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Author(s)	Ohtuka, Hiroshi
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Hydro-Cracking of Gas Oil, II.

By

Hirosi Ohtuka.

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In the previous report, the authors studied on the hydro-cracking of gas oil in the presence of molybdenum catalyst. In this paper, the hydro-cracking of the same gas oil with nickel catalyst is studied.

SAMPLE AND CATALYST.

Sample. Siragiku gas oil (Ogura Oil Co.) was used as the sample. Its properties were shown in the previous report.

Catalyst. Nickel oxide on pumice stone was used as the catalyst. 862 g. of bean-size pumice stone was added to nickel nitrate solution containing 1 kg. of this reagent and impregnated with the nitrate by evaporating the solution with constant stirring. The nickel nitrate absorbed on pumice was then converted to nickel oxide by heating the catalyst mass in an electric furnace at ca. 450 deg. C.

APPARATUS AND PROCEDURE.

Apparatus and procedure employed in this experiment were quite the same as described in the previous paper. But in Expt. A-14, thermal cracking of the gas oil was conducted under nitrogen atmosphere without high pressure hydrogen and catalyst.

EXPERIMENT RESULTS.

Table 1. Reaction Conditions.

Exp. No.	A-14	A-15	A-16	A-17
Sample (g.)	250	250	250	250
Catalyst (g.)	0	25	25	25
H ₂ compressed (Wt. %)	—*	7.6	7.5	7.5
Init. press. { (atm.)	0*	54	54	54
{ (°C.)	14	21	20	20
React. press. (atm.)	43*	149	148	121
End press. { (atm.)	16*	48	43	53
{ (°C.)	15	21	19	22
React. temp. (°C.)	450	450	500	400
React. time (min.)	60	60	60	60

Remarks: *A-14 was a thermal cracking expt. under atmospheric N₂.

Table 2. Yields of Produced Oils. (Wt. %)

Exp. No.	A-14	A-15	A-16	A-17
React. temp. (°C.)	450	450	500	400
Yield of total produced oil (%) }	62.2	56.7	21.9	89.6
I. B. P. ~ 97°C. (%)	6.8	11.7	3.6	0.7
97 ~ 120°C. (%)	4.0	6.2	2.8	0.9
120 ~ 200°C. (%)	14.6	15.3	6.8	7.5
200 ~ 300°C. (%)	24.2	19.3	5.4	75.5
300°C. over (%)	4.5	0	0	0
Residue (%)	4.3	4.0	2.9	1.2
Loss (%)	3.8	0.2	0.4	3.8
D. P. (°C.)	360	285	245	295

Table 3. Specific Gravities of Produced Oil. (D_{15}^{15}).

Exp. No.	A-14	A-15	A-16	A-17
React. temp. (°C.)	450	450	500	400
I. B. P. ~ 97°C.	0.6963	0.6972	0.7614	0.7183
97 ~ 120°C.	0.7741	0.7579	0.8207	0.7209
120 ~ 200°C.	0.8439	0.8234	0.8722	0.7786
200 ~ 300°C.	0.9094	0.9080	0.9655	0.8362
300°C. over	1.013	—	—	—
Mean sp. gr.	0.982	0.781	0.902	0.804

Table 4. Volatilities of Produced Gasoline.

Exp. No.	A-14	A-15	A-16	A-17
React. temp. (°C.)	450	450	500	400
I. B. P. (°C.)	29.5	35.0	65.0	63.0
I. B. P. ~ 97°C. (%)	26.8	35.3	27.3	8.1
97 ~ 120°C. (%)	15.7	18.7	21.3	9.8
120 ~ 200°C (%)	57.5	46.0	51.4	82.1

Table 5. Composition of Produced Gasoline. (Wt. %)

Exp. No.	A-14	A-15	A-16	A-17		
React. temp. (°C.)	450	450	500	400		
I. B. P. ~ 97°C.	Unsatur. hydrocarbons (%)	6	0	6	—	
	Arom. "	21	6	34	—	
	Naph. "	11	25	45	—	
	Para. "	62	69	15	—	
	After washing	Aniline pt. (°C.)		66	62	45
	with 99% H ₂ SO ₄ { Sp. gr. (D_{15}^{15})		0.7101	0.7036	0.7408	—
97 ~ 120°C.	Unsatur. hydrocarbons (%)	29	11	23	—	
	Arom. "	38	16	—	—	
	Naph. "	9	25	—	—	
	Para. "	24	48	—	—	
	After washing	Aniline pt. (°C.)		64	62	—
	with 99% H ₂ SO ₄ { Sp. gr. (D_{15}^{15})		0.7374	0.7269	—	—

Table 5. (Continued)

Exp. No.	A-14	A-15	A-16	A-17	
React. temp. (°C.)	450	450	500	400	
120 ~ 200°C. {	Unsat. hydrocarbons (%)	5	6	—	0
	Arom. „	56	44	—	30
	Naph. „	21	34	—	0
	Para. „	18	16	—	70
	After washing { Aniline pt. (°C.)	63	60	—	76
with 99% H ₂ SO ₄ { Sp. gr. (D ₁₅ ¹⁵)	0.7830	0.7815	—	0.7602	

Table 6. Composition of the Fraction above 200°C. (Wt. %)

Exp. No.	A-14	A-15	A-16	A-17
React. temp. (°C.)	450	450	500	400
Unsat. hydrocarbons (%)	9	7	10	0
Arom. „	61	63	—	20
Naph. „	9	11	—	18
Para. „	21	19	—	62
After washing { Aniline pt. (°C.)	78	73	—	89
with 99% H ₂ SO ₄ { Sp. gr. (D ₁₅ ¹⁵)	0.8316	0.8340	—	0.8080

Table 7. Distribution of Unsaturated Hydrocarbons in the Produced Oil. (Wt. of each fraction = 100%)

Exp. No.	A-14	A-15	A-16	A-17
React. temp. (°C.)	450	450	500	400
I. B. P. ~ 97°C. (%)	6	0	6	—
97 ~ 120°C. (%)	29	11	23	—
120 ~ 200°C. (%)	5	6	—	0
200°C. over (%)	9	7	10	0

Table 8. Distribution of Aromatic Hydrocarbons in the Produced Oil. (Wt. of each fraction = 100%)

Exp. No.	A-14	A-15	A-16	A-17
React. temp. (°C.)	450	450	500	400
I. B. P. ~ 97°C. (%)	21	6	34	—
97 ~ 120°C. (%)	38	16	—	—
120 ~ 200°C. (%)	56	44	—	30
200°C. over (%)	61	63	—	20

Table 9. Distribution of Naphthene Hydrocarbons in the Produced Oil. (Wt. of each fraction = 100%)

Exp. No.	A-14	A-15	A-16	A-17
React. temp. (°C.)	450	450	500	400
I. B. P. ~ 97°C. (%)	11	25	45	—
97 ~ 120°C. (%)	9	25	—	—
120 ~ 200°C. (%)	21	34	—	0
200°C. over (%)	9	11	—	18

Table 10. Distribution of Paraffin Hydrocarbons in the Produced Oil.
(Wt. of each fraction = 100%)

Exp. No.	A-14	A-15	A-16	A-17
React. temp. (°C.)	450	450	500	400
I. B. P. ~ 97°C. (%)	62	69	15	—
97 ~ 120°C. (%)	24	48	—	—
120 ~ 200°C. (%)	18	16	—	70
200°C. over (%)	21	19	—	62

Table 11. Produced Gas.

Exp. No.	A-14	A-15	A-16	A-17
React. temp. (°C.)	450	450	500	400
Vol. of produced gas (l./100 g. sample)	9.08	6.99	10.95	1.62
Sp. gr. of produced gas (g./l.)	—	0.92	0.93	0.90
Wt. of produced gas (g./100 g. sample)	—	6.43	10.18	1.46
Wt. increase of active charcoal (g./100 g. sample)	16.10	26.30	43.80	2.00
Amount of total produced gas (g./100 g. sample)	—	32.73	53.98	3.46

Table 12. Amount of Produced Methane.

Exp. No.	A-14	A-15	A-16	A-17
React. temp. (°C.)	450	450	500	400
Amount of produced methane (g./100 g. sample)	—	4.16	6.40	0.94
Vol. % of methane in the produced gas	—	83.3	82.3	83.4
Vol. of produced methane (l./100 g. sample)	—	5.82	8.96	1.32

Table 13. Consumption of H₂. (Wt. %)

Exp. No.	A-14	A-15	A-16	A-17
React. temp. (°C.)	450	450	500	400
H ₂ consumed (%)	—	2.96	4.13	3.32

Table 14. Material Balance.

Exp. No.	A-15		A-16		A-17	
	450		500		400	
React. temp. (°C.)	450		500		400	
Sample	250 g.	97.1%	250 g.	96.0%	250 g.	96.8%
Consumed H ₂	7.4 g.	2.9%	10.3 g.	4.0%	8.3 g.	3.2%
	257.4 g.	100.0%	260.3 g.	100.0%	258.3 g.	100.0%
Produced oil	To 200°C.	84.6 g. 32.8%	34.0 g. 13.1%	32.2 g. 12.5%		
	200°C. over	59.0 g. 22.9%	8.3 g. 3.2%	191.8 g. 74.3%		
	Oil adhered to catalyst	9.3 g. 3.6%	11.3 g. 4.3%	12.6 g. 4.9%		
Produced oil	Light gas	16.1 g. 6.3%	25.5 g. 9.8%	3.7 g. 1.4%		
	Gas adsorbed on active charcoal	65.8 g. 25.6%	109.7 g. 42.1%	5.0 g. 1.9%		
Loss	22.6 g. 8.8%	71.5 g. 27.5%	13.0 g. 5.0%			
	257.4 g. 100.0%	260.3 g. 100.0%	258.3 g. 100.0%			

DISCUSSION OF THE RESULTS.

1. In the hydro-cracking experiments with nickel catalyst, reaction temperature exerted the same influence as in the case of molybdenum catalyst upon the yield of oily products. The maximum yield of hydro-cracked gasoline was obtained at the reaction temperature of 450 deg. C.
2. In each experiment, the hydrogen consumption always amounted to 3~4 wt.% of the sample.
3. The amount of gaseous products, including heavy hydrocarbon gases adsorbed on active charcoal, increased rapidly with the rise of reaction temperature.
4. Comparing the experimental results of this report with those of the previous report, the following conclusions may be derived with regard to the catalytic action of nickel and molybdenum catalysts.
 - (i) Both the catalysts gave the maximum yield of hydro-cracked gasoline at the reaction temperature of 450 deg. C. However, the molybdenum catalyst had a wider optimum temperature range for the production of hydro-cracked gasoline as compared with the nickel catalyst.
 - (ii) In the case of the nickel catalyst, the amount of gaseous products was generally larger than in the case of the molybdenum catalyst, except in the experiment at 400 deg. C.
 - (iii) In short, the nickel catalyst was more destructive in action and more sensitive to temperature than the molybdenum catalyst.
5. Under the same reaction conditions of temperature and time, thermal cracking gave a lower yield of gasoline than hydro-cracking. The thermal cracked gasoline was richer in aromatics and poorer in naphthenes as compared with the hydro-cracked gasoline.

(Fuel Laboratory, Faculty of Engineering,
Hokkaido Imperial University)
