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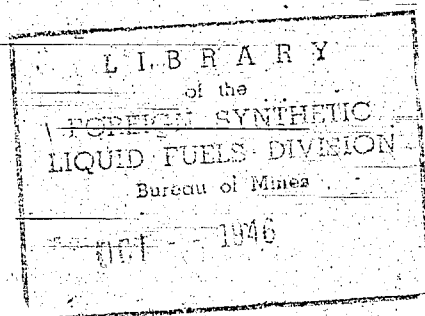
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by Col. R. H. Evans
E. W. L.

FISCHER TROPSCH
AND ALLIED PROCESSES

Strochkin, N.
Lowdermilk, F. R.



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COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE

RESTRICTED

FISCHER TROPSCH AND ALLIED PROCESSES

26 June 1945

Reported by:

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31 July 1945

CIGS Target Nos. 30/6.01, 30/5.01, 22/1264, 22/196
Miscellaneous Chemicals
Fuels and Lubricants

COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE
G-2 Division, SHAEF (Rear) APO 413

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FISCHER TROPSCH AND ALLIED PROCESSES

KAISER WILHELM INSTITUTE FUER KOHLEFORSCHUNG MUELHEIM/RUHR

1. INTRODUCTION.

The Kaiser Wilhelm Institute was visited on 25 June 1945. Dr. H. Pichler, Assistant Director, and Dr. H. Koch, Department Head, were interviewed and all information received came from them.

The Institute was founded by leading industrialists of the Ruhr, principally coal operators, in 1912. In the same year the Director was chosen in the person of Dr. Franz Fischer, Prof. of the Royal Inst. of Technologie, Berlin. During the early part of 1913 the plans for the buildings were approved and, in June, 1914, the whole project was completed. It included a modern laboratory building with adequate shops, to which later equipment for high pressures and low temperatures were added together with pilot buildings and installations. The official opening took place in 1914 shortly before the outbreak of World War I.

2. ORGANIZATION.

Director.....Dr. Franz Fischer, 1912-1943
Dr. Karl Ziegler, 1943 - date
(residing at Halle, at present)

Dept. Heads.....Dr. H. Tropsch, 1921-1928 (died 1935)
Dr. H. Koch, 1927 - date (Dept. head since 1935)
Dr. H. Pichler, 1927 - date (Dept head since 1936, Asst. Dir. since 1943)

3. SCIENTIFIC DEVELOPMENTS.

Conforming to the wishes of the founders, Fischer started his work with the object of providing improved and new methods for utilization of coal in manufacture of coke, tar, oils, and gas. A treatise on coal tar distillation was the result of these first efforts. During the early twenties Fischer turned to the utilization of water gas with the following developments to his credit:

<u>YEAR</u>	<u>PROCESS</u>	<u>PRESSURE ATM.</u>	<u>CATALYST</u>	<u>DEVELOPED BY</u>
1922	Synthol. Oxygen cont. derivatives of hydrocarbons	100	Fe, CO, Ni	Fischer- Tropsch
1923	Synthane: Hydrogenated synthol	-	-	" "
1925	Benzine, some olefines	Normal	Fe, CO, Ni	" "
1936	Benzine, little olefines	5-20	CO	Fischer- Pichler
1937	" much olefines	5-20	Fe	" "
1938	Solid paraffins high melting	over	Ru	" "
1941	Iso paraffins	100 over		" "
1942- 43	Naphthenes, aromatic, formic acid, fatty acids, alcohols, substitu- tion of iron in catalysts for metals becoming scarce during the war.	100	Ru	" "

4. LABORATORY AND PILOT PLANT EQUIPMENT OF NOTE.

The pilot plant contains equipment for production and storage of water gas and synthesis gas. A storage tank for synthesis gas at 1000 atm was 1' diameter, 12' length. The converter units are 3" diameter by 10' length and water cooled. Compressors are installed for compression up to 1,000 atm. There are also units for catalyst testing by flow and static methods. Other equipment included:

Laboratory distillation columns with revolving ribbon agitator.

Furnaces with temperature control maintaining 10C.

High pressure equipment built by Andreas Hofer, mechanic of the Institute.

Liquid air plant consisting of 4-stage compressor was built by Hofer with a capacity 0.7 liters of liquid air per hour; precooling with water raises the capacity to 14 liters per hour. Precooling by refrigeration increases the capacity still higher.

5. COMMERCIAL DEVELOPMENTS.

In 1934 Franz Fischer believed that the time was ripe to carry on large pilot plant and commercial developments of the processes of the Kaiser Wilhelm Institute and he put the matter before its governing body. This body subsequently asked a number of industrial concerns for proposals and bids. It was only natural that the bid of the Ruhr-Chemie A.G. was accepted. This concern was founded in 1928 by the principal coal operators of the Ruhr and adjacent districts for the

purpose of exploitation of processes and products incidental to the coal industry. The fixation of nitrogen was already carried out by this concern on a very large scale based on watergas and coke oven gas for the production of synthetic ammonia, nitric acids, and nitrates. As the result Ruhrchemie became the exclusive licensee of the Fischer-Tropsch and allied processes all over the world.

RUHR CHEMIE A.G., HOLTEN-STERKRADE.

1. GENERAL.

Ruhr Chemie is owned by approximately 80% of the coal and coal tar producers of the Ruhr represented by the Ruhr Kohlen Syndicate. It was organized about 1928 for the purpose of chemical exploitation of coal and coal products.

a. Organization.

Prof. Dr. F. Martin	Managing Director (Chemist)
Dr. Hageman	Director (Chemist)
Dr. Paul	Director (Mech. Engineer)
Mr. Weibell	Director (Business Mgr)

After the paralyzing bombing attacks of October and November, 1944, the German government requested evacuation of the managing personnel as well as some of the equipment and the more important documents and records. The latter were shipped to Reelkirchen (near Detmold). A few technicians in subordinate positions remained at Holten-Sterkrade. Among these were Dr. Spanier, one of the superintendents of the Nitrogenous Products Department; Dr. Lehreiber, Research Chemist, engaged in dehydrogenation work; Dr. Landgraf, General Manager of a subsidiary, Oxo Gesellschaft m.b.H., and Mr. Meby, Power Plant Engineer. Dr. Paul and Mr. Weibel, both directors, returned to the plant on or about June 20th under instructions from their principal to take any steps necessary for the rehabilitation of the plant. Both were interviewed on June 23rd.

b. Plant General.

(1) Estimated Value of Plant.

Buildings -	RM 20,000,000
Equipment	100,000,000
Total	120,000,000

(2) Number Employees.

Administration	200
Technical	350
Skilled Labor	700

Unskilled Labor	780
Control Staff	100
Research	150
Total	<u>2280</u>

(3) Principal Products.

(a) Nitrogenous Product (expressed as N_2) 55-60,000 tons/year representing 5% of Germany's prewar production.

(b) Hydrocarbon Products:

Paraffin	6000 tons/year	
Lubricating Oil	6000 "	"
Diesel Oil	19000 "	"
Benzine	25000 "	"
Propane/Butane	6000 "	"
Total	<u>62000</u> "	"
		corresp to 3-4% of Germany's production
Catalyst production:	900 "	"

(4) Power.

50% self generated, 50% purchased at RM .02/KWH

Fuel: 222,000 tons/year cost RM 11-12/ton

Water: From Rheinisch-Westphaelische Water Works -
RM .06-.07/cbm. Hardness-12°

Steam Consumption: 1,500,000 tons/year at 18 and 35 atm
Cost - RM 13/tons

c. Historical.

The first plants to be built by Ruhr Chemie after its organization were the synthetic ammonia and ammonia oxidation plant, of which the first units were completed in 1930. Linde plants for manufacture of the necessary nitrogen with oxygen as a by-product were built at the same time.

In 1935, after obtaining exclusive license for all processes originating at the Kaiser Wilhelm Institute, constitution of the first Fischer Tropsch Unit was started and completed during the following year. At the time of the bomb attacks, which closed down production the following plant units were in operation:

(1) Complete nitrogenous products plant manufacturing synthetic ammonia, nitric acid and nitrogenous fertilizers.

(2) Hydrogen plant From coke oven gas.

(3) Linde plant for manufacture of nitrogen with oxygen as a by-product.

(4) Complete low pressure Fischer Tropsch plant for manufacture of benzine and Diesel fuel.

(5) Complete medium pressure synthesis plant for manufacture of olefins.

(6) Complete lubricating oil plant for manufacture of lubricating oil from olefins.

(7) Complete plant for manufacture of catalyst for own and licensee's use.

In addition to these production units, there were a number of operating pilot plants for the following processes:

(1) Manufacture of benzine with high octane rating.

(2) Manufacture of isoparaffins directly from synthesis gas.

(3) Isomerization of paraffins.

(4) Substitution of the cobalt catalyst by an iron catalyst.

d. Bomb Damage.

Bombing attacks during October and November, 1944, put an end to operations. Hardly a building was left undamaged. The equipment suffered less and some of it was evacuated on order of the government. The bomb damage is estimated at RM 46 - 50,000,000. Plans have been made for the resumption of operations on a limited scale. Provided that power and raw materials could be made available, the management indicates they could operate partially:

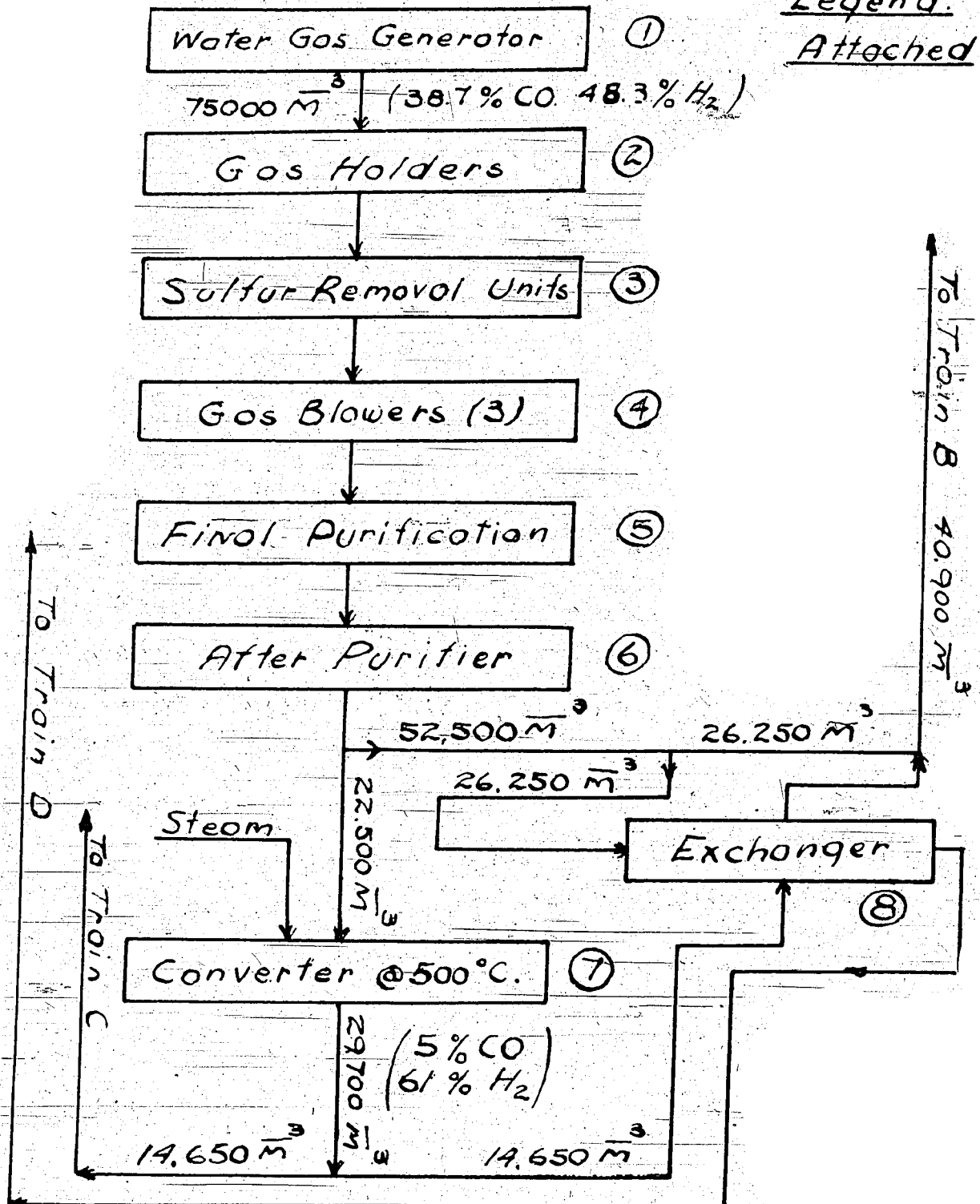
Oxygen and Nitrogen plant	- within 1-2 weeks
Nitrogenous compound plant	- " 4 months
Hydrocarbon plant	- " 6-8 months

2. PROCESSES.

a. Water Gas, Normal, Purified, and Hydrogen Enriched.
(Reference: Flow Sketch No. 1)

Water gas containing 38.7% CO and 48.3% H₂ was generated in the usual manner from steam and coke and was stored in gas holders. Sulfur

Legend:
Attached



FLOW SKETCH NO. 1 - TRAIN A
WATER GAS PURIFICATION
HOURLY FLOW RATES

LEGEND FOR TRAIN A.

- (1) Water gas generator, capacity 75,000 cu m/hr gas of 38.7% CO, 48.3% H₂.
- (2) Gas holders, each 20,000 cu m capacity.
- (3) Sulfur Removal Units, 8 towers (2 as spares), each packed with 60 T luxmasse, capacity 6 towers: 80,000 cu m/hr.
- (4) Gas Blowers (3), 1 @ 80000 cu m/hr, 2 each @ 40,000 cu m/hr gas handling capacity.
- (5) Final Purification Unit, 10 towers, gas heated to operate @ 200-250°C, with series flow through a pair of towers; towers each packed with 60 T of special material (70% luxmasse, 30% Na₂CO₃) and each of 10,000 cu m/hr gas handling capacity.
- (6) After Purifier, 2 towers, operating @ 190°C, parallel flow, each of 40000 cu m/hr gas capacity.
- (7) Converter, steel construction, for conversion CO to CO₂ H₂, using steam and I.G. catalyst (FeO, MgO, etc.) placed on 4 shelves. (Catalyst Life - 2 yr).
- (8) Gas to Gas Heat Exchanger

removal was effected by passing the gas in parallel flow through 6 towers, packed with luxmasse; two additional towers were held in reserve. The 6 towers in run have a purification capacity for 80000 cu m/hr of water gas. Gas from the towers entered booster blowers, 2 in run and 1 spare, each with a capacity of 40000 cu m/hr, and was delivered to a final purification unit consisting of 10 towers, each packed with 60 T of a mixture of 70% luxmasse and 30% anhydrous Na₂CO₃. These latter purification towers operated at 200-250°C (gas fired) with series flow through a pair of towers. Capacity per pair is 20,000 cu m/hr; 1 pair remains in stand-by condition. The gas next entered the final purification unit, which consists of 2 towers operating in parallel at 190-195°C and packed with reduced metals such as Fe, Cu, Ni, etc. This reduced sulfur content to 0.1 gr per 100 cu m of "synthesis gas." Capacity of the 2 towers is 80,000 cu m/hr of gas.

Gas from final purification was partly used without further treatment for blending with hydrogen enriched gas to form "synthesis gas."

Hydrogen enrichment was effected by subjecting a part of the normal purified water gas to the water gas reaction in a converter, steel construction, operated at 500°C and less than one atm pressure. The catalyst in this converter was a special I.G. type (Fe O, Mg O, etc.) held on 4 trays; its normal life is 2 years.

b. Normal Pressure Synthesis.
(Reference: Flow Sketch No. 2, Train B).

The "synthesis gas" fed to this operation was obtained by mixing normal purified water gas with hydrogen enriched gas to effect a $H_2:CO$ ratio of 2:1 and was conditioned by diverting a portion in a gas to gas heat exchanger prior to entrance to converter (1).

Converter (1), all steel, is of box-type construction (See Fig 1) water cooled, and operated at $200^\circ C$ ($1^\circ C$) and 0.2-0.3 atm gauge.

It was packed between the vertical divider plates with 1-3 mm size particles of catalyst of following composition:

CO (as metal)	30%
ThO_2 , MgO mixture	10%
Kiesel guhr	60%

Gases from converter (1) were cooled by water spray directly into a tower, which resulted in condensing an oil of $150-360^\circ C$ boiling range from the gas stream. The oil and water layers were continuously withdrawn at this point. The non-condensed vapors then flow through a steam-heated, funnel heater to preheat them to $180^\circ C$ prior to entering second converter (4) which was packed with same catalyst as in (1). Operating temperatures were raised from 180 to $200^\circ C$ over the life of the catalyst, but were held within $10^\circ C$ during conventional operation. Synthesis of a large portion of the residual gas into oil products was done here. A second condensation and separation of oils was carried out in condenser (5) in the same manner as above.

The remaining gases passed through towers packed with activated carbon which absorbed low molecular weight oils, C_3 and C_4 hydrocarbons, equal to 3% of the weight of the carbon, from the gas stream. Towers operated on stream in $1/2$ hour cycles; during the off-stream cycle adsorbed materials were stripped by steam blowing the carbon. The residual gases were used as fuel.

Product was largely straight chain paraffins, with a large quantity of liquid, and small quantities of gases and solids.

c. Medium Pressure Synthesis.
(Reference: Flow Sketch No. 3, Train C)

This process operated at 11 atm abs on a synthesis gas with a $H_2:CO$ ratio of 2:1, but portions of the hydrogen rich gas were introduced between the first and second converters and between the second and last converters. This process may be operated with either Co or Fe catalyst; Co catalyst was used at Ruhr Chemie. With Co the operating temperatures were $190-200^\circ C$ and the product distribution fairly equal in both liquids and solids. With iron catalyst, the operating temperatures were $225-240^\circ C$ and the products were largely liquids with small quantities of solids and gases and more unsaturates than were obtained with Co.

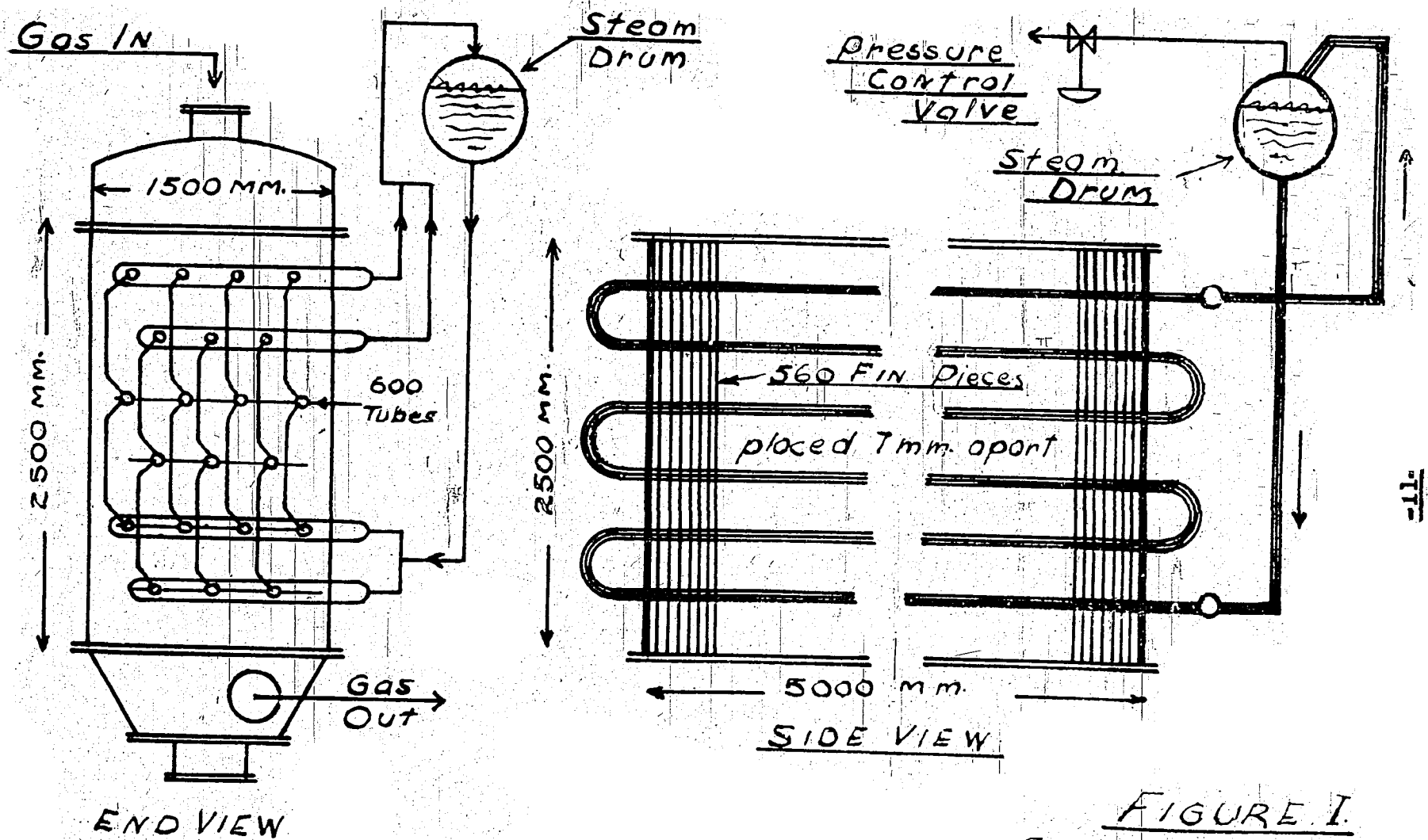
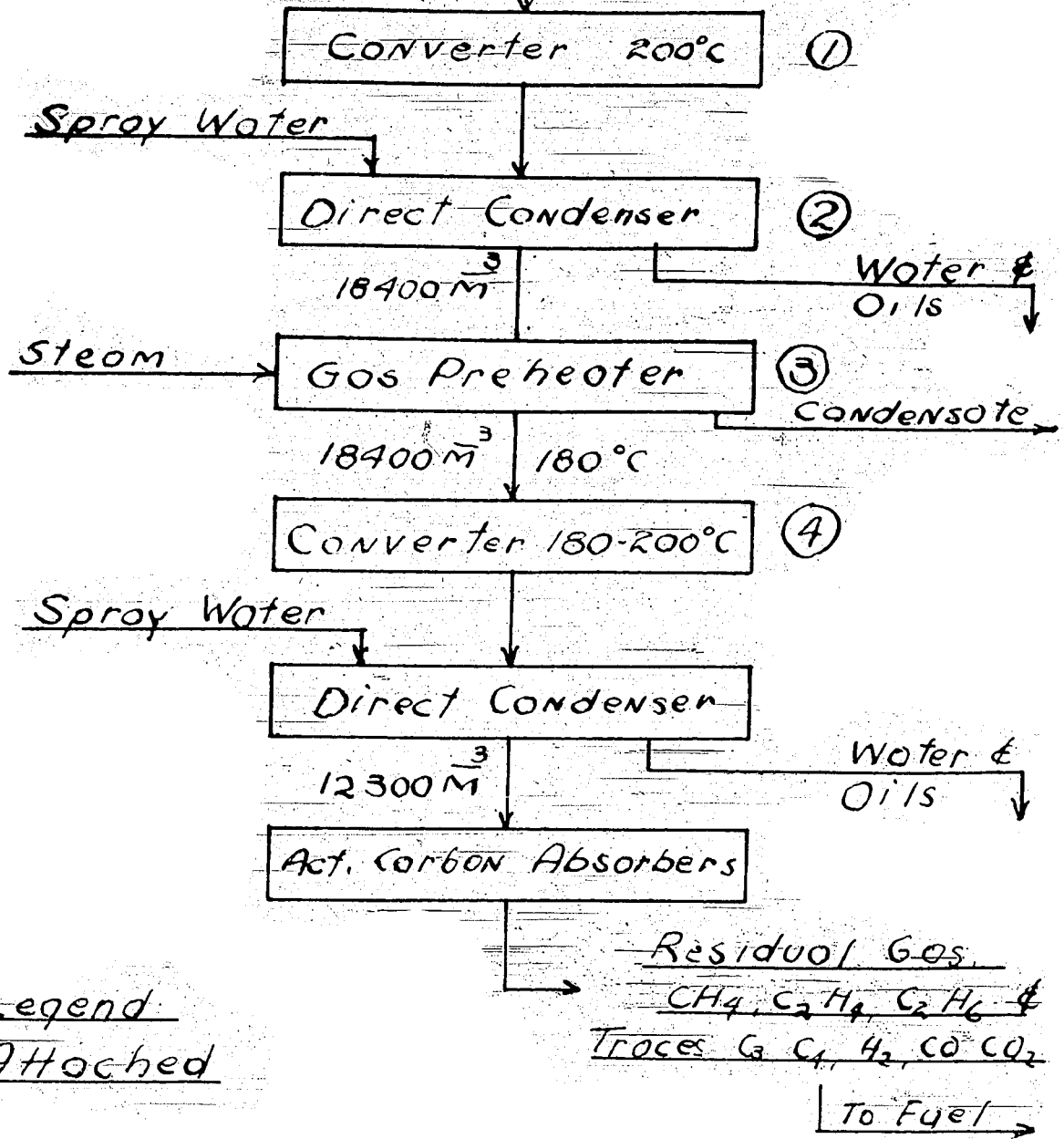


FIGURE I.
CONVERTER
NORM. PRES. SYNTHESIS

F.R.C. 7-23-48

From Train A
40900 m³ Syn. Gas
(26.5% CO 53.0 H₂)

Pressure:
0.2-0.3 atm. gage



Legend
Attached

FLOW SKETCH No. 2 — TRAIN B
NORMAL PRESSURE SYNTHESIS
HOURLY FLOW RATES

LEGEND FOR TRAIN B. (p. 12)

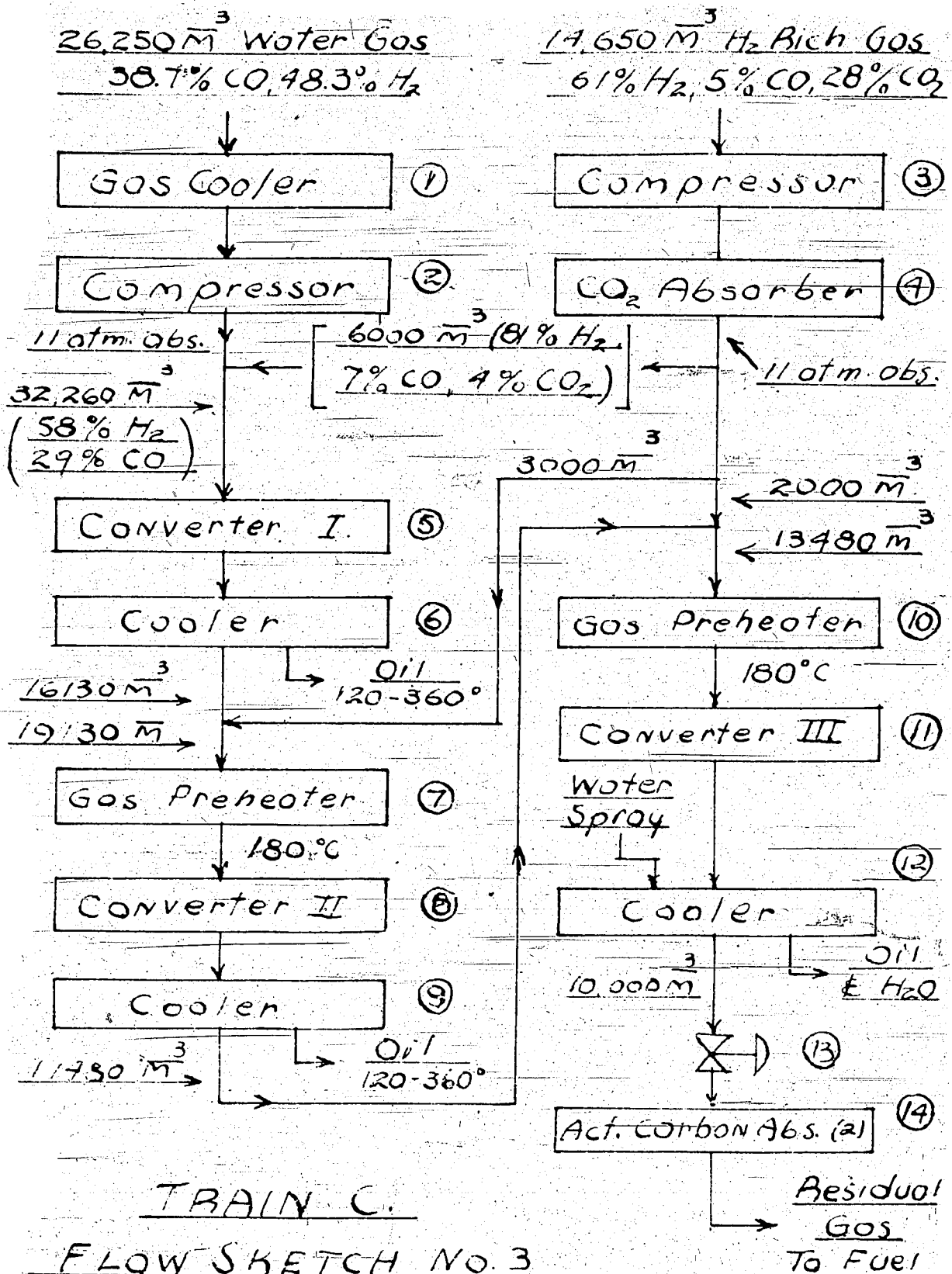
- (1) Converter I, packed with catalyst consisting of 30% Co (as metal equivalent), mixture of 10% ThO₂ and MgO, 60% Kieselguhr. and water cooled for close (1°C) temperature control.
- (2) Condenser, direct water spray type.
- (3) Preheater, tubular, finned, condensing steam in tubes.
- (4) Converter II, construction and catalyst same as in item (1).
- (5) Condenser, same as in item (2).
- (6) Absorbers (2) packed with activated carbon, cyclic operation, followed by steam stripping of adsorbed oils from carbon.

LEGEND FOR TRAIN C. (p. 14)

- (1) Gas cooler, for water gas portion of feed.
- (2) Gas compressor, discharging @ 11 atm abs.
- (3) " " , for hydrogen rich portion feed gas.
- (4) CO₂ Absorber, (CO₂ absorbed in water under pressure).
- (5) Converter I, Tubular type, packed with catalyst consisting of 30% Co (as metal equivalent) 10% as a mixture of ThO₂ and MgO, 60% Kieselguhr, and water cooled with water for close temperature (1°C) control. (See Sk converter for medium pressure synthesis.
- (6) Cooler and condenser, shell and tube, gas in tubes, water in shell.
- (7) Gas preheater, shell and tube, steam in shell.
- (8) Converter II, same as item (5) packed with same catalyst.
- (9) Cooler and condenser, same as item (6).
- (10) Gas Preheater, same as item (7).
- (11) Converter III, same as item (5), packed with same catalyst.
- (12) Cooler and condenser, direct type, employing water spray counter to gas flow in vertical tower.
- (13) Expansion valve (reduction pressure to 1 atm abs)
- (14) Activated carbon absorbers (2), operating in 1/2 cycles to adsorb as 3% of wt of carbon, oil constituents, which are desorbed by direct steam blowing during off-run cycle.

.....

Cooled water gas entered gas compressor (2) and was discharged at 11 atm abs to a mix-tee where hydrogen rich gas from compression and CO₂ removal units (3) and (4) was admixed to produce synthesis gas for Converter (1), where reaction was effected with conversion to the extent of 50% at 180-200°C, depending upon age of catalyst. This converter is constructed of steel, shell and tube design, water cooled, with special annulus - type catalyst holding tubes (see Fig. 2). Water cooling is used for a temperature control of 1°C. Catalyst of size and composition described in (b) above, was packed in the annular



TRAIN C.

FLOW SKETCH NO. 3

PRESSURE SYNTHESIS

HOURLY FLOW RATES (N.T.P.)

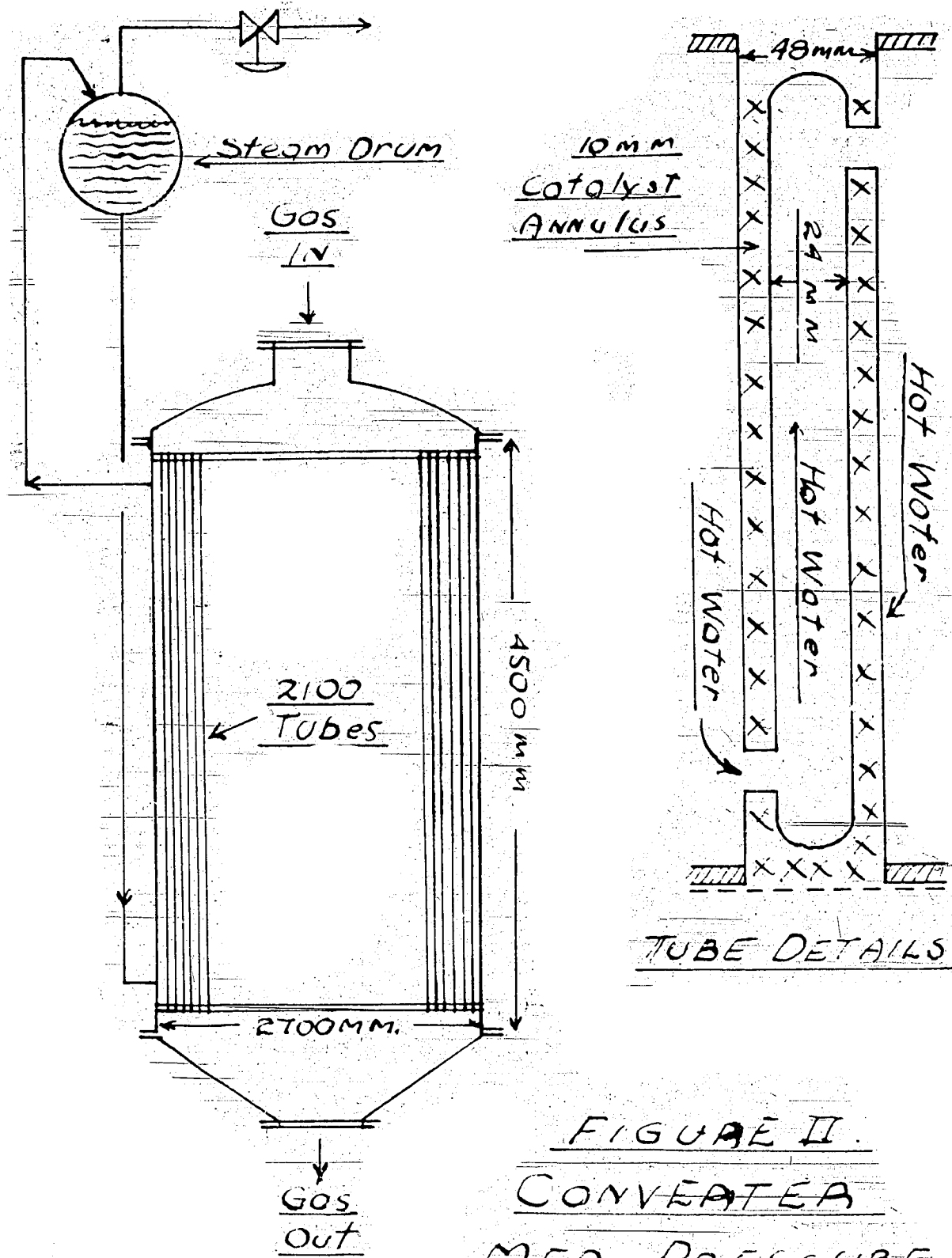


FIGURE II.
CONVERTER
MED. PRESSURE
SYNTHESIS

section of the double concentric tube. All converters were of identical construction and were packed with same type catalyst.

Oil products of a boiling range 120-360°C were condensed from gas from Converter (1) in an indirect Condenser (6), the condensed oil layer being continuously withdrawn. Gases from Condenser (6) were fortified with additional hydrogen rich gas and the mixture then preheated to 180°C in steam heated preheater (7) prior to entering Converter II.

Converter II also was operated over the temperature range of 180-200°C to effect a 30% conversion of feed gas. Oil product of boiling range 120-360°C was condensed from the gas stream in condenser (9) from which it was continuously withdrawn. Exit gas was again fortified with additional hydrogen rich gas and the mixture preheated to 180°C in preheater (10) prior to entering Converter III.

Converter III was operated similarly to the other converters to effect a 21.5% conversion of feed gas following which cooling, condensation, and oil separation steps, as previously described, were made.

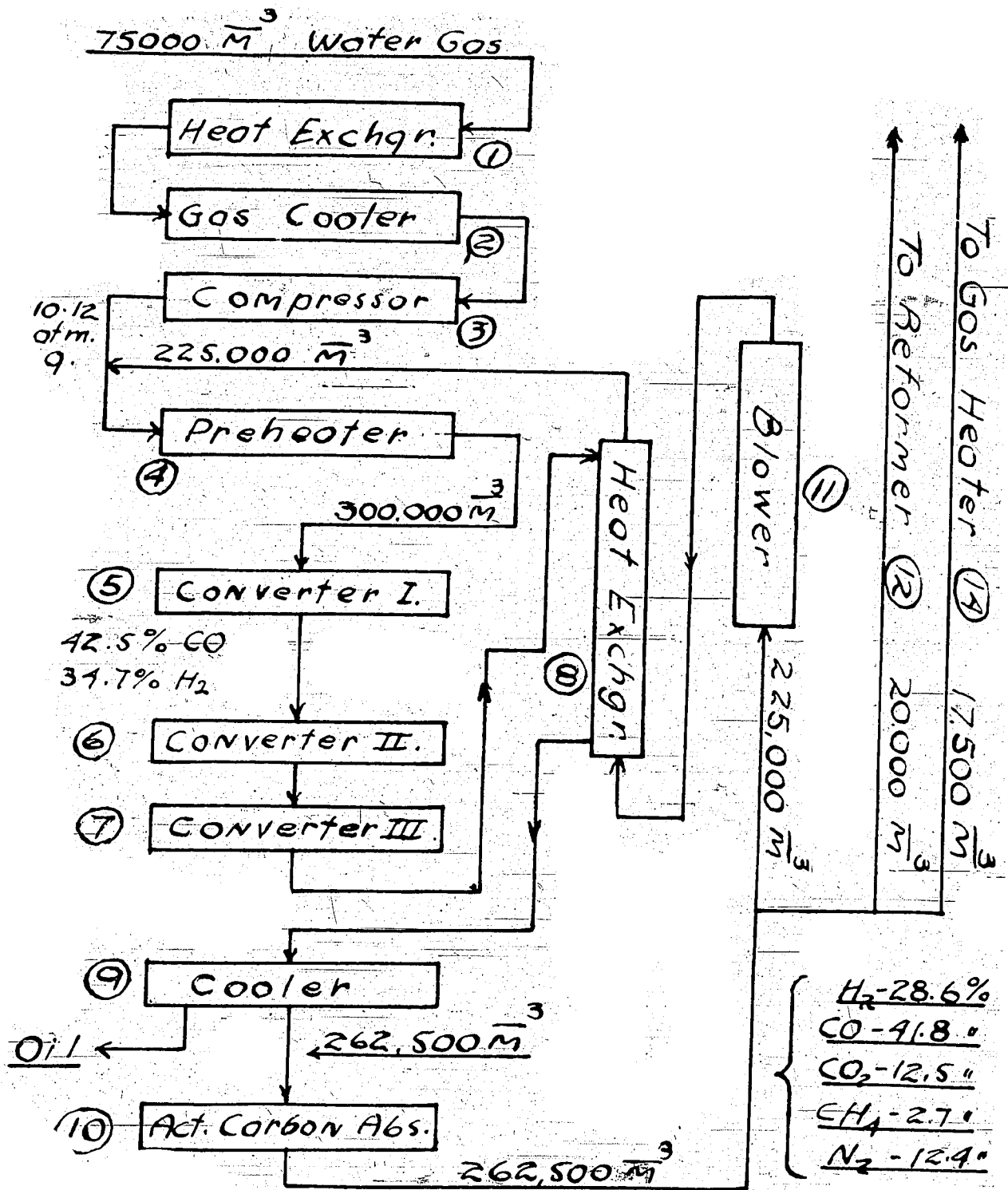
Gases from the final cooler (12) passed through a reducing valve to cyclic-operating activated carbon absorbers, operated at atmospheric pressure, where low molecular weight oils and C₃, C₄ hydrocarbons were adsorbed, then desorbed by stream stripping as described (2) above. Residual gases were used as fuel.

d. Olefin Synthesis.

(Reference: Flow Sketch No. 4, Train D)

In this synthesis purified water gas was the feed stock, but the addition of three volumes of recycle gas to the feed gas prior to the conversion step resulted in a converter feed gas containing H₂: CO in the ratio of 1.00:1.23 together with CO₂, CH₄, N₂. Two converter trains were used, the first with three converters was operated at 10-12 atm gauge; the second was a conventional normal pressure train of two converters, operated at 1 atm gauge and 190-200°C and was employed to complete the synthesis from the residual gases from the first train. Catalysts in both trains were the cobalt type. The products from the first converter train contained more unsaturates and considerably less solid hydrocarbons than those from the second train (a normal pressure synthesis train, using CO catalyst).

Purified water gas was fed to the system through gas to gas heat exchanger (1), through gas cooler (2) and into suction of compressor (3) which discharged at 10-12 atm gauge to a mix-tee where 3 volumes of recycle gas mixed with the compressed water gas. The combined gas streams were preheated to 195-200°C in gas heater (4) and flowed in series to and through Converters I, II, III.

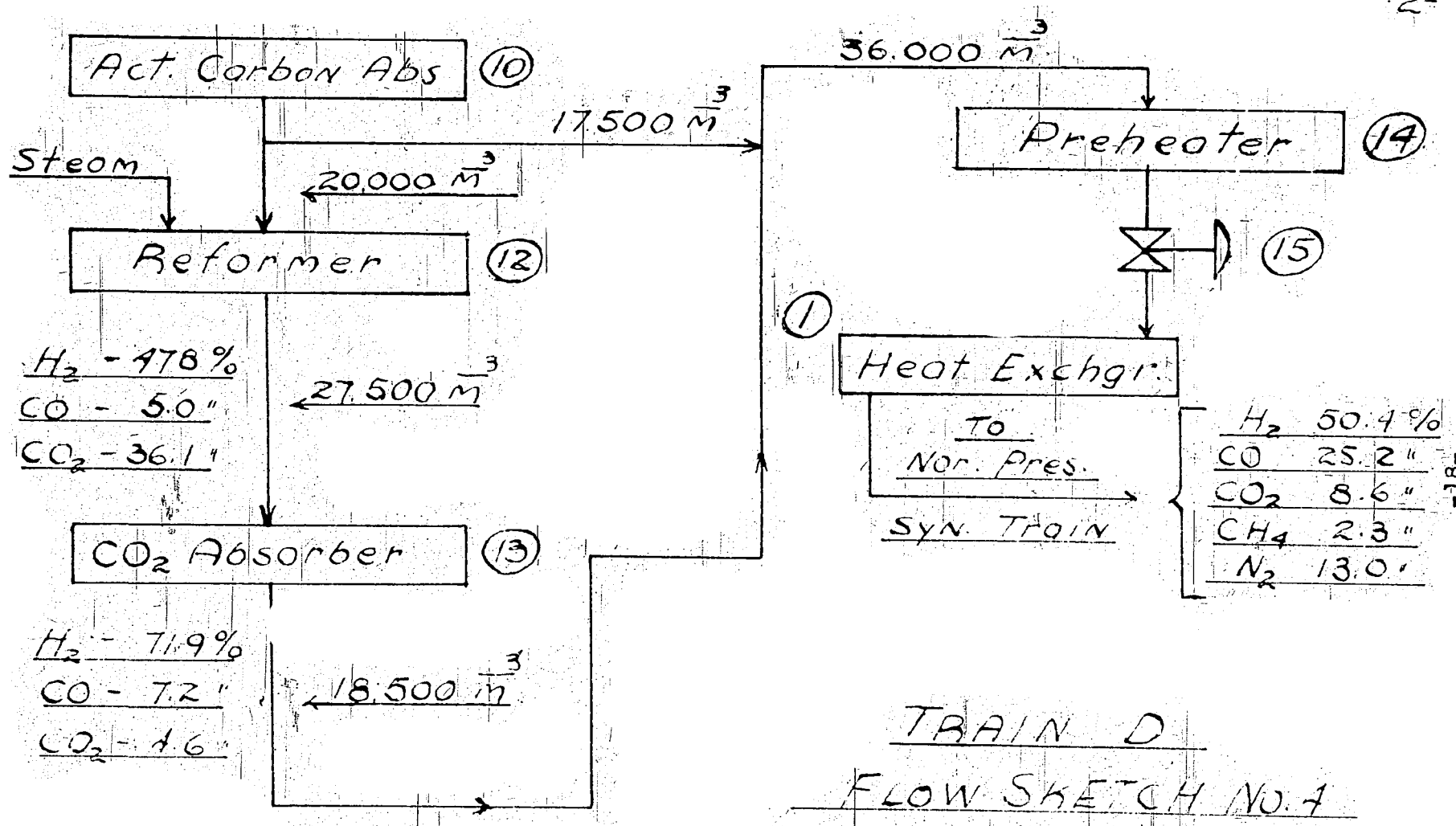


TRAIN D.

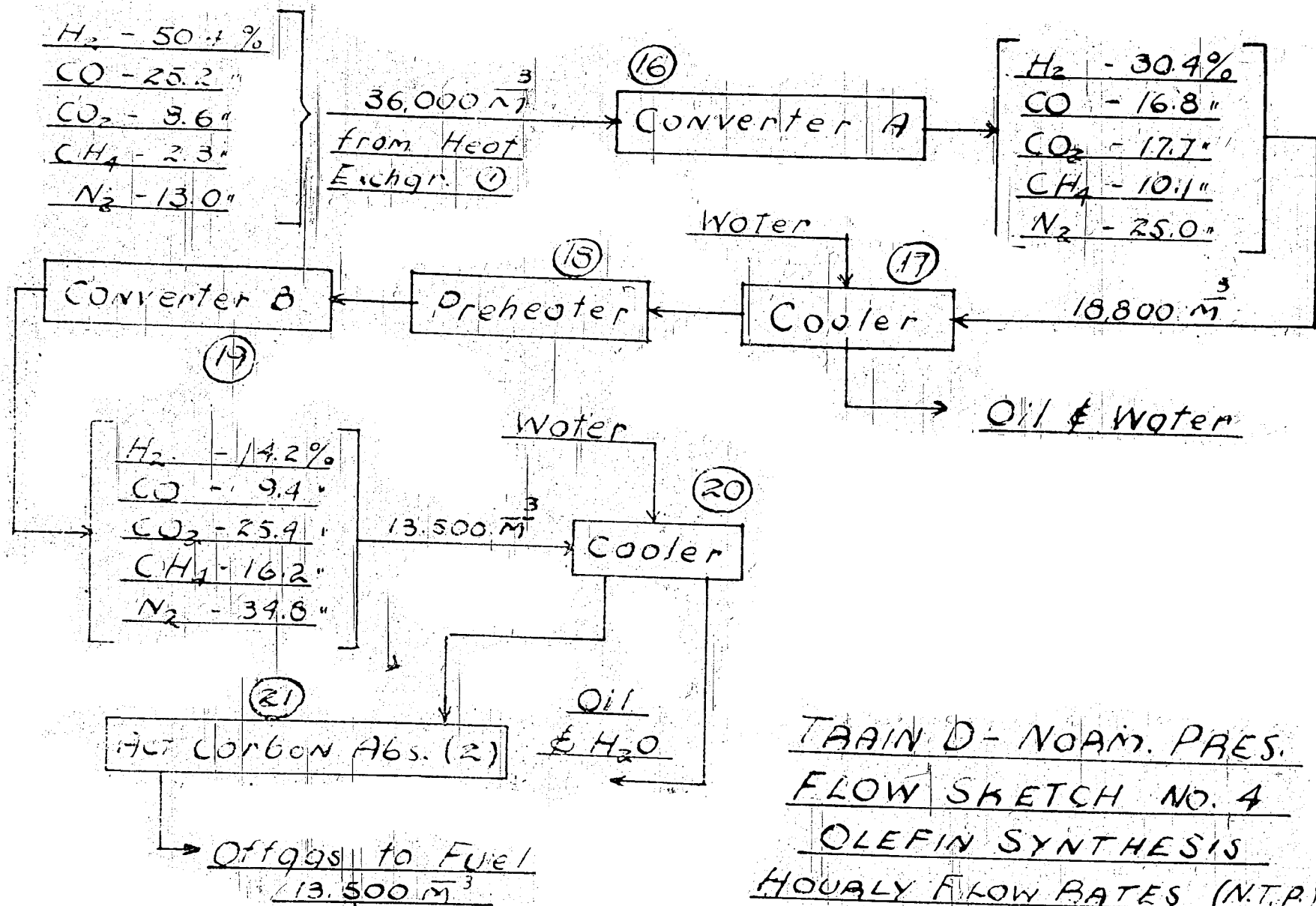
FLOW SKETCH No. 4

OLEFIN SYNTHESIS

HOURLY FLOW RATES (N.T.P.)



TRAIN D
FLOW SKETCH NO. 1
OLEFIN SYNTHESIS
HOURLY FLOW RATES (NTP)



-19-

TRAIN D - NOAM. PRES.
FLOW SKETCH NO. 4
OLEFIN SYNTHESIS
HOURLY FLOW RATES (N.T.P.)

F.R.L. 7-23-95

LEGEND FOR TRAIN D.

- (1) Gas to Gas Heat Exchanger.
- (2) Gas cooler, shell and tube.
- (3) Gas compressor, 20 atm max discharge pressure.
- (4) Gas heater, shell and tube, steam in shell.
- (5) Converter I, " " " (see sk "Medium Pressure Converter").
- (6) Converter II, same as item (5).
- (7) " III " " " "
- (8) Gas to gas heat exchanger.
- (9) Condenser and cooler, shell and tube.
- (10) Activated carbon absorbers (2).
- (11) Recycle gas blower.
- (12) Reformer, (CO and steam reaction for H₂ production).
- (13) CO₂ absorber (H₂O under pressure).
- (14) Gas preheater, shell and tube.
- (15) Pressure reducing valve.
- (16) Converter A (See sk "Low Pressure Synthesis Converter").
- (17) Cooler and condenser, direct water spray type.
- (18) Gas preheater, tubular, finned, steam in tubes.
- (19) Converter B, same as item (16).
- (20) Cooler and condenser, same as item (17).
- (21) Activated carbon absorbers (2).

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Converters were operated at 200-210°C, depending upon the age of catalyst. Converter construction and design were identical with those used for medium pressure synthesis (see Fig. 2). Space velocity was 80 cu m gas per cu m catalyst volume per hr.

Converter exit product passed through heat exchanger (8) counter current to recycle gas; thence to condenser and cooler (9) to effect condensation of oil from the gas stream; the condensed oil was continuously withdrawn from this unit. The exit gases were further stripped of oil products by activated carbon absorber units (10) which were operated as previously described.

Gas from absorbers (10) was split into three streams - (a) the largest portion (recycle gas) was delivered to recycle blower (11) which discharged it at 10-12 atm gauge through heat exchanger (8) to mix with incoming feed gas ahead of gas heater (4); (b) a second portion flowed directly to gas heater (14) for preliminary conditioning for the normal pressure synthesis; (c) a third portion was directed through reformer (12) where the catalytic water gas reaction occurs, resulting in hydrogen enrichment and CO₂ formation. CO₂ was scrubbed out in CO₂ absorber (13) and the scrubbed gas was mixed with portion (b) prior to passage through gas heater (14) and pressure reducing valve (15) then through heat exchanger (1) in counter current flow with hot, purified water gas.

The resulting conditioned gas was fed to a normal pressure synthesis train, which was identical in design and operation with that described in (2) above.

Yields were experienced as follows:

Train 1	-	10 gr liquifiable gas)	per cu m feed gas
		120 gr liquid products)	
Train 2		30 gr total products	" " " "
		160 gr liquid and gaseous products	" " " "

CHEMISCHE FABRIK HOLTEN, HOLTEN-STERKRADE.

1. GENERAL.

This concern is located adjacent to Ruhr Chemie and is owned jointly by the latter, I.G. Farben, and Th Goldschmidt A.G., each holding a 1/3 interest. Managing and technical personnel were not present although the general manager, Dr. Fuller, was expected to arrive shortly.

The plant has been badly damaged.

2. OPERATIONS.

The company purchased ethylene and propylene from Ruhr Chemie and converted them into the oxides by the chlorhydrin process. The capacity was 500 to 600 tons per month, 90% of which was ethylene oxide and 10% propylene oxide.

The finished materials were shipped to one of the Goldschmidt plants for conversion into the respective glycols.

OXO GESELLSCHAFT m.b.H., HOLTEN-STERKRADE.

1. GENERAL.

(a) The company was organized in order to exploit the discovery of Dr. Roelen of Ruhr Chemie, which consisted of the formation of aliphatic aldehydes by catalytic reaction of water gas on olefines followed by the reduction of the aldehydes to alcohols. I.G. Farben and Henkel and Co. were both interested in these alcohols for production of detergents by sulfonation. These two companies therefore joined with Ruhr Chemie in the formation of the new company, each participating 1/3. Since Ruhr Chemie furnished the raw materials, the plant was built adjacent to its property. The finished products were to be shipped to the plant of Henkel and Co. in Witten (Ruhr) to be converted into detergents by sulfonation.

Construction of the plant was about completed at the time of the severe bombing attack in October and November, 1944, but it had never operated.

(b) Bomb Damage.

The bomb damage was relatively slight probably not exceeding 15-20% of the investment. As was the case in the Ruhr Chemie plant, the equipment installed underground, presumably bombproof, consisting of tanks and pipelines suffered a good deal more than the tall reactors and distillation columns above ground. This was caused by extensive cave-ins which demolished the equipment.

(c) Organization.

The investment of the rather lavishly constructed plant was RM 11,000,000. The general manager, Dr. Landgraf, remained in Holten-Sterkrade after the bombing and is now engaged in rebuilding the plant.

Annual production: 10,000 tons higher aliphatic alcohols $C_{12}-C_{18}$

Estimated Cost of Product: RM .90-.95/kg

Market Price: RM 2.50/kg

Raw Materials: Quantities
& Cost.

Power:	1,500,000 KWH /yr at RM .025-.03
Water:	200-cbm/hr
Watergas:	750 cbm/hr at RM .03-.035/cbm
Olefins:	1,250 kg/hr at RM .38-.40/kg
Hydrogen:	400 cbm/hr at RM .60-.70/cbm
Steam:	300 tons/day

2. PROCESS.

An olefin fraction from C_{11} to C_{17} was treated catalytically with purified water gas at medium temperatures under pressure to produce aldehydes which contain one more carbon atom than the parent olefins. The olefins may be derived either directly from the Fischer Tropsch synthesis or by Dubbs cracking of higher paraffins from C_{20} to C_{40} which were produced by Ruhr Chemie. The olefins should be predominately straight chain with the double bond at the end. The reason for this is that sulfonated alcohols derived from the other types of olefins have too low a melting point for the desired commercial uses although their detergent properties may not be impaired. The reaction was carried out batchwise, at temperatures of 125°C initially, 135°C at midstage, and 145°C at the finishing point, under 150 atm, using the standard Ruhr Chemie cobalt catalyst. The catalyst was finely ground to about 200

mesh and suspended in the olefin charge. The purified water gas, which was obtained from Ruhr Chemie, contained 38% CO, 48-49% H₂, with the balance being nitrogen and CO₂.

FLOWSHEET - (See Sketch Attached).

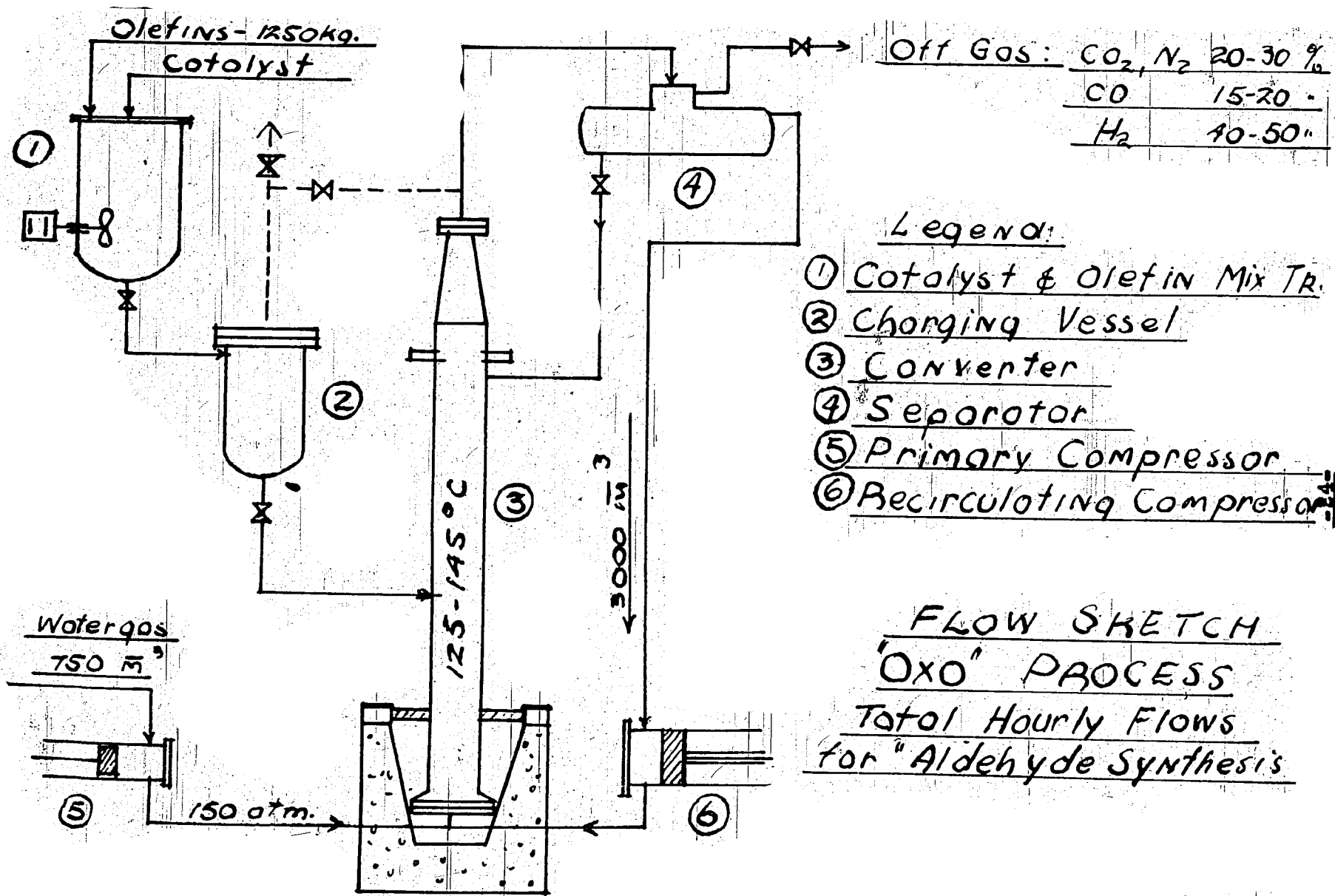
A batch charge of liquid olefins was mixed with the necessary catalyst in tank #1, transferred into charging vessel #2 after which the pressure was equalized with the system. The charge was then emptied by gravity into the converter #3, equipped with a bundle of bayonet heating and cooling tubes. Compressor #5 supplied water gas to the converter while its exit gas was largely recycled by compressor #6 and vented from separator #4 sufficiently to maintain adequate hydrogen and CO concentration within the system. The batch cycle was 1 hour, half of which was reaction time and the other half required for charging and discharging. Gas circulation was maintained during charging and discharging.

Batches of crude aldehyde were collected in storage and converted to alcohols by hydrogenation using the same catalyst and the same type of equipment as used for the aldehyde synthesis. The operation was also batchwise, with pressure of 150 atm and temperature of 180°C. Hydrogen of 90-92% purity (balance nitrogen) was used and recycled similar to the watergas in the aldehyde synthesis.

Of the 9 converter units installed, 5 were to be used for aldehyde synthesis and 4 for hydrogenation.

The crude alcohol was purified by vacuum distillation in batch stills and plate columns.

1 pound of olefin was expected to yield approximately 1 pound distilled alcohol.



F.B.L.
7-21-45.