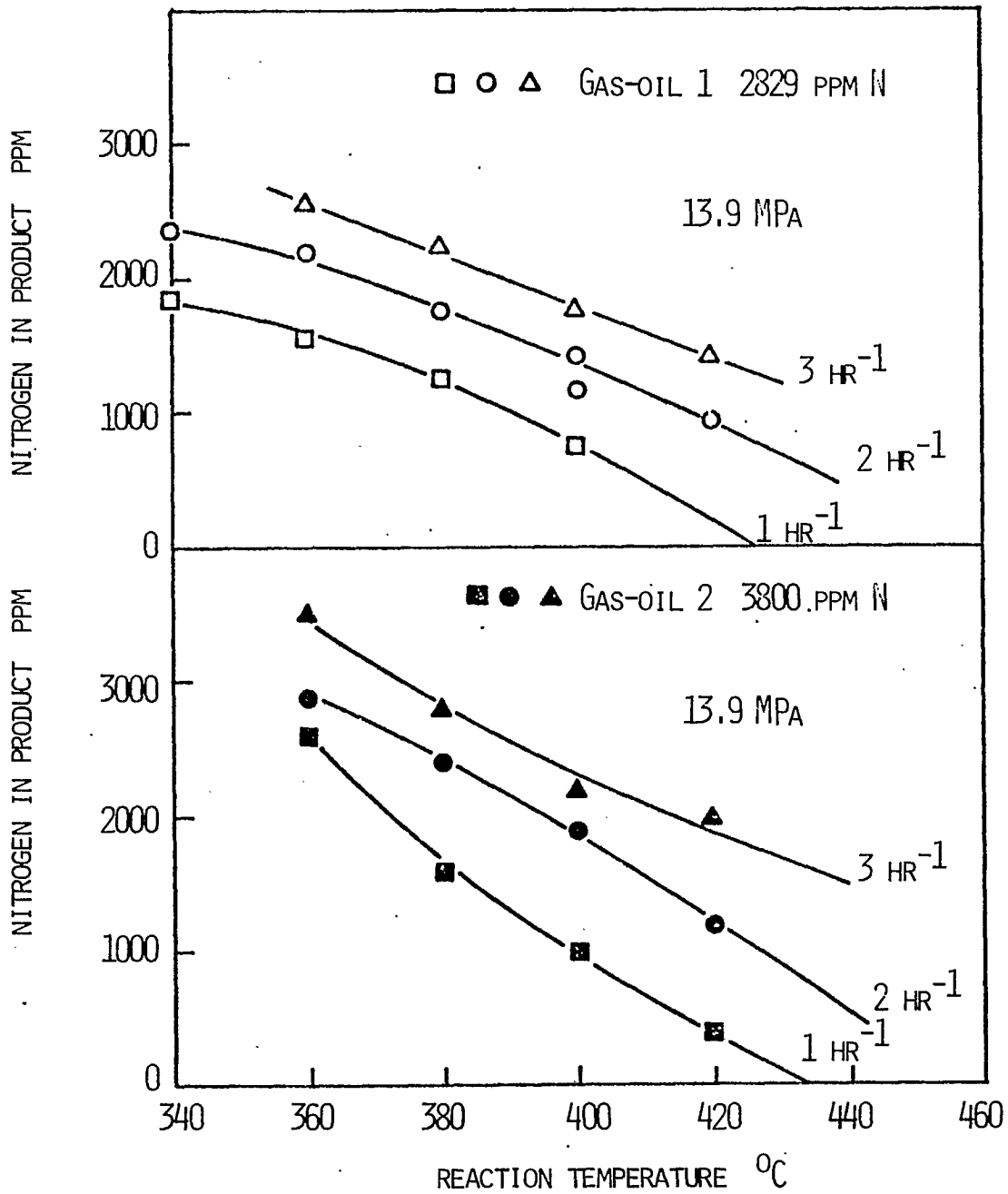


FIGURE 2. NITROGEN IN PRODUCT AT VARIOUS REACTION CONDITIONS FOR TWO DIFFERENT FEED CONCENTRATIONS

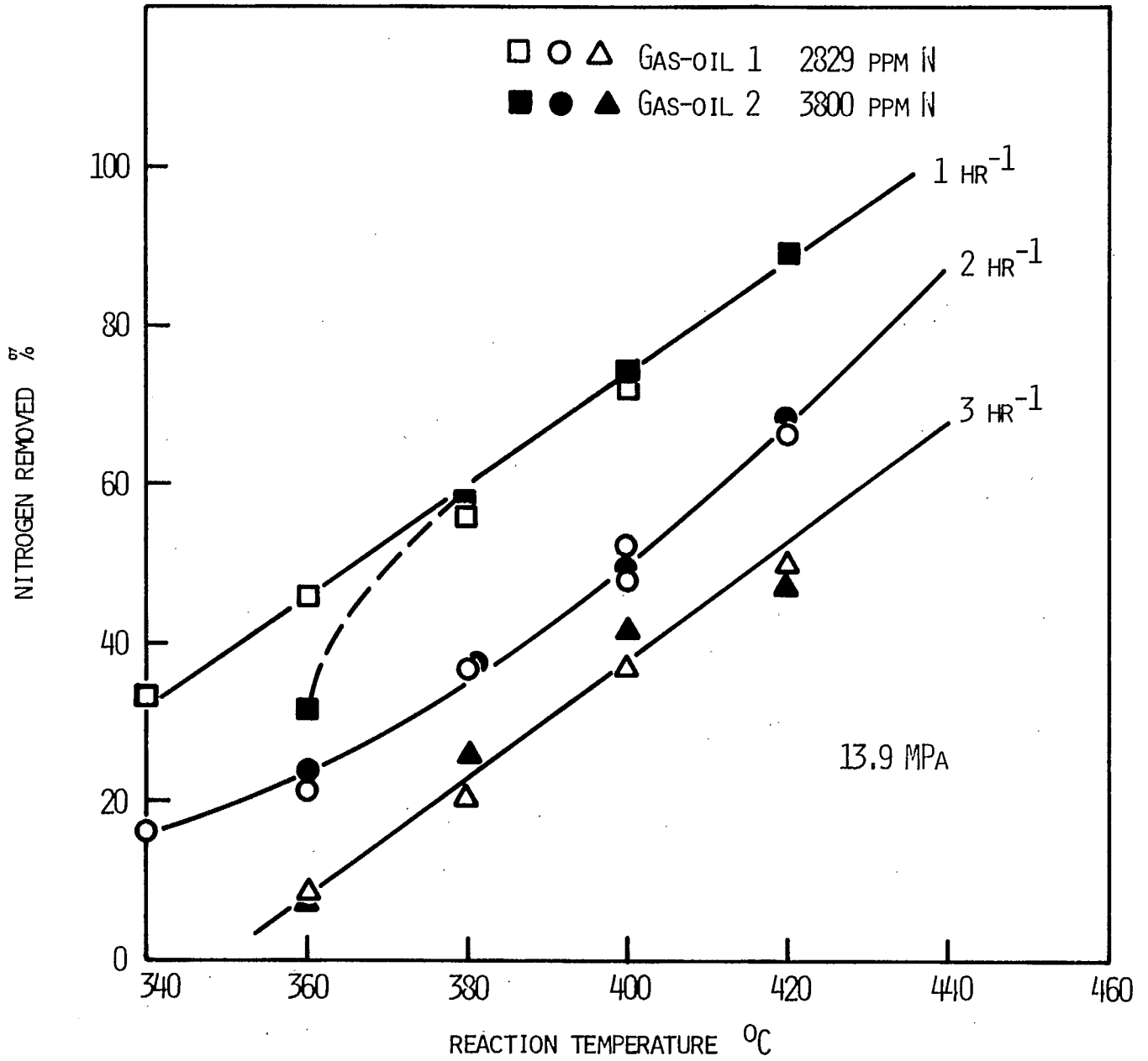


FIGURE 3. EFFECT OF FEED NITROGEN CONTENT ON THE DENITROGENATION OF THERMAL-HYDROCRACKER GAS-OILS.

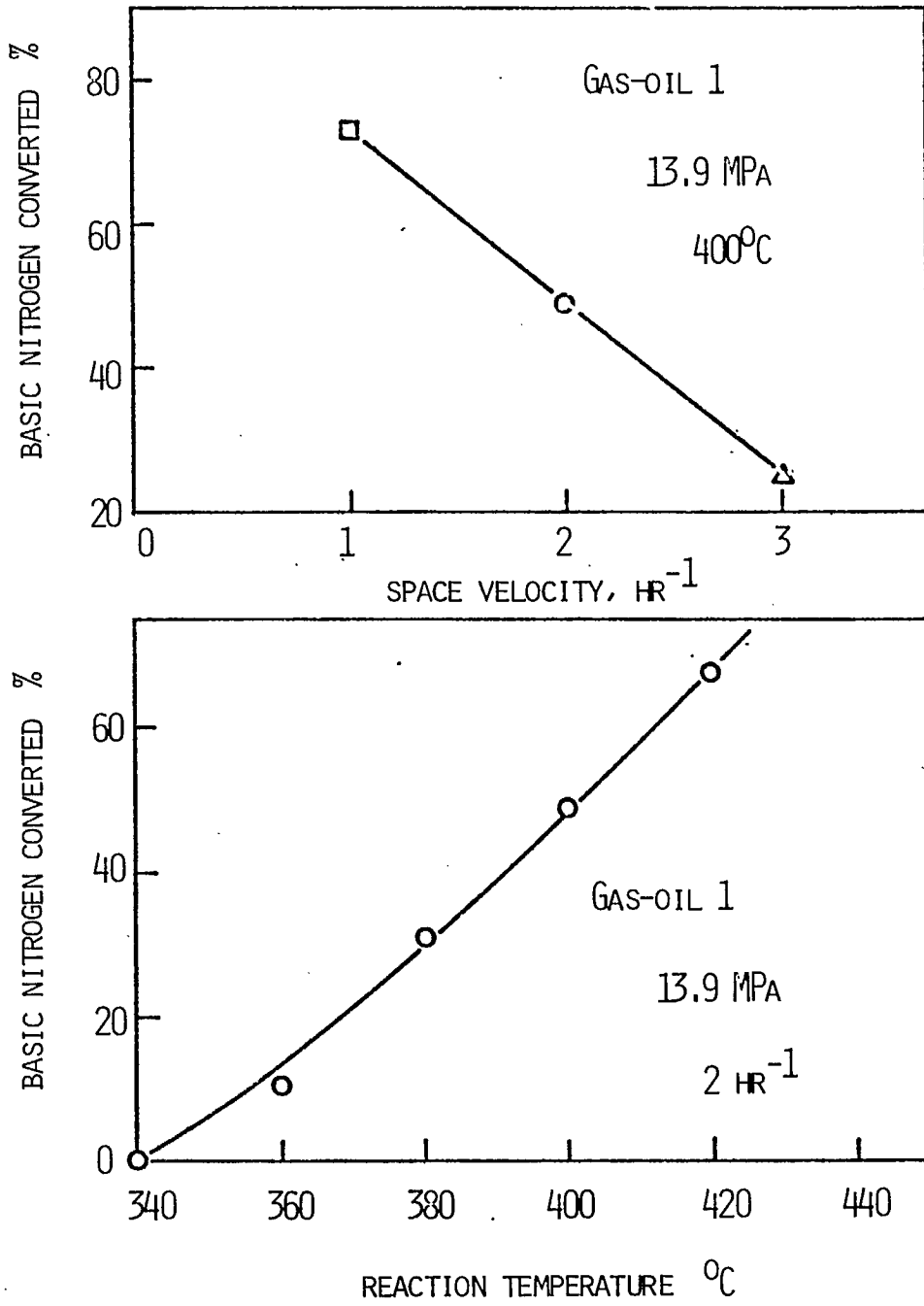


FIGURE 4. BASIC-NITROGEN REMOVAL FROM THERMAL-HYDROCRACKER GAS-OIL.

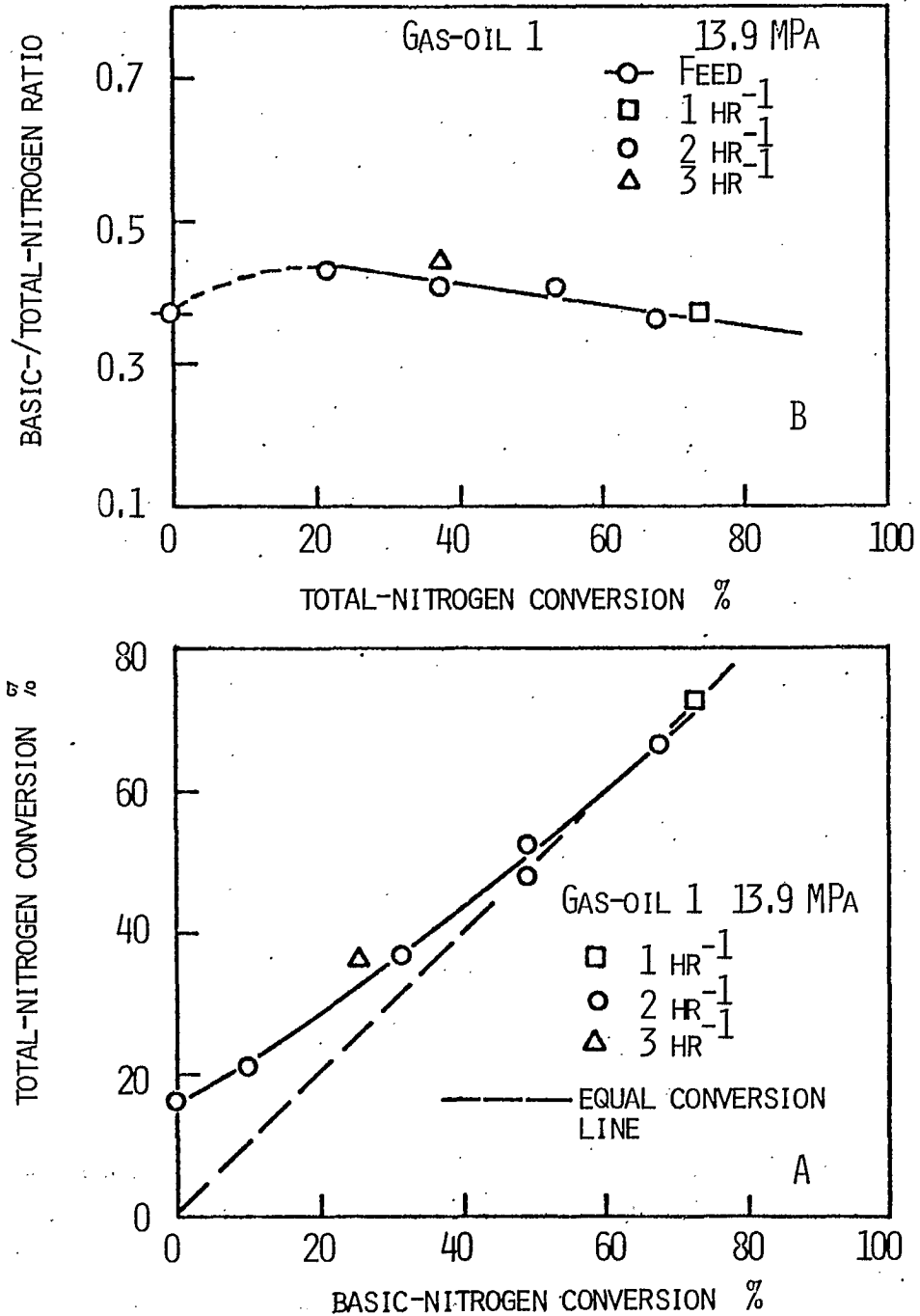


FIGURE 5. RELATION BETWEEN BASIC-NITROGEN CONVERSION, TOTAL-NITROGEN CONVERSION AND BASIC-/TOTAL-NITROGEN RATIO.

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