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ENLARGING AND IMPROVING THE HEAT REDUCTION INSTALLATION  
FOR FUSED IRON CATALYST OF THE SYNCE SYNTHESIS

The purpose of the discussion was to plan any ~~current~~ improvements in the process and to increase the capacity of the installation to 10,000 t/yr size.

1. Dr. Braynich started by describing the installation No. 453 and the present interpretation of its defects.

The reduction is made with a very pure hydrogen ( $S < 0.02$  mg) delivered under about 250 atm pressure, and therefore practically moisture-free after the pressure has been released.

Hydrogen is admitted through layers of IFC coke heated to 100° (G1 and G2) into the reduction circuit. The circuit is fed by the circuit piston blowers A1 and A2 (capacity 1000 cbm/hr against about 60 mm Hg when the preheater is hot, and 1500 cbm/hr with a cold preheater). Hydrogen passes through a preheater H1 (diameter 7") into the reduction vessel R (diameter 1400; hydrogen velocity 20 cm per second) is cooled in the cooler K1 to 30° and in the ammonia cooler K2 to 49, and then passes through the silica gel tower (K3, and K2). Each tower is about 300 li capacity, the particle size is 2-6 mm, (lower half silica gel B, upper half silica gel A) and then returned to the blower.

The height of the catalyst layer with 200-300 li capacity is about 35 cm, and the expanded space is 1:2000. The silica gel tower is dried by a second hydrogen circuit consisting of a blower A3 (capacity about 200 cfm) preheater V2 (heated with high pressure steam) and cooler E3.

50 cfm of make-up hydrogen per hour, later reduced to 35 cfm, are fed into the circuit, and 10-25 cfm are used up, while the rest has heretofore to be discharged into the air. The reduction temperature is 450°, the time for the reduction 56 hours. After changing over from the 1-2 mm catalyst size to 0.3-1 mm, the reduction time could be somewhat reduced, and 48 hours should become sufficient.

After passing the silica gel tower, the reduction hydrogen contains 1-2 gms H<sub>2</sub>O per cfm. Counting all the auxiliary operation, about 6 days are required for the reduction of the 200 li charge. About 10 hours each are necessary for the heating up and for the cooling down to 30°, which must be added to the reduction time because the preheater itself must be heated up and cooled each time. The rest of the time is required for purging the apparatus with carbon dioxide and with air, adjusting the gauge grates and for removal and filling of the catalyst, and is liberally estimated. The installation is used to capacity if the ratio of 1:2000 is kept. The bottle necks are in the capacity of the blower and in the preheater. The heating gas blower for the burner is operated to its maximum (140 per cfm). The heating coil may not be heated above 700°C and its i.d. is 30 mm and the hydrogen velocity at an average temperature of 400° is about

200 m per second. The coil forms therefore also the principal resistance of the circuit (about 200 mm Hg) while the resistance of the layer of the catalyst is at most equal to 2-5 cm of water column. When the circulation gas is cooled to  $4^{\circ}$  by the ammonia cooler, the steam is cooled to the dew point, and all the water formed in the catalyst is absorbed by the silica gel. The silica gel temperature will then be raised by about  $25^{\circ}$  by the liberated heat of adsorption and the ammonia cooler still retains a certain value.

About 2-3 kg  $H_2$  is formed during the reduction from the nitrogen in hydrogen, and it appears in the water of the silica gel as an about  $n/2$  kg-solution. The  $H_2$  presumably enlarges the pores of the silica gel and reduces gradually its capacity for the absorption of water.

2. The following suggestions for the improvement and enlarging of the installation and for an eventual new installation were made during the discussion.

Reduction vessels: The preparation and after treatment occupy a relatively long time in comparison with the reduction time, and a second vessel of the same size may be installed parallel to the present one, as shown in drawing 2. One vessel (A or B) is always in operation. The cooling of the catalyst may be done from the same circuit through the valve V (heat exchanger C is included in the drawing for a better heat recovery).

An apparatus for continuous operations can also be devised. We may consider two such arrangements.

In figure 3 the hydrogen enters from below through a system of concentric rings of a suitable profile, opening downward (W1) and passes upwards from below through the filling cone in B. The worm drive M discharges the catalyst continuously. There are hardly any objections in principle to such an arrangement because of the excellent flow properties of the catalyst and its great hardness. Any possible irregularities in the distribution of the layer could be regulated by lead-in troughs and by suitable shaping of the H<sub>2</sub> intake grate.

Figure 4 is designed to have a minimum thickness of the layer. Two concentric cylinders (Z1, Z2) are perforated in the length S1. The hydrogen enters either from the inside (W1) to the outside (W2), or possibly in the reversed direction with the same distribution.

The container B in figures 3 and 4 can be cooled with the make-up hydrogen entering the circuit (W2-WA2), because the amount of hydrogen for cooling of small amounts of catalyst is sufficient per unit time. The use of make-up hydrogen offers the advantage of having the catalyst cone in contact only with very dry hydrogen after the reduction.

The ammonia cooler in the circuit has in the present arrangement the single purpose to cool the gas, because no water will be separated at 4°C. One can operate without it if coolers are introduced into the silica gel containers of the larger reduction installation. In that case water can be used advantageously, and is introduced into the water cooler of the circuit (K1 in fig. 1).

The 2-4 kg ammonia formed per batch during the reduction interferes with the drying of the gas. It could be removed by an acid wash in front of the silica gel, using a trickle tower, a bubble tower, or pumice saturated with acid. It must however be borne in mind that the silica gel containing some ammonia solution would remove any  $\text{CO}_2$  present, which would be objectionable in the reduction. Moreover experiments should be made to determine whether  $\text{MnO}$  serves to improve the properties of the catalyst.

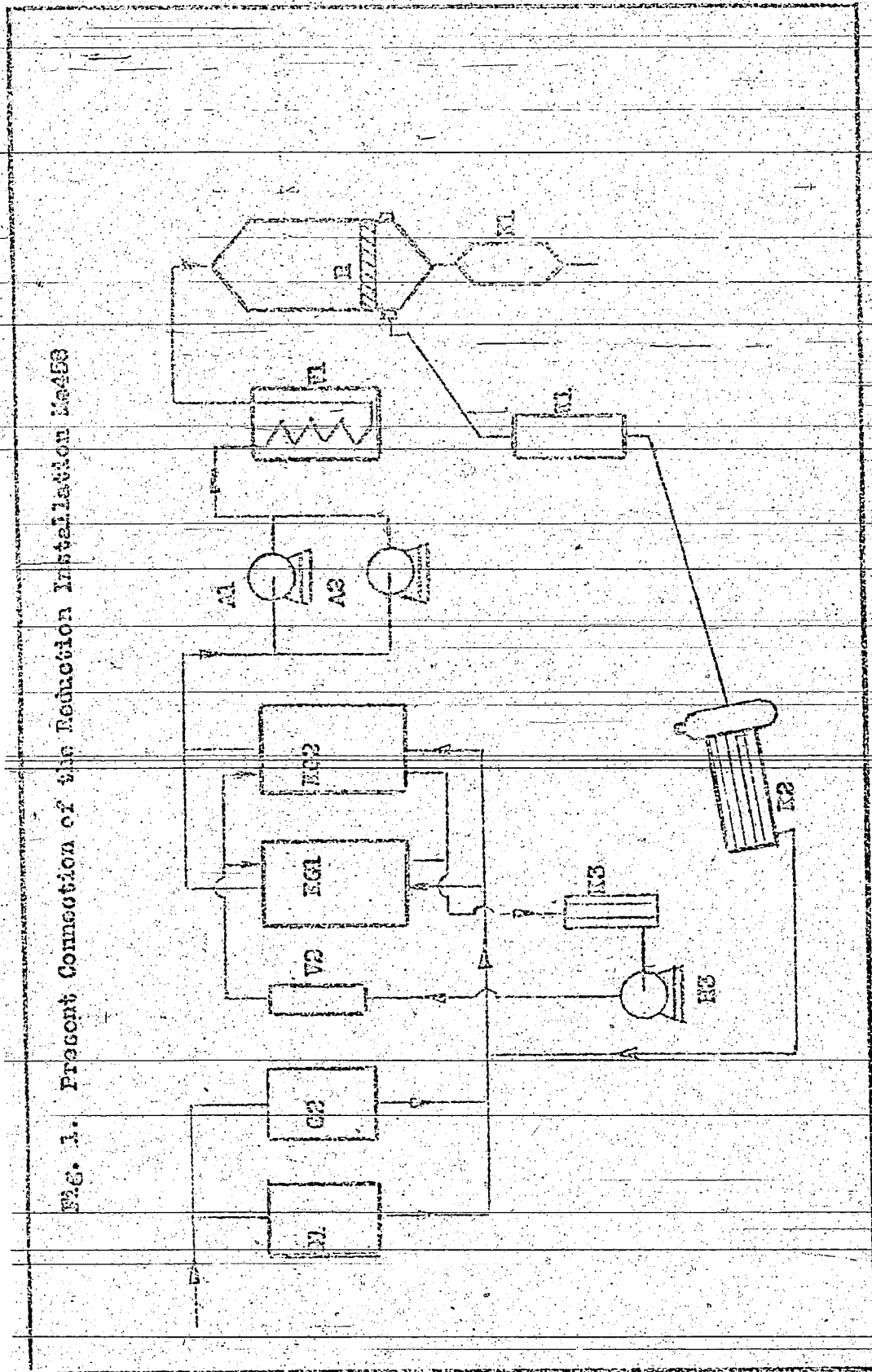
A better drying of hydrogen could be brought about by the addition of 2 more silica gel towers, G3 and G4 to the two in place, G1 G2 (fig. 1). These might be dried by means of dried hydrogen from the main circuit, possibly behind the reducer C at D. This drying hydrogen would be returned to the main circuit after passing a cooler E2. In this case the hydrogen would be branched off at c for cooling. Reduction experiments with different moisture content in hydrogen are at present being conducted in Me488.

3. Capacity of the available installation and of the eventually enlarged and improved installation: Six days are required to dry 0.6 cbm catalyst in the present discontinuous operation. 30 cbm could be reduced during one year. A 10,000  $\text{kg/yr}$  installation would require 155 cbm of catalyst per year with a yield of 540 kg of the products per cbm of catalyst and per day and with a catalyst life of 150 days. By installing a second catalyst unit in parallel, as shown in figure 2, additional 30 cbm could be reduced without any further changes. The addition of a second blower in parallel

(capacity about 1000 cbm/hr), the introduction of a heat exchanger and enlarging the preheater coil from 80 mm to 150 mm i.d. could increase the amount of catalyst to 180 cbm per year (with a catalyst layer height of 1 m). Installing a continuous drier would also bring the amount up to 180-200 cbm per year. In addition, the operations in the latter case were considerably simplified with a probable improvement in the quality of the catalyst.

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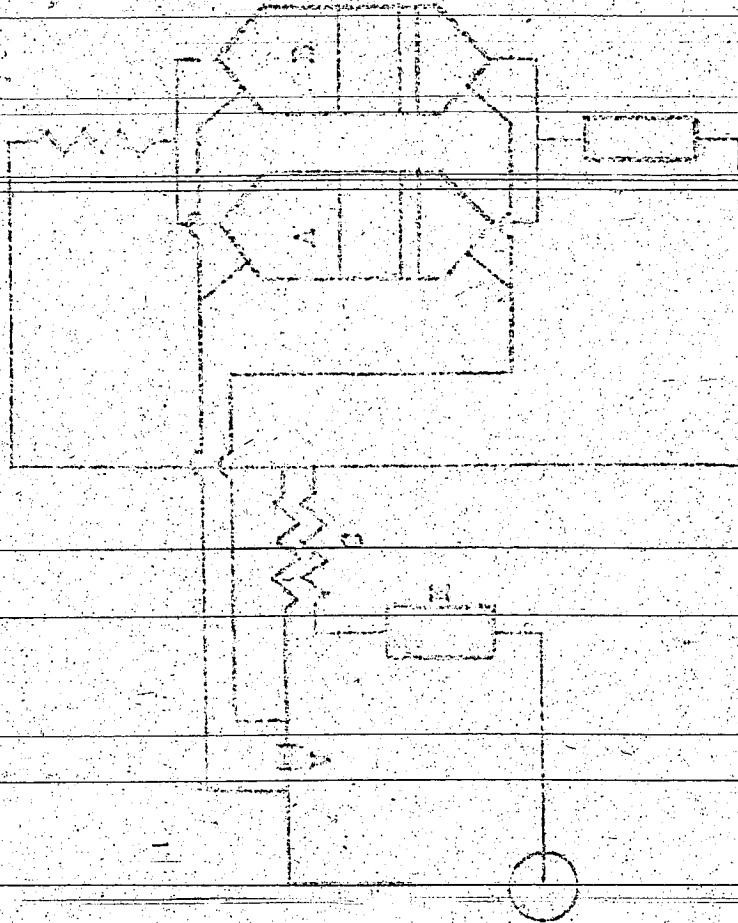
Fig. 1. Present Connection of the Reduction Installation M-453



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Completion of the Substitution Installation in 9458 at 1000  
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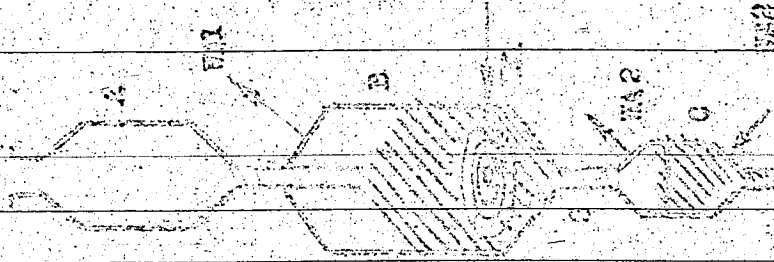
Fig. 2



Completed Connections

Number

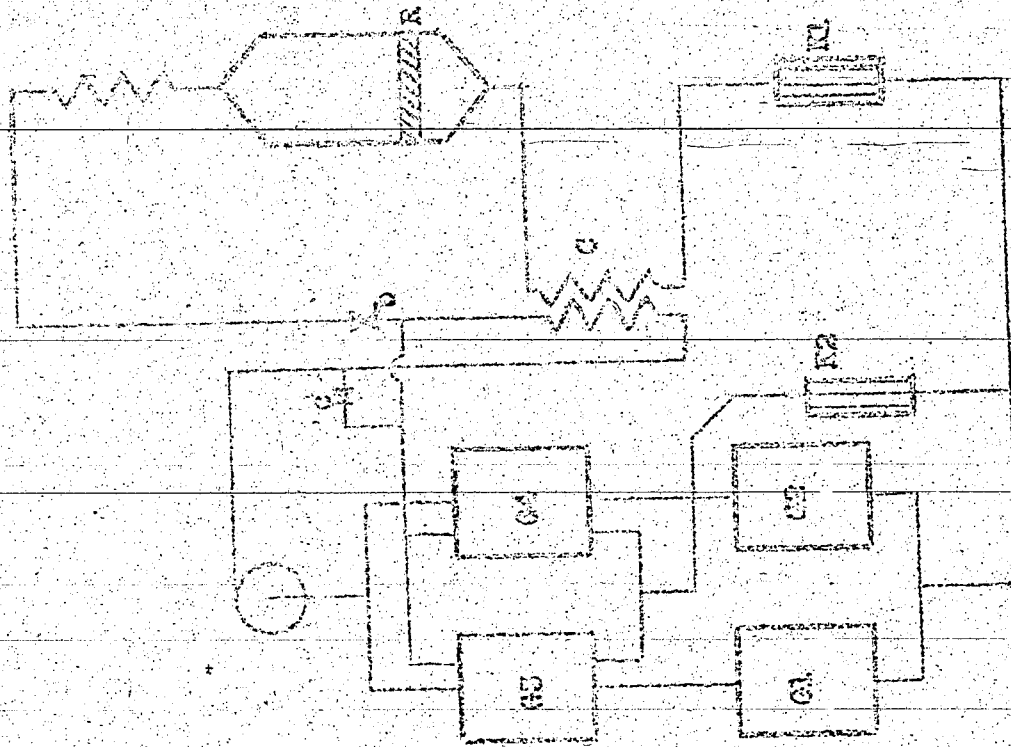
Fig. 3





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ASOCIACION TECNICA DEL YUCATO  
FIG. 5



2. Schematic Diagram of Continuous  
Catalytic Reducer  
FIG. 2

