

Bag 2743 Target No. 30/4.09
Scholven - Item 4 (d)

March 6, 1940

Report on Trip to Engineering Office, Uhde, Leuna Works, Merseberg.
Date: February 14-24, 1940. Subject: T52 Plant

Present: Leuna Works: Messrs. Keinke, Fischer, Jagermann, Strätz, Gerlach,
Weis,
Uhde Eng. Office: Messrs. Falzer, Griephan,
Scholven: Messrs. Schiwy, Fabian, and Rumpf.

There has already been prepared a report by Mr. Fabian on the discussions of February 14-17, 1940, to which the reader is referred.

A. Utilization of Waste Heat

The following possibilities of utilization of waste heat in plant T52 were considered:

(a) The recycle butane (about 15 metric tons per hour) can be recirculated in gaseous state for the purpose of saving the liquefaction and evaporation costs.

(b) Butane evaporation with waste heat.

(c) Absorption cooling system.

In respect of (a): It is not considered feasible to recirculate the recycle butane in the gaseous state since the dehydrogenation must be operated uniformly, so that storage of the recycle butane is necessary. It is difficult in the gaseous state, but simple in the liquid state because the recycle butane is stored in tanks.

In respect of (b): In the product gas from the hydrogenation there is available about 1,100,000 cal. per hour at a temperature drop of from 200° to 100°. It was agreed to design an evaporator for the combined employment of this waste heat of the gas and of the heating steam. During the discussion such an evaporator was sketched by Uhde and later considered in detail from the design viewpoint. Its employment is possible.

About 2.2 metric tons per hour of steam; and	=	5.00 RM/hour
About 100 cubic meters per hour of cooling water	=	1.50 RM/hour
	=	<u>6.50 RM/hour</u>
	=	52,000 RM/year

are saved by this waste-heat utilization.

The slightly higher plant cost for the evaporator is not important in comparison with this saving.

In respect of (c): The employment of the product waste heat for the operation of an absorption cooling system has been eliminated through (b). The employment of the waste heat of the flue gas from the boiler house for the production of cold does not pay.

(1) Only about 250,000 cal. per hour are effectively present as compared with a demand of about 850,000 cal. per hour, and

(2) At a cooling of the flue gas below 200°, according to experience at Leuna, there is present the danger of sulfur precipitation. The sulfur clogs the pipes and at the same time causes corrosion.

B. Drying of Isobutane

As is known, the contact of the dehydrogenation process is very sensitive to water. The isobutane must therefore be well dried, both the recycle isobutane and the fresh isobutane. The drying plant is preferably placed ahead of the dehydrogenation, and the isobutane dried in the liquid state. There are principally two methods: (a) freezing, (b) drying with caustic potash or soda.

(a) Mr. Gerlach proposed the employment of the butane itself as the refrigerant, to precool it in a heat exchanger and then to expand it to -10°. The butane vaporized in this way is compressed and liquefied. Thus, it is itself the refrigerant. At -10° there can be expected a sufficiently reliable freezing out of the water. Whether the ice is also sufficiently separated from the liquid butane, or whether emulsions do not sometimes form, still remains to be clarified.

(b) The employment of the potash solution obtained in drying with caustic potash was discussed. Leuna will look into the question of procuring its caustic potash from an electrotechnical works in the neighborhood and returning the potash solution.

The employment of caustic soda was discussed as the soda solution can be further employed for the benzine washing. However, caustic soda does not dry as thoroughly.

Leuna will test which of the various drying methods is the most suitable.

C. Sulfur Purification

An organic sulfur purification of the hydrocarbon gas is practiced at Leuna. The hydrocarbon gas is conducted at about 300° over a catalyst, and the organic sulfur is converted into H₂S. Leuna is willing to have Uhde make us an offer on such an installation.

The sulfur purification removes COS and mercaptans but not thiophene. It operates under a slight pressure. According to data by Messrs. Ester and Keinke, there is obtained a purity of about 2 mg. sulfur per cubic meter. The isobutane treated in the Leuna T52 plant contains up to 10 mg. of organic sulfur per cubic meter of isobutane gas or 7 mg. organic sulfur per kg. isobutane. Since it may be assumed that sulfur is concentrated in the fuel gas, the two reports are in rough agreement.

The treatment with "Doctor" solution does not remove the organic sulfur but only converts it into disulfide which is later decomposed, for example on heating in a column. This method is therefore useless.

It can be seen that the Leuna sulfur purification of the hydrocarbon gas is not entirely satisfactory. The gas purification also has the disadvantage that all the gas must be purified while only the purification of the fuel gas is necessary.

D. Polymerisation of Propylene Formed in Stabilisation Column

About 540 Kg. per hour of liquid product, containing about 30% or 160 kg. per hour of propylene and about 40% or 220 kg. per hour of propane are separated from the isobutane. Two possibilities of further treatment have been found:

(a) To polymerize this propylene and separate the polymer.

(b) To polymerize the propylene in a special plant and add it to the benzine.

In respect of (a): This procedure is not suitable because there occurs a mixed polymerisation of butylene and propylene, and the yield of iso-octane is decreased.

In respect of (b): Leuna has already polymerized this propylene in the laboratory, whereby they obtained a polymer having an octane number of about 55-60. Under these circumstances it seems more suitable to mix the propylene-propane mixture with the fuel gas.

E. C₃-C₂ Column

A refrigerating capacity of about 200,000-250,000 cal. per hour is necessary for this column. Normally the condensation of the ethane would take place at about -5°. It is however also possible to condense the ethane at +5° and the corresponding pressure. In the latter case the ammonia of 0° provided in the refrigerating machine of the T52 plant can be removed directly and conducted to the C₃-C₂ column to cool the ethane to +5° and condense it. It would be advisable to provide twice the usual number of condensers for this column in order to be able to change over immediately in the event of disturbances. According to the Firma Borsig such a method of operation is also possible with the refrigerating installation. Part of the ammonia can be directly branched off to the C₃-C₂ column, and the remainder can be employed for cooling the refrigerating water of the T52 plant.

The specification of a suitable degree of purity in the C₃-C₂ column was also discussed. It makes little difference in the operating costs whether the product is obtained in 90% or 97% purity. In the latter case only two more column trays are necessary. For this reason a purity of 97% is recommended. The general development tends toward a further chemical treatment of the propane and ethane as well. Therefore, in any new plant: for a special installation for obtaining a separation of C₃ and C₂ hydrocarbons, it is advisable to provide for the possibility of obtaining a separation of the two products in pure form.

Mixing the ethane with the fuel gas can be effected in that the gaseous ethane is mixed with the liquid C₃-C₄ solution. Since in doing this the heat of condensation of the ethane is liberated, it is desirable to pump the C₃-C₄ mixture in the circuit and to add the ethane ahead of the cooler. Such an arrangement has the further advantage that the pumping and mixing can easily be stopped at the instant the desired composition of the fuel gas has been reached.

The propane-propylene mixture obtained in the column of the T52 plant is preferably not sent through the C_3-C_2 column as the methane contained therein causes a disturbance in the column. Instead, the fuel gas should be added after the column. In the event that there will later be necessary a sharp separation of all of the fractions including the propylene mixture, the propylene would have to be gasified and introduced into the hydrocarbon gas in order that the methane will be removed in the benzene separation.

The space required for the C_3-C_2 column amounts to about 10 x 10 meters.

F. Tank-Level Measurements

The measurement of the level in the pressure tanks of the T52 plant was discussed.

(a) According to Dr. Weis the Weis eye is not suitable for measuring such a tank, since it is, in the case of deviations of the middle position, highly sensitive to temperature in the end position, and gives errors up to 10%. A temperature compensation can up to now be satisfactory only for the middle position. For this reason the Weis eye can be satisfactorily employed, for example, for maintaining the tank level.

(b) Level measurement by injection of nitrogen is technically reliable. However, it must be determined whether nitrogen is desirable in the product.

(c) Injection of vaporized product gas is also possible. The manometer and the inlets would have to be suitably heated. Such a measurement can be still further simplified by operating according to the Hampson principle. In this the liquid product is withdrawn from the tank at the bottom, and immediately vaporized. The second pipeline is removed from the gas space above. The Hampson connection is patented by the Linde company, and is employed by Linde in all of their installations.

(d) In the case of measurement with a level tube it must be taken into consideration that the product present under its own vapor pressure is readily vaporized in the level tube, and thus makes the readings unreliable. It is therefore necessary to provide wide lines to the level tube.

(e) A reliable measurement is obtained if a float is allowed to ascend and descend in the tank, and its movement is transmitted to the outside either mechanically or magnetically. In the T52 hydrogenation plant at Leuna I have seen in operation such a measurement with mechanical transmission. According to the manager it works satisfactorily.

G. Hydrogenation

The T52 hydrogenation plant at Leuna was inspected and the conditions in the furnace and regenerator were calculated from data in the operating books. In the hydrogenation of iso-octane at Leuna the following periods can be distinguished:

(a) In 1938 pure di-isobutylene was hydrogenated and pure iso-octane (trimethylpentane) was produced.

(b) Starting in April of 1939 Leuna received permission from the

Ministry of Aviation to add 18% tri-isobutylene.

(c) In August 1939 it was permitted to add 25% tri-isobutylene to use up the old supplies.

The following operating conditions occurred at the Leuna plant in the three cases:

Hydrogenation, T52, Leuna

	A	B	C
	Pure di-, No tri-, no tetra- iso butylene (Liquid, % by Volume	82% di-, 18% tri-, no tetra- iso- butylene Engler)	75% di-, 25% tri-, trace of tetra- isobutylene
200 ϕ Furnace			
Date	Sept. 1938	May 1939	Feb. 1940
Throughput, l/hr.	1000	1000	900
<u>Throughput</u>			
Contact volume	5	5	4.5
Specific gravity	0.725	0.735	0.740
Throughput in kg./hr.	725	735	660
Fresh hydrogen, approx. m ³ /hr 15°/735	190	230	240
Quantity of recycle gas, m ³ /hr, approx.	10,000	5500-7000	?
Temperatures:			
Regenerator outlet	202°C	190°C	174°C
Preheater outlet or furnace inlet	202°	199°	192°
5 Measuring places in furnace	202° 253° 262° 264° 264°	200° 241° 273° 276° 276°	204° 280° 296° 296° 296°
Regenerator inlet	264°	276°	296°
Regenerator outlet	77°	95°	104°
Temp. rise in furnace	62°	77°	104°
Pressure in furnace, atm.	200	200	200
Pressure differential in generator, atm.	6	9	9
Bromine number of inlet product	10,000 = 100% olefins		
Inlet prod. hydrogenated to	1 = 0.01% olefins		
(Hydrogenation is regarded as satisfactory up to a bromine number of 20 - Dr. Fischer of Leuna)			

Calculation of the Leuna conditions on the basis of our Scholven plant shows that we could operate autothermally with the regenerator of 500 ϕ and 12 m length as long as the regenerator is new, and overloading is not required. At a 30% overload there would probably be necessary a supplementary preheating. Heating to 180° is possible with the Scholven steam of 15 atm, but not to the 200° inlet temperature which has hitherto been employed at

Leuna. For this reason Leuna has agreed to operate the hydrogenation plant at 180° inlet temperature to establish whether this is possible.

The provision of a gas-heated preheater would not pay as it can be assumed that the chamber operates autothermally at normal operation.

A change-over from a 500 ϕ to an 800 ϕ regenerator would only give a slight improvement since the gas velocity becomes correspondingly less. The 500 ϕ regenerator has 125 square meters of exchange surface, and the 800 ϕ regenerator has 238. As a result of the lesser velocity, the 125 square meters of the 500 ϕ regenerator corresponds to 205 of the 800 ϕ regenerator, so that the gain would only amount to an increase of 205 to 238 square meters, and hence an improvement which would not justify the higher capital cost. For this reason it has in the meantime been agreed between Leuna, Uhde, and ourselves to provide for the Scholven T52 installation one regenerator of 500 ϕ and 12 meters in length, and one similar reserve regenerator.