V. CO_2 PURIFICATION

(a) The unit consists of eight scrubber columns, each two (2) meters in diameter and twenty (20) meters high. In this purification step, the CO_2 content of the synthesis gas is stripped down to a trace. The operation is carried out at twenty-eight (28) atmospheres pressure.

(b) Simultaneously with the CO_2 purification, the remaining H_2S is also removed. The absorbed gases are released from the water by expansion. The quantity of wash water is fixed by the required purity from CO_2 in the synthesis gas and by the allowable combustible gas quantity in the CO_2. The process is carried out in such a way that the CO_2 content of the washed hydrogen is from 1.5 to two (2) percent, while the $CO + H_2$ content in this CO_2 is never above 7.5 percent. The water containing the absorbed gases is expanded in Pelton turbines which drive the fresh water pumps. The process is shown on the attached flow sheet (Fig. 8).

(c) The expansion releases eighty (80) percent of the absorbed CO_2, the inert gases and the largest part of the H_2S. The expanded CO_2 normally contains six (6) to six and one-half (6.5)

Principal

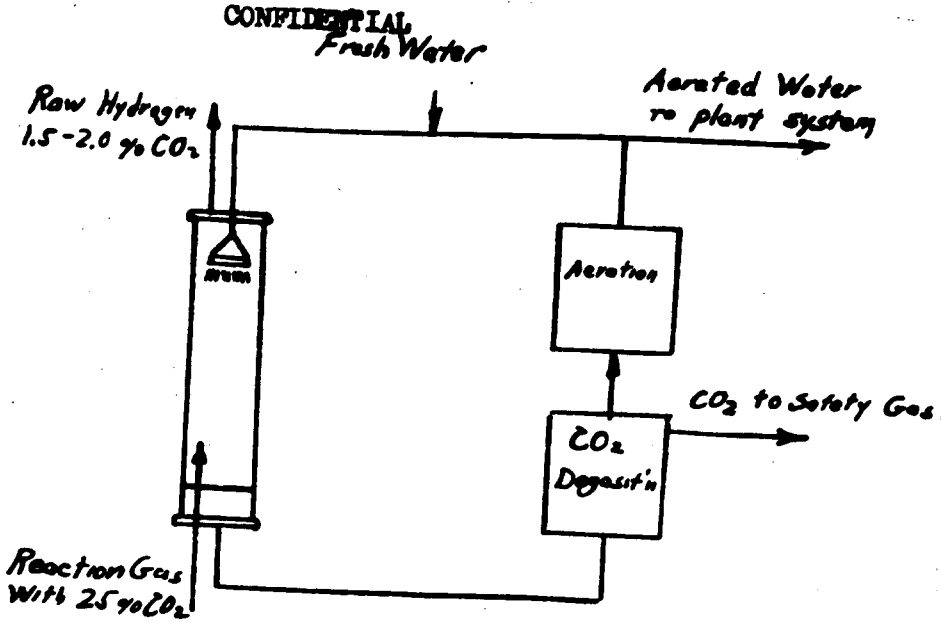
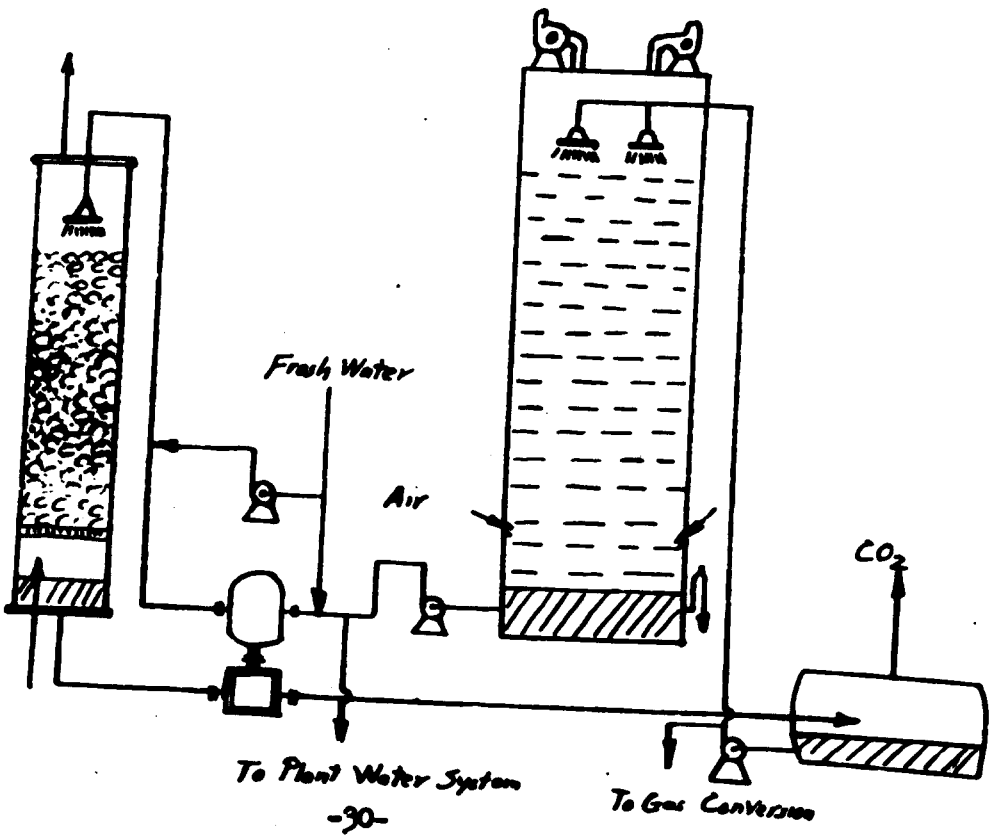


FIG. 8

CO_2 Removal

Flow Sheet



percent of H_2 and CO, and approximately five hundred (500) milligrams/cubic meter of H_2S.

(d) The expanded wash water is aerated to remove the remaining CO_2 and H_2S, and used again. partly for CO_2 washing and part returning to the plant water system.

(e) The re-using of aerated water in the CO_2 removal, serves to introduce the proper amount of O_2 in the raw hydrogen, as it tends to maintain the proper copper salt balance in the cupric caustic wash to follow in CO removal.

(f) Since the plant water, coming from wells, has only two (2) to three (3) milligrams/cubic meter of O_2, which is insufficient, the introduction of the ten (10) milligrams/cubic meter of O_2 is important. The plant well water and the re-used aërated water are mixed 1 : 1.

(g) The CO_2 wash towers and the H_2O regeneration gave adequate capacity for both the gas producing units and the methane cracking plant. This pre-supposes that no operating difficulties exist in thw wash towers. These can be clogged up by algae growth on the packing rings. Due to this, the capacity of the CO_2 wash towers have sometimes been reduced to half.

(h) In order to maintain production, definite steps had to be taken. Since the central water pumping station could not assist, the algae had to be removed from each wash tower individually. This was done by circulating chlorine water in each tower.

(i) The towers were filled with water at twenty-five (25) to thirty (30) degrees centigrade. The chlorine was injected in batches every two (2) hours and circulated. After each injection, the chlorine content ran from one hundred (100) to two hundred (200) milligrams/liter. Each washing required fifty (50) to eighty (80) kilograms of chlorine and took two (2) days to complete. The algae were completely removed and the towers were able to run as if new.

(j) Another possibility for improving operation is in using larger tower packing. By increasing the size of the ceramic

V. CO_2 PURIFICATION. (c) (Cont'd.)

rings from sixty (60) millimeters to eighty (80) millimeters size, the capacity of the unit was increased fifteen (15) percent.

OPERATING RESULTS

HOURLY QUANTITIES

	1943	1944
Synthesis gas flow	103,300 cu. meters/hr	120,000 cu.meters/hr
Avg. wash tower load	15,700 cu.meters /hr	17,000 cu.meters/hr

CO_2 REMOVAL

CO_2 - gas absorbed in	23,600 cu.meters/hr	26,000 cu.meters/hr
H_2O solution	5,400	6,000
CO_2 in synthesis gas	27.4 %	26.8 %
CO_2 in washed H_2	1.8 %	1.8 %
$CO + H_2$ in CO_2	7.1 %	7.1 %

WASH WATER

Water used	4,520 cu.meters/hr	4,800 cu.meters/hr
Fresh Plant Water	55 %	55 %
Re-circulated Water	45 %	45 %
Water needed/100 cu.meters of	44 m^3	42 m^3
Synthesis gas water temperature	9-18°C.	8.5-15°C.

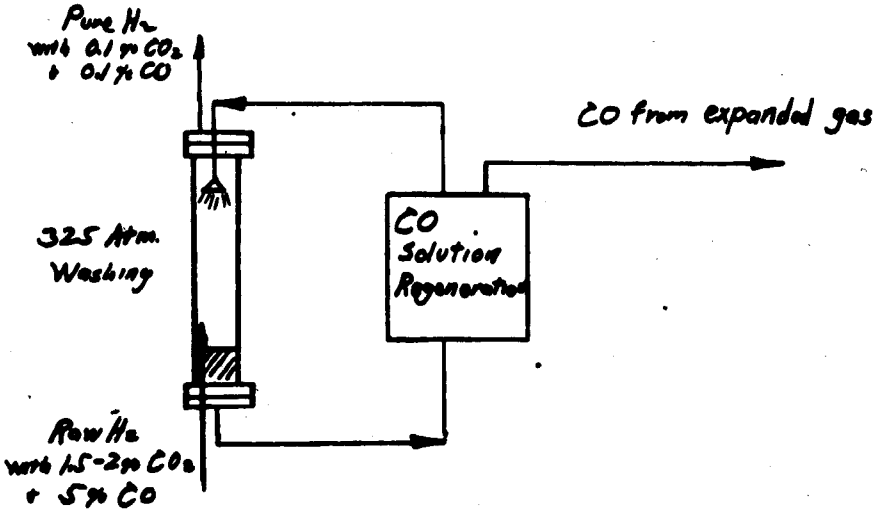
VI. CO PURIFICATION.

(a) The CO purification unit consists of seven wash towers and equipment. In this step, the raw synthesis gas is stripped of the remaining CO and CO_2 by absorption at three hundred twenty-five (325) atmospheres (Fig. 9). The absorption medium is an ammoniacal copper solution. After use, it is regenerated by a mild heating both at atmosphere and sub-atmosphere pressures. The gases removed are next washed to remove NH_3 entrainment and used in the gas conversion process. The expanded gas energy in

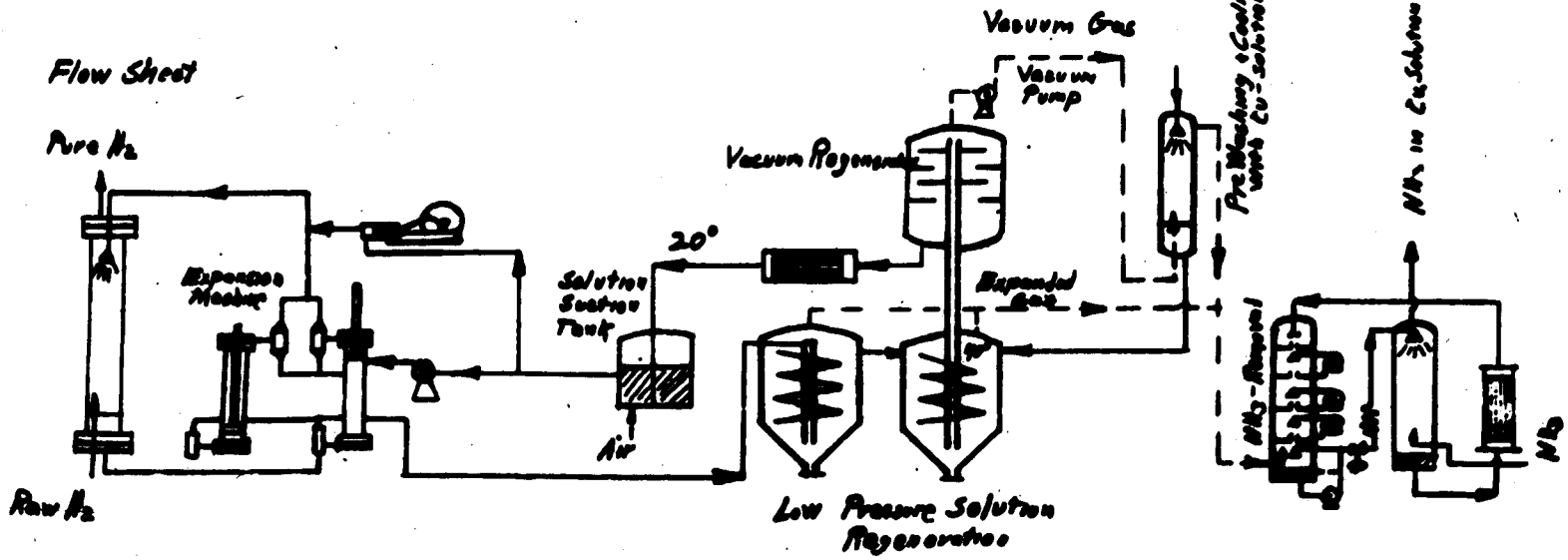
Fig. 9

Schematic Flow of CO Removal

Principal



Flow Sheet



VI. CO PURIFICATION. (a) (Cont'd.)

the stripped gases is recovered one hundred (100) percent by driving the pumps which handle the fresh copper solution.

(b) The copper solution contains CU. NH_3 and CO_2. In its operating state it is as follows:

$$CU \cdot \begin{matrix} NH_3 \\ NH_3 \end{matrix} \quad CO_3$$

$$CU \cdot \begin{matrix} NH_3 \\ NH_3 \end{matrix}$$

It is cuprous tetramine carbonate. Hence one (1) molecule of copoer can absorb one (1) molecule of CO. This absorption is increased as the ammonia concentration is increased. However, a limit of the allowable NH_3 is reached because of the base with which ammonium bicarbonate drops out of solution.

(c) On heating, the CO acts as a reducer, leaving a solution of metallic copper. In order to arrest this tendency a definite quantity of cupric copper must be on hand. However, since cupric copper can not absorb CO, its content must be minimized, and the rest made cuprous copper. The optimum solution has the following specifications:

Sp.gr. @ 20°C.	1.15	
Cuprous salt content	12	mols/10 liters = 6.6 %
Cupric salt content	2	mols/10 liters = 1.1
NH_3	0.480	mols/10 liters = 7.1
CO_2	0.240	mols/10 liters = 9.2

(d) Two-thirds (2/3) of the ammonia is in the complex, the rest is tied up with the carbon dioxide. The cuprous containing complex is colorless as the blue color of copper solution is due to cupric rather than cuprous salts. Theoretically, this solution can absorb twenty-seven (27) times its volume of CO but in practice only two-thirds (2/3) of this figure is obtained.

VI. CO PURIFICATION. (Cont'd.)

(e) The old method of making the solution entailed considerable loss of NH_3. It consisted in simultaneously pumping CO_2, air and ammonia water over copper. This generated much heat which had to be removed.

(f) The new method consists in pumping standard copper solution over new copper and results in no ammonia loss. Although the process is slow, it is still sufficiently fast as not to cause delay, and requires no supervision.

(g) The unit started up without difficulty and has since run well. The only operating difficulty was in packing the expansion machine and the pumps, as it was difficult to obtain chrome tanned leather. Buna rubber has proved to be a good substitute.

(h) The capacity of the unit is fixed by the head available on the high pressure pump of the wash system, as high capacity operation produces high resistance due to its complicated piping system. By simplifying the piping, the copper solution quantities was raised from two hundred forty (240) to two hundred eighty (280) cubic meters/hour. With this amount of solution, eighty-five thousand (85,000) cubic meters/hour of raw hydrogen, having a CO content of 5.5 percent, can be treated. By lowering the CO content to 4.5 percent, as should occur by properly balancing the water gas - methane cracking gas quantity, one hundred thousand (100,000) cubic meters/hour can be treated.

OPERATING RESULTS

HOURLY QUANTITIES

	1943	1944
Avg. raw H_2 quantity treated	74,100 cu.m/hr	86,000 cu.m/hr
Avg. H_2 finished product	67,500 cu.m/hr	78,000 cu.m/hr
of which @ 325 Atm. H_2	23.5 %	28.5 %
700 Atm. H_2	76.5 %	71.5 %
Avg. Wash Tower load	16,800 cu.m/hr	17,500 cu.m/hr

VI. CO PURIFICATION. (h) (Cont'd.)

CO REMOVAL

	1943	1944
CO removed	5,900 cu.m/hr	7,000 cu.m/hr
CO content in raw H_2	5.4 %	6.0 %
CO content in finished H_2	0.2 %	0.2 %

ANALYSES OF CO REMOVED

CO_2	21.0 %	21.0 %
CO	63.1 %	63.0 %
H_2	13.8 %	14.0 %
NH_3	3 mg/m^3	2 mg/m^3

COPPER SOLUTION

Solution used	251 cu.m/hr	255 cu.m/hr
Colution required for 100 cu. meter of pure H_2	3.7 cu.meter	3.2 cu.meter
Temp. of fresh solution	20.5°C	20.7°C
Temp. of regeneration	40.6°C	39.5°C

SOLUTION ANALYSES

Cu_2O mol/10 liters solution	11.0	12.0
CuO mol/10 liters solution	2.1	2.2
NH mol/10 c. c. solution	0.488	0.490
CO mol/10 c. c. solution	0.248	0.250
Copper loss (% of makeup)	1.83 %	1.3 %

VII. HYDRCGEN COMPRESSORS AND RECIRCULATORS.

(a) The hydrogen compressors, eleven in number, operate with six (6) stages and are driven by synchronous motors. In stages one (1) to three (3), the synthesis is compressed to twenty-eight (28) atmospheres whence it goes to the CO_2 purification step. The purified gas is then compressed in stages four (4) to six (6), up to three hundred twenty-five (325) atmospheres. At

VII. HYDROGEN COMPRESSORS AND RECIRCULATORS. (a) (Cont'd.)

this pressure, part of the total gas is used for the gas phase hydrogenation. The remainder is compressed to seven hundred (700) atmospheres in seven single stage boosters for the sump phase step. The boosters are hydraulically operated.

(b) The compressors required numerous alteration. The original piston bearings were held in place by springs, these were later shrunk in. The piston rings had to be so fitted, as not to rotate duting operation thereby allowing the gas to by-pass the joints. The "Presko" piston ring spring was substituted in the fourth stage for "Thermit" springs. By the above changes, the compressors were able to operate without repairs for an average operating period of five thousand (5,000) hours.

(c) The boosters experienced no particular difficulties. The most reliable discharge temperature was found to be ninety (90) degrees centigrade, which temperature is controlled by the difference in pressaure between suction and discharge. To hold ninety (90) degrees centigrade temperature, the discharge pressure was made twice the suction pressure.

(d) The lubrication of the compressors called for special oils. An explosion occurred in one unit while being tested on air, due to lubricating oil. The best oil was one having an Engler viscosity from fifteen (15) to twenty (20) at fifty (50) degrees centigrade. The running parts used the same oil as the cylinders. The oil used amounted to 0.7 grams/H.P. hour or 3.8 kilograms/hour for each compressor and 6.5 grams/H.P. hour or 5.4 kilograms/hour foreach booster. The largest oil loss was in the discharge of each cylinder. This oil was regenerated by centrifuging and the loss made up with fresh oil.

(e) The loss in the machine and the centrifuging amounted to twenty (20) percent, which for eight (8) machines amounted to six (6) tons/month. This loss was later cut in half by improving the oil separation through settling in two (2) decanting tanks at sixty-five (65) degrees centigrade every twenty-four hours.

(f) The capacity of the compressors was to have been increased by reboring the cylinders. Only stages four (4) to six (6) would have needed this as stages one (1) to three (3) were already over-

Principal

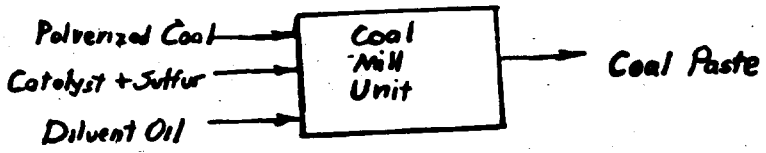
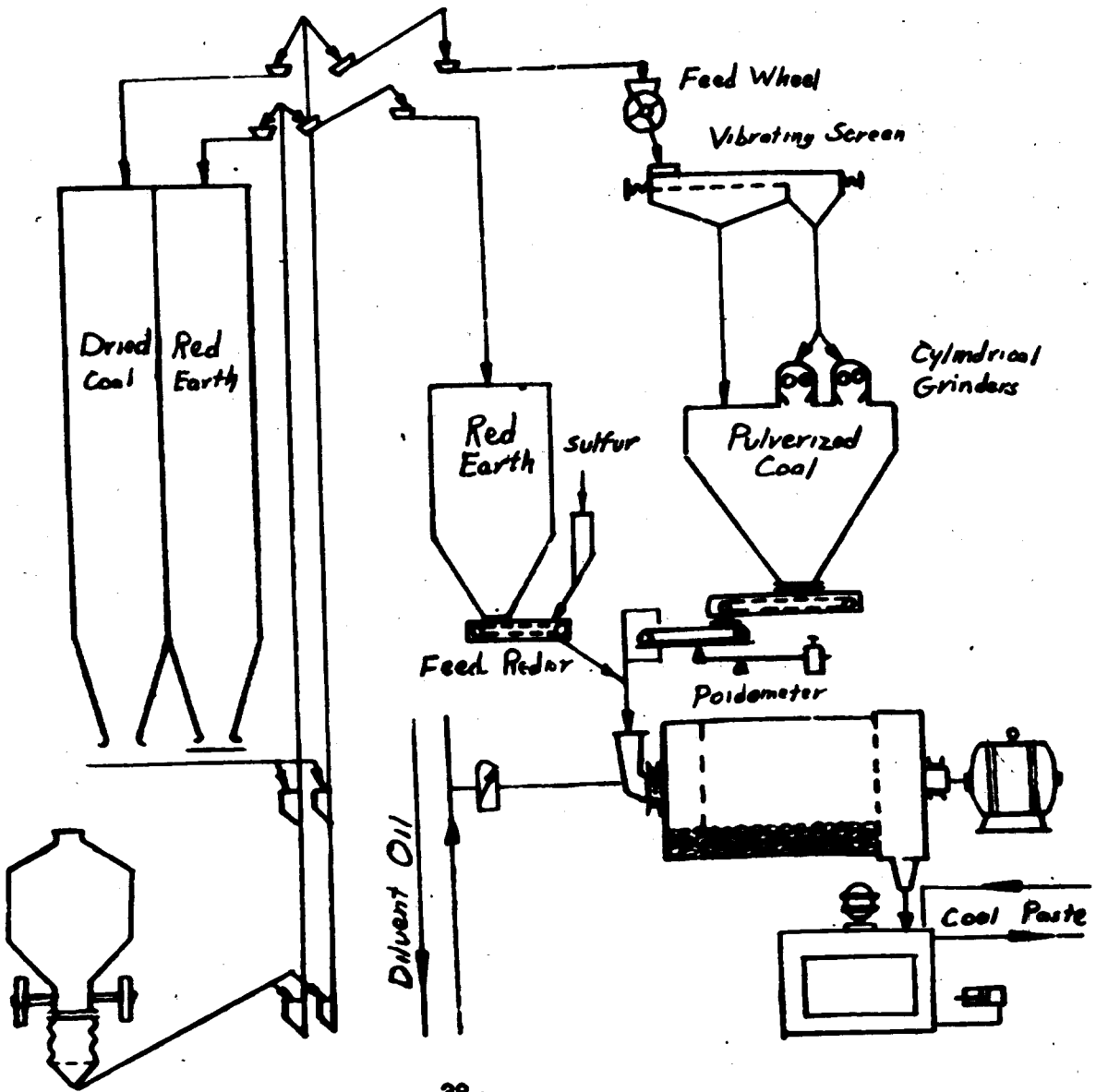


FIG. 10

Coal Preparation Unit

Flow Sheet



VII. HYDROGEN COMPRESSORS AND RECIRCULATORS. (f) (Cont'd.)

sized. By such changes the compressor output would have increased twenty (20) percent and the booster ten (10) percent. The electric drives were sufficient for the increase.

OPERATING RESULTS

COMPRESSORS

	1943	1944
Total gas throughput	103,100 cu.m/hr	120,000 cu.m/hr
CO_2 purified gas	74,100 cu.m/hr	85,000 cu.m/hr

INDIVIDUAL MACHINES

Stages 1 - 3	13,300 cu.m/hr	14,400 cu.m/hr
Stages 4 - 6	9,400 cu.m/hr	10,400 cu.m/hr
Avg. working period	4,000 hours	5,000 hours

BOOSTERS

Avg. 700 Atm. H_2 quantity	51,000 cu.m/hr	55,000 cu.m/hr
Individual machines	10,800 cu.m/hr	10,900 cu.m/hr