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ENCLOSURE: (F)

REPORT ÓN THE COAL HYDROGENATION PLANT

AT FUSHUN, WANCHURIA

ENCLOSURE (F)

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I. INTRODUCTION

This report records and summarizes technical data on the South Manchurian Railway Co.'s coal hydrogenation plant at FUSHUN, Manchuria, obtained by the Petroleum Section of NewTechJap on 11 and 17 January, 1946. In this report, South Manchurian Railway Co. will be designated by the abbreviation S.M.R.

This plant was not inspected by NavTechJap personnel, since NavTechJap's request, via official channels, to visit the Fushun plant was disapproved in view of the unsettled military conditions prevailing in that area. The information presented was obtained by interview in TOKYO with Mr. T. MIYAMA, who was Technical Director of the Fushun plant from 1937 to 1942, and is now residing in TOKYO as Technical Director of Teikoku Nenryo (Teinen). He has been connected with Teinen since 1942.

The material in this report is based on Mr. MIYAMA's memory, and on memoranda from his personal notes. He stated that he no longer had any official records of Fushun, since those in his possession were burned when his home in TOKYO was destroyed in a fire-bomb raid.

II. HISTORY

Intensive experimentation directed toward establishing the Fushun coel hydrogenation plant began about 1928. At that time, an informal co-operative agreement was made between S.M.R. and the Japanese Imperial Navy's Laboratory at TOKUYAMA, to the effect that both organizations would share research data on coal hydrogenation, with erection of a full-scale plant at FUSHUN as the goal. S.M.R.'s work was done at the Company's Central Research Laboratory at DIAREN, under the direction of Dr. R. ABE, who later became assistant to Mr. MIYAMA at FUSHUN.

Co-operation on this undertaking presumably continued throughout what may be termed, the development period, which ended in 1936. At that time, definite commitments were made toward erection of a plant, the design of which would combine the best festures of the Navy's research and the work at DAIRIN. The Mavy stipulated that all machinery and process equipment must be made in Japan. Accordingly, pressure vessels for converters and separators were ordered from the Kure Navy Yard. The largest of these vessels, which were to become the converters, were 7.3 meters long by 95cm inside dismeter. It is of interest that these vessels, according to Mr. MIYAMA, were the largest of this type which could be made in Japan at that time. Neither annealing furnaces nor ingots were available for the manufacture of larger pieces.

The Fushun plant was erected between 1936 and 1939. The Mavy-type converter, which was to be 7.3 meters long am equipped with a propeller-type stirrer, was not installed because of objections offered by S.M.R. technical advisers. In its place, two of the 7.3 meter vessels were joined end-to-end by bolting, thus doubling the size. The Navy cetalyst (ZnCl₂) was likewise rejected in favor of S.M.R.'s precipitated FeS.

The plant comprised a single converter, liquid-phase system, designed to run on Oyans coal. Preliminary operation began in April, 1939. In November of that year, a second converter was added. Liquid phase hydrogenation was continued until February, 1940, when the addition of a separate vapor phase reactor system (installed at the direction of the Navy) was completed. From this time until July, 1942, the plant operating schedule was divided between liquid-phase coal hydrogenation, and vapor-phase hydrogenation of fractions from low-temperature tar and from the liquid-phase coal process. From July, 1942 until the end of the war, coal hydrogenation was abendoned, at the direction of the

Navy, and full attention was given to the manufacture of aviation gasoline by hydrogracking of gas oil and kerosene.

Throughput capacity of the liquid phase plant was about 10,000 metric tons of coal per year. Mr. MIYAMA estimates that a total of about 15,000 metric tons of product was made during the entire period of the plant's operation, the greater part of which was heavy oil, a primary product from the liquid-phase operation.

III. DESCRIPTION OF PLANT AND PROCESS

A. Liquid Phase System

Plate I(F) is a flow diagram of the liquid phase system for coal hydrogenation. This diagram was prepared by Mr. MIYAMA and his associates for NavTechJap.

The converter originally proposed by the Navy was not used. This converter was to be 7.3 m long, with an I.D. of 95cm and a free folume of 4.5 m. Its outside diameter could not be ascertained. It was to be provided with a propeller-type stirring device, designed to rotate at 200 r.p.m. Each propeller was to have two blades, and there was to be pitch reversal between adjacent propellers. The supporting shart was to pass through both converter heads, which were to be equipped with external packing glands. Results at TOKUYAMA indicated that agitation, such as would be previded by this device, would increase throughput by about 40%.

The technical staff of S.M.R. foresaw a serious leakage problem with every type of packing gland suggested; the staff also believed the converter size should be doubled. Accordingly, the converter which was installed had no stirring apparatus, and the desired size was obtained by bolting together two of the short sections end-to-end at the bedy flauges. Material of construction of the converters could not be determined.

S.M.R. technologists also objected to the use of the ZmCl₂ entalyst recommended by the Mayy. Their own experiences indicated that the use of this entalyst would result in severe corrosion of equipment. They also believed zine to be too expensive for this purpose, since the amount required was 1% of the weight of the coal processed, and recovery from the residue was not economically feasible. They had found a less expensive entalyst, precipitated FeS, to be effective at the same concentration employed for zino. It was agreed to use the S.M.R. catalyst, and Mr. MIYAMA stated that, to his knowledge, no other catalyst was ever employed in the liquid-phase plant. As a matter of record, it was also stated that no form of iron oxide was found to be as effective as ferrous sulfide.

The system for hydrogen manufacture comprised a blue water gas generator, sulfur purification equipment, w water gas shift plant, and a carbon dioxide removal system. The gas generator used high-temperature coke as new material, producing a gas having the approximate composition H_2 , 50%; CO, 40%; CO, 5%; H_2 , 5%. The desulfurizer consisted of a conventional dry iron-oxide box. Although this provided me means for organic sulfur removal, Mr. MIYAMA stated that, after the water shift and scrubbing, the total sulfur content was about 0.02 gm/m².

The shift catalyst comprised a mixture of 95% Fe and 5% Cr, prepared by precipitation from a solution of ferrous and chromic sulfates. This catalyst operated at 540°C. Mr. MIYAMA could not recall the space velocity of reactants over the catalyst, or whether a bulking agent or carrier was used in its preparation. Carbon dioxide was removed by a water wash at 12 atmospheres of pressure.

Ohyama was the only coal processed during the period of Mr. MIYAMA's connection with the plant. He could not recall the analysis of this coal, but stated that it is a pit mine product from FUSHUM, containing about 42% volatile matter and averaging 8-10% ash. He described it as "a very high quality brown coal, approaching bituminous." Possibly it would be classified as sub-bituminous. An analysis of Ohyama coal, obtained from other sources, is presented in Table I(F)

The preliminary steps of coal preparation consisted in grinding the coal to 4 mesh and smaller in a roll crusher, after which, de-ashing was effected in a Dyster system. Ash was thereby reduced to 3-4% in the material charged to the hydrogenation plant.

Paste preparation was operated as a continuous process. The vehicle consisted of about 7% low-temperature tar and 93% heavy oil from coal hydrogenation and was stored at 80°C for feeding to the tube mill mixer. Coal and catalyst were fed to the mill in the wet condition. The coal was charged as the 4 mesh material from the crusher, and, during the mixing operation, was ground to about 200 mesh.

For making a hypothetical 250-ton batch of paste, the following materials would be required (metric tons-dry basis):

COB1 10	ю
Low temperature tar	.0
Heavy oil from coal hydrogenation 12	.5
Catalyst	1
Water (adhering to coal and catalyst)	4
25	_

According to Mr. MIYAMA's verbal statement, paste input to the converter was 60 tons per day. This is not in agreement with the material flow as indicated on Plate I(F), which shows "normal" input to be 4 tons per hour or 96 tons per day. It should also be noted that only one converter is shown on the flow sheet, whereas Mr. MIYAMA had stated that two were in operation after 1939.

Hydrogenation conditions for the liquid-phase system were 200 atmospheres pressure and 4,00°C. The only serious operating difficulty admitted was severe erosion of the valve throttling the discharge of heavy oil, containing ash, from the high temperature separator. A serviceable arrangement, requiring change about once in two months, was found to be a valve with needle tip and seat made of tungsten carbide. The stem of this valve was rotated continuously at about 60 r.p.m.

B. Vapor-Phase System

Figure 1(F) is a flow diagram of the plant modified to operate on vapor-

phase hydrogenation. This system was designed to process about 40 kl of liquid per day at 200 atmospheres pressure over a fixed catalyst. Two converters were in series, the reaction temperature of the first being 430°C and the second, 440°C. The preferred catalyst was a pelleted mixture of 40% MoS3 and 60% acid clay. Charging stock to the vapor-phase plant was either a fraction from low-temperature tar, boiling between 180° and 240°C, or a fraction of similar boiling range obtained from liquid-phase coal hydrogenation. The operating plan for coal hydrogenation work, after installation of the vapor-phase unit, was to run the liquid-phase plant 3 months, storing the product, then shut down and operate 12 months on vapor phase, using the stored product as charging stock.

The overall yield of products obtained from 250 tons of paste, by combining liquid and vapor-phase operation, was said to be as follows (metric tons):

Aviation gasoline	
Cresols and phenol	 3.2
Gasol (C ₃ +C ₄) Pitch coke (from centrifuge	 20.0
residue, contains 50% ash) Recycle oil	 60.0*
Residual (heavy) oil	 65.0*

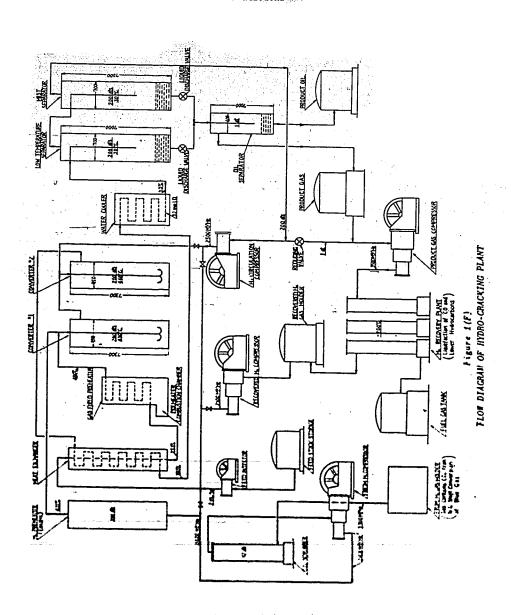
*Returned to paste making.

This represents about 35% by weight, conversion of coal to gasoline, in terms of raw coal charged.

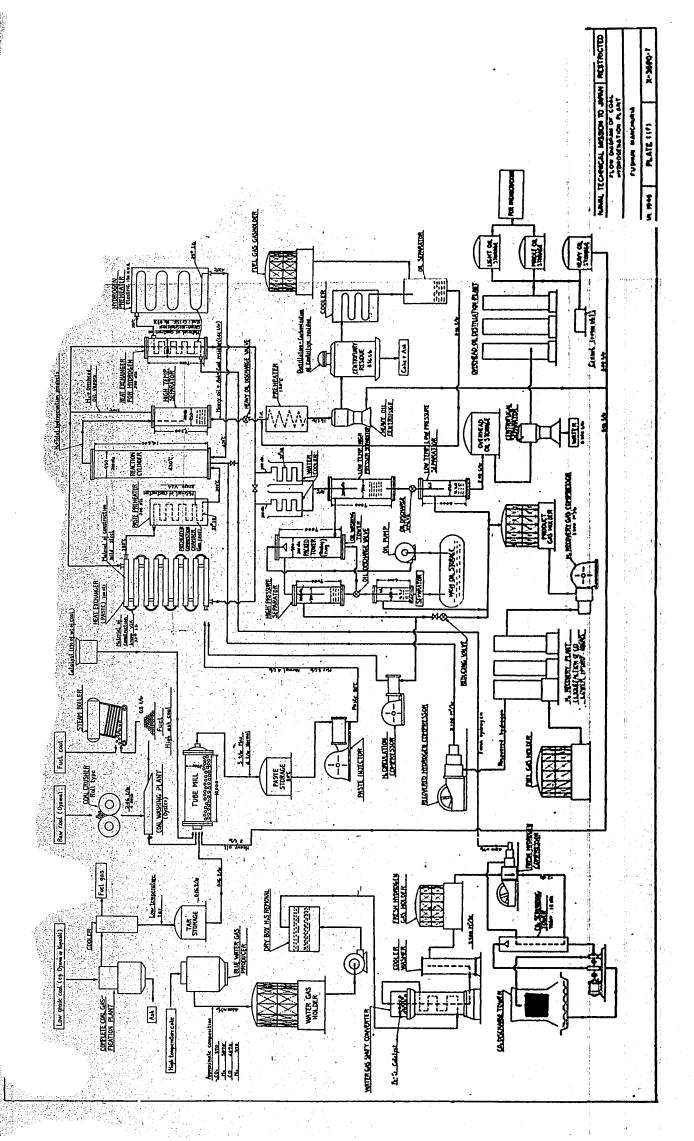
It was maintained by Mr. MIYAMA that, although the Fushun plant was of full scale commercial size, with nominal capacity for processing about 10,000 metric tons of coal per year, it had been regarded as an experimental plant. An experimental operating program may, therefore, partly explain the low total production, which he estimated to be 15,000 metric tons. Most of this product was the heavy oil obtained from the liquid-phase hydrogenation of coal.

Table I(F)
ANALYSIS OF CYAMA COAL

Proximate				Ultimate					
H20	Ash	Volatile Matter	Fixed C	Heating value cal/gm	C	H	0	8	M
5.5	7.0	41.1	46.4	7200	73.7	5.7	12.0	0.5	1.2



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