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Interregional Petrochemical Symposium on the
Development of the Petrochemical Industries in
Developing Countries

Baku, USSR, 21 - 31 October 1969

PET. SYMP. B/6

PROCESSES FOR THE PRODUCTION OF PETROCHEMICAL BASIC BUILDING BLOCKS [1]

by

H. Höfermann
H.K. Kamptner
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presented by

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SUMMARY

PROCESS FOR THE PRODUCTION OF PETROCHEMICAL BASIC BUILDING BLOCKS [1]

by

H.K. Kamptner
Hoechst-Uhde International GmbH
Bad Soden
Federal Republic of Germany

In this paper the author discusses some of the processes now in operation or being developed which enable petrochemical building blocks to be made from raw materials others than naphtha. Recently developed methods for producing of [illegible] purer "building blocks" from [illegible] are also described.

(1) Production of ethylene from crude oil by a fluidized flow process. Preheated crude oil is injected into the fluidized bed of petroleum coke and [illegible] at [illegible]. The advantage of the fluidized flow process over the other fluidized bed processes [illegible] the use of crude oil [illegible] of ethylene, [illegible] propylene, [illegible]

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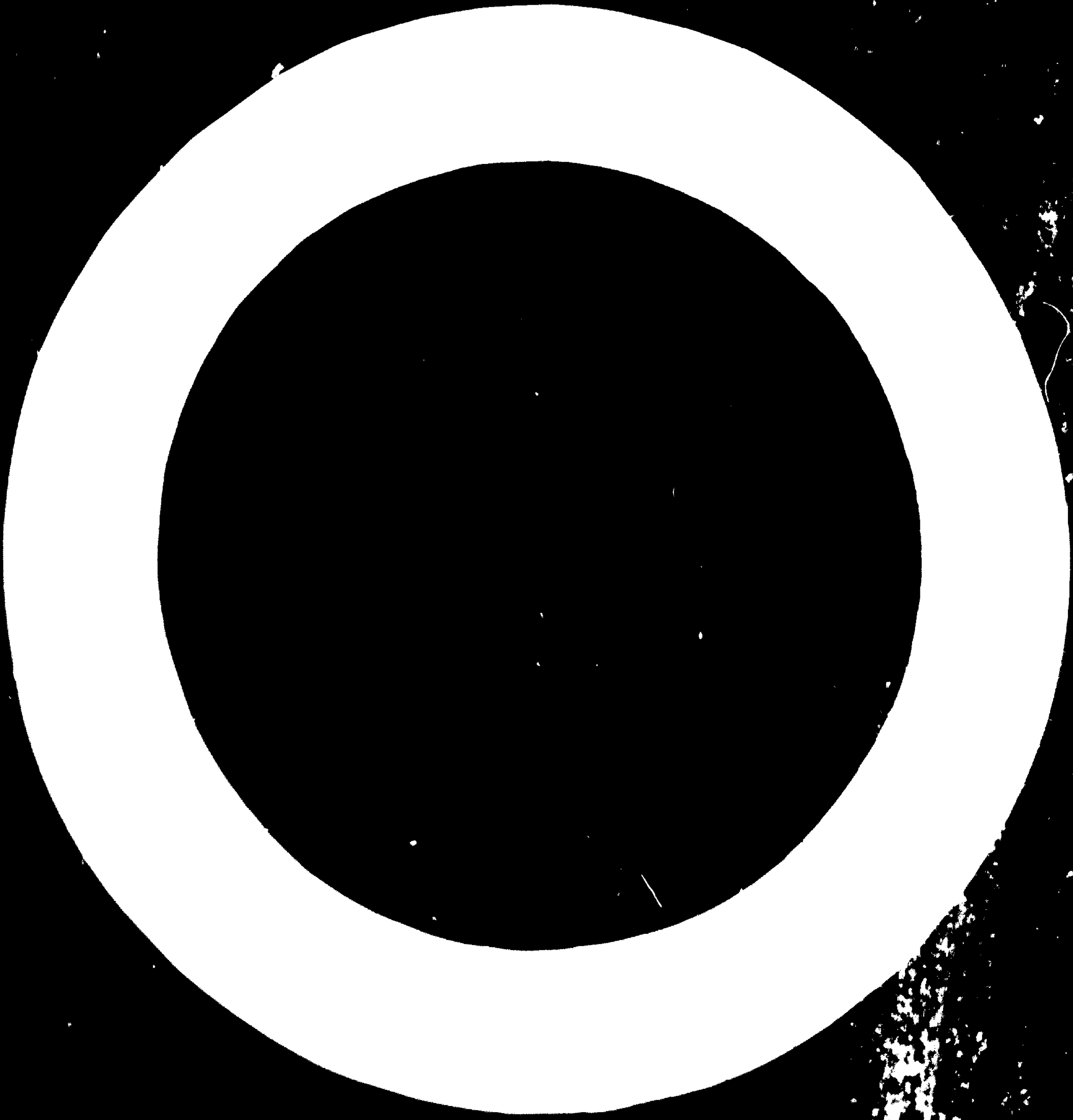
[illegible]



C_4 stream (55% butadiene), and 0.45 tons of pyrolysis naphtha.

(2) Production of acetylene and ethylene by the submerged flame process. In this process oxygen is, as it were, burnt underneath the surface of the oil, as a result of which cracking of the oil at a very high temperature in the neighbourhood of the flame takes place, the large volume of oil leading to rapid quenching of the products of cracking and hence retention of a considerable quantity of acetylene. The details of the process and its economics are described. 1 ton of acetylene and 1.18 tons of ethylene are obtained from [illegible].27 tons of crude oil and 5.1 tons of oxygen.

(3) Crude oil cracking by the Hoechst [illegible] process. This is at the development stage. The principle lies in the combustion of the tail gas with oxygen followed by the injection of secondary steam and the petroleum fraction to be cracked. The temperature is controlled by the quantity of petroleum injected which itself determines the acetylene - ethylene ratio. The process is fully described including an economic assessment.

(4) The B.A.S.F. process for the manufacture of acetylene. The current process is described in which natural gas, liquid gas or naphtha is cracked by the heat developed from the incomplete combustion of the raw material with oxygen.

(5) The hydrogen electric arc process. This enables a much higher yield of acetylene from hydrocarbon raw material to be obtained. Hydrogen is used as a carrier gas. The techniques of avoiding soot and the burning of electrodes are described.

(6) Purification and conversion of olefin fractions by hydrogenation. The propylene fraction is cold hydrogenated to remove methylacetylene and propadiene for the production of polymer grade propylene. Various combinations of these operations are discussed and evaluated. If there is no demand for propylene it can be fully hydrogenated and returned to the cracking plant.

Butadiene is normally removed from the C_4 stream (see section 7).

The residual stream must be hydrogenated to free from the last traces of butadiene. Di- or triisobutene produced in the Bayer oligomerisation process can be hydrogenated by the Bayer cold hydrogenation process to very pure isoparaffin.

(7) The NMP process of BASF using N-methylpyrrolidone with 5 to 10 water to extract butadiene from the C_4 stream is described in full detail with costings.

(8) Isobutene is sometimes required as a monomer, e.g. for the production of rubber polymers. The BASF process for its recovery via sulphuric acid treatment is described.

(9) The BASF isoprene extraction process using N-methylpyrrolidone is described. This is still at a development stage.

(10) The cold hydrogenation refining of a pyrolysis naphtha to free the product from acetylene and diolefines to make it suitable as motor gasoline is described.

(11) The BASF-Scholven process for the gas phase hydrogenation of pyrolysis naphtha is described. This removes sulphur and olefins and makes it suitable for the extraction of aromatics.

(12) The extraction of aromatics by N-methyl pyrrolidone from pyrolysis naphtha by the Lurgi-Arosolvan process is described in considerable detail and fully evaluated.

(13) Aromatics can be dealkylated e.g. toluene converted to benzene by the BASF-Scholven process. Toluene and crude hydrogen e.g. from a catalytic reformer are converted catalytically to benzene and methane. Recirculation of unconverted toluene is carried out and about 1.5% diphenyl produced as a by-product.

Contents

The raw materials which are suitably used for the production of the petrochemical basic building blocks such as ethylene, acetylene, propylene and the C_4-olefins, will always be adapted to the given local conditions. These interrelations were decisive for the fact that two large chemical companies in the Federal Republic of Germany, independently of one another, developed processes for the cracking of full range crude oils. Cracking was carried out in the middle temperature region (below 900° C) for the production of the olefins ethylene, propylene and butylenes. These processes are known as the BASF fluidized bed process and the Hoechst moving pebbles process.

Compared with the USA, the pattern of consumption of petroleum products is completely different in Europe, and especially so in the Federal Republic of Germany. This also has a bearing on the selection of raw materials which are available for the manufacture of petrochemical basic building blocks. In the USA, [illegible] % of the ethylene is made from ethane, [illegible] % from propane and only [illegible] % from liquid petroleum hydrocarbons (condensate, naphtha). In Europe, the ethylene requirement is obtained nearly exclusively from the thermal cracking of naphtha. Prior to the large-scale production of ethylene as an aliphatic basic building block in the Federal Republic of Germany, a special position was occupied by acetylene obtained from calcium carbide. This development must be considered in order to understand that there was a special interest in [illegible] processes for the manufacture of acetylene from petroleum [illegible] for naphtha for ethylene production and the striving for independence from the refineries in respect of raw material supply [illegible] the interest in crude oil cracking for producing petrochemical basic building blocks in the chemical industry during recent years.

The wide experience with electrothermal processes also triggered the development of hydrocarbon cracking using the hydrogen-electrical arc technique. Special attention was directed in the same way to the development of processes for the recovery of individual olefines of very high purity grade. Technological contributions have also come from our country with regard to separation of the aromatics from the previously hydrogenated pyrolysis naphthas.

In the following, it will be reported on the processes being operated on a commercial scale already, as well as those which are still in developing stage.

1 The BASF fluidized flow process for the production of ethylene from crude oil

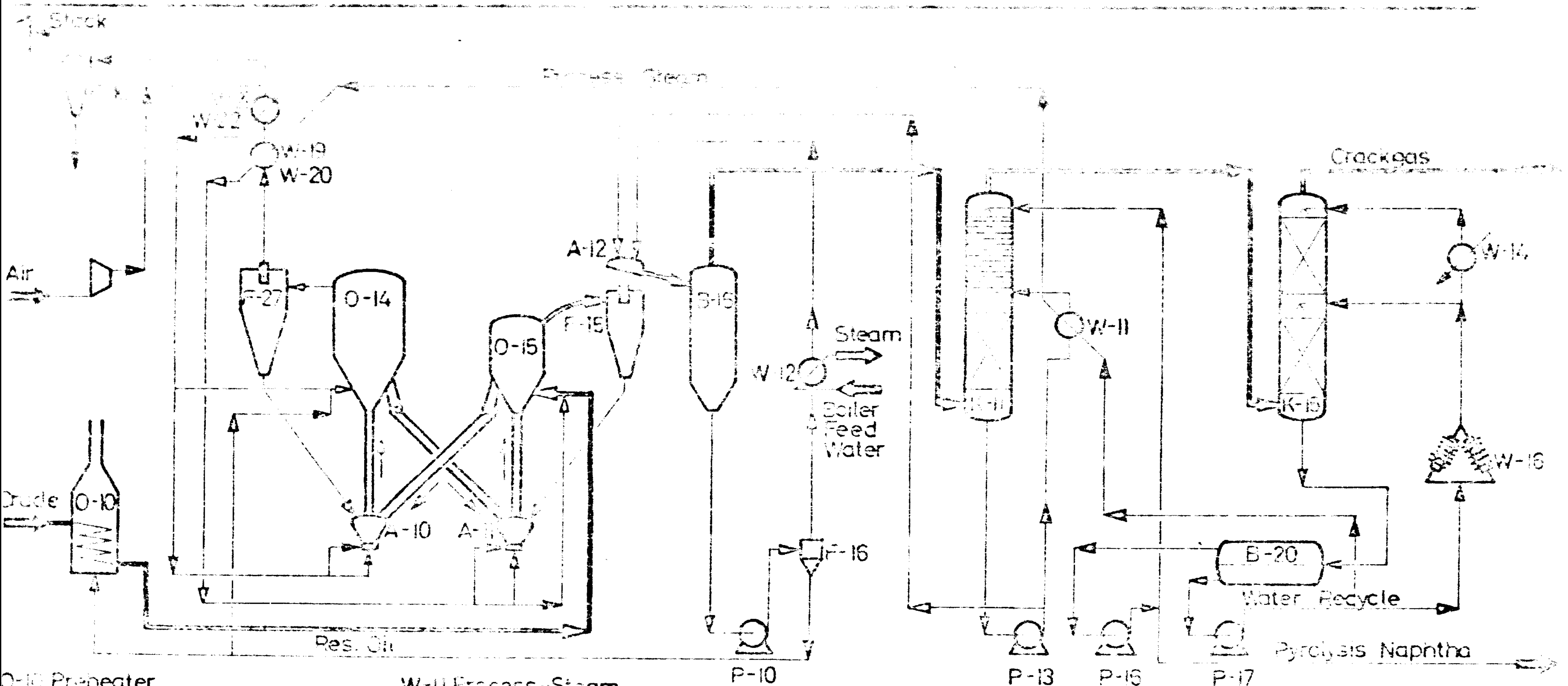
1.1 Description of the process

The production of ethylene from crude oil is of interest because it represents a method which is independent of naphtha prices.

The BASF fluidized-bed process requires oxygen for supplying the heat of reaction. The reaction takes place at about 750 °C in a fluidized bed of petroleum coke. Steam serves as the fluidizing medium to which oxygen is added. The heavy fractions of the reaction products are recycled to the fluidized bed and are burnt there together with part of the petroleum coke.

The cracked gas is diluted with flue gas. This, as well as the need for oxygen and the [illegible] equipment are a disadvantage of the process.

The fluidized-flow process circumvents these drawbacks by carrying out



O-10 Preheater
O-14 Regenerator
O-15 Reactor
P-10 Quenchoil Pump
Bottoms-Pump
Reflux-Pump
Water Recycle Pump

W-11 Process-Steam Generator
W-12 Heatexchanger
W-14 Cooler
W-16 Aircooler
W-19 } Steam
W-20 } Superheater
W-21 Air-Preheater

W-22 Air-Preheater
A-10 Liftpot
A-11 Liftpot
A-12 Quench-Head
B-16 Quenchoil-Gas Separator
B-20 Refluxdrum

F-10 Multiclone
F-15 Quench-Cyclone
F-16 Hydro-Cyclone
F-27 Regenerator Cyclone
K-11 Crackgas-Column
K-15 Direct-Cooling System

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Crude-Oil - Fluidized Flow Cracking (BASF-Process)

Fig I

the generation of heat and the cracking reaction in separate parts of the cracking unit. In this way, air can be used as the combustion agent. The flue gas escapes into the atmosphere which means that crackgas separation [illegible]. On the other hand it is necessary to circulate large amounts of heat carrier between the reactor and regenerator. This heat carrier circulation is the basic difference between this process and the fluidized bed process, and required many years of development work.

An inorganic material, as spherical as possible, is used as the heat carrier whose abrasion under operating conditions must be low. A mullite with a grain size between 0.5 and 2 mm has proved the most suitable material.

The main processing steps can be taken from Fig. 1. The heat carrier circulates between the reactor 0-15 and the regenerator C-[illegible], in the direction indicated by arrows. The amount circulated is controlled by [illegible] and the pneumatic conveying starts up [illegible]. The conveying medium on the regenerator side is [illegible], on the reactor side, steam. The length of the sloping downpipes [illegible] dimensioned that reactor and regenerator are safely separated on the [illegible]. For this reason, the two large apparatuses have to be placed at a height of about 2[illegible] m.

In the [illegible] trials, fluidized beds with grids were used both for the reactor and the regenerator. However, there were difficulties as regards [illegible] and conveying of solids; this was best overcome by doing away with the grid and using a conical bed. The conical bed resulted in excellent operating conditions and did not entail any loss in yield compared to the fluidized bed with grid. However, it cannot be described as an actual fluidized bed any more. It is a [illegible] with powerful jolt [illegible], [illegible] central deflector [illegible] solids [illegible]. The preheated crude oil is [illegible], together [illegible] steam, through lateral [illegible]. About 0.7 ton of steam are used per ton of crude oil. Owing to the slight [illegible] in the subsequent equipment, the reactor operates at slightly [illegible] pressure. The cracking temperature lies between 700 and [illegible] C, the residence time in the reactor, as in the fluidized bed process, is a [illegible] seconds.

[illegible] oil [illegible] the process is used as fuel for the [illegible] generator; [illegible] for combustion [illegible] burns [illegible] oil feed [illegible] is thus heated [illegible] heat of the [illegible] for preheating [illegible].

In [illegible], a proportion of the [illegible] are removed with the gas from the fluidized bed. [illegible] is provided in the [illegible] unit in order to [illegible] of [illegible] and thereby to [illegible] apparatus [illegible] possible. The volume of the fluidized bed in the reactor and consequently the residence time [illegible] kept small in order to [illegible] secondary reactions. For this reason, about [illegible] of the circulated heat carrier are conveyed into the quenching cyclone [illegible] and are recycled from there to the [illegible] circulation. There is no [illegible] of erosion here since it is possible to use SiC-bricks [illegible] under operating conditions, the coking is [illegible] hours.

[illegible] are placed over the fluidised bed, [illegible] fluidised bed. [illegible] of the plant shown [illegible] [illegible] being arranged [illegible] corresponding [illegible] [illegible]

[illegible] where [illegible] 1 shows [illegible] product [illegible] are [illegible] keep the solids [illegible] is built into the [illegible] is removed from [illegible] the crude oil [illegible] regenerator [illegible]

[illegible] cracked gas [illegible] below K 11 and is [illegible] fluidised bed process. [illegible] taken off at 0.5 atm gauge [illegible], and fed to the [illegible] plant.

Pyrolysis naphtha is formed as a by-product which is similar to the corresponding product obtained in the tubular-furnace cracking process.

[illegible]

[illegible]

[illegible]

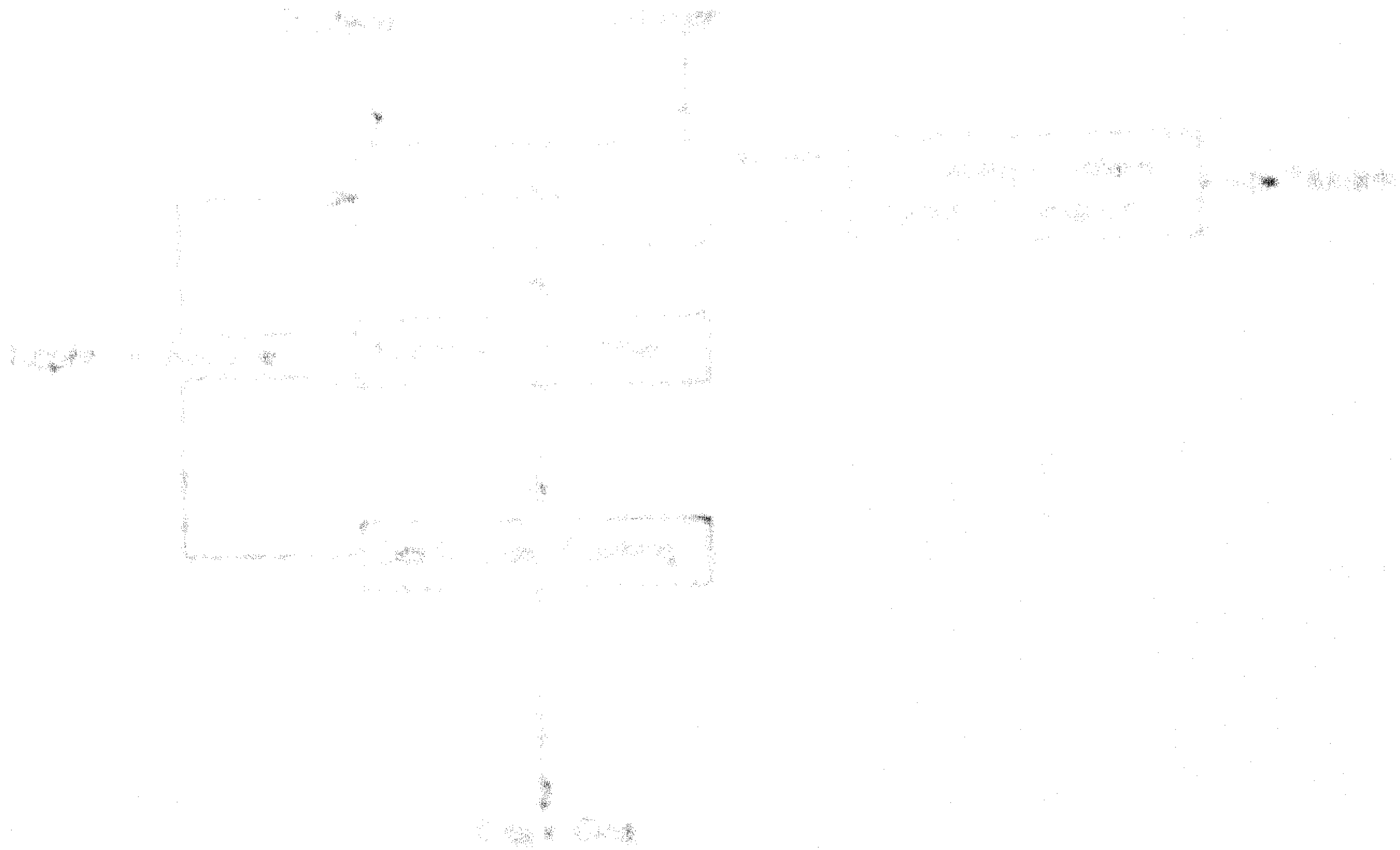
[illegible]

[illegible]

[illegible]

[illegible]

If [illegible] will be used, [illegible] m.t. of crude [illegible] per m.t.



[illegible]

[illegible]

[illegible]

Fig [illegible]

of ethylene produced. The following yields of by-products are obtained:

C_3-cut (95 % propylene)	0.50 ton/ton C_2H_4
C_4-cut ([illegible] % butadiene)	0.25 " "
Pyrolysis naphtha	0.45 " "

The residual oil formed is burnt quantitatively in the process, whilst an excess of 0.7 ton/ton C_2H_4 of fuel gas (H_2 + CH_4) remains.

If

the C_3-cut is valued at DM 165/m.t. (4 DM = 1 US $)
the C_4-cut at DM 105/m.t.
the pyrolysis naphtha at DM [illegible]/m.t.
and the fuel gas at DM 80/m.t.

a manufacturing price of DM 0.21/kg C_2H_4 (2.4 US Cents/lb) is arrived at, [illegible] the investment costs of DM 170,000,000 (US $ [illegible]) [illegible] depreciation, taxes, interest and maintenance.

[illegible]

[illegible] Chem.-Ing.-Techn. 36 (1964) No. 9, pp. 889-898
[illegible] 44 (1965) 3, pp. 134-142

2. [illegible] Acetylene and Ethylene by the BASF [illegible]

[illegible] acetylene and ethylene [illegible] basis for several [illegible] processes has been developed for this purpose [illegible] flame process. [illegible] variant [illegible] cracking [illegible] oil; vacuum residues [illegible]

2.1 [illegible] the cracking section consists in the [illegible] of [illegible]

[illegible] burning in an [illegible]

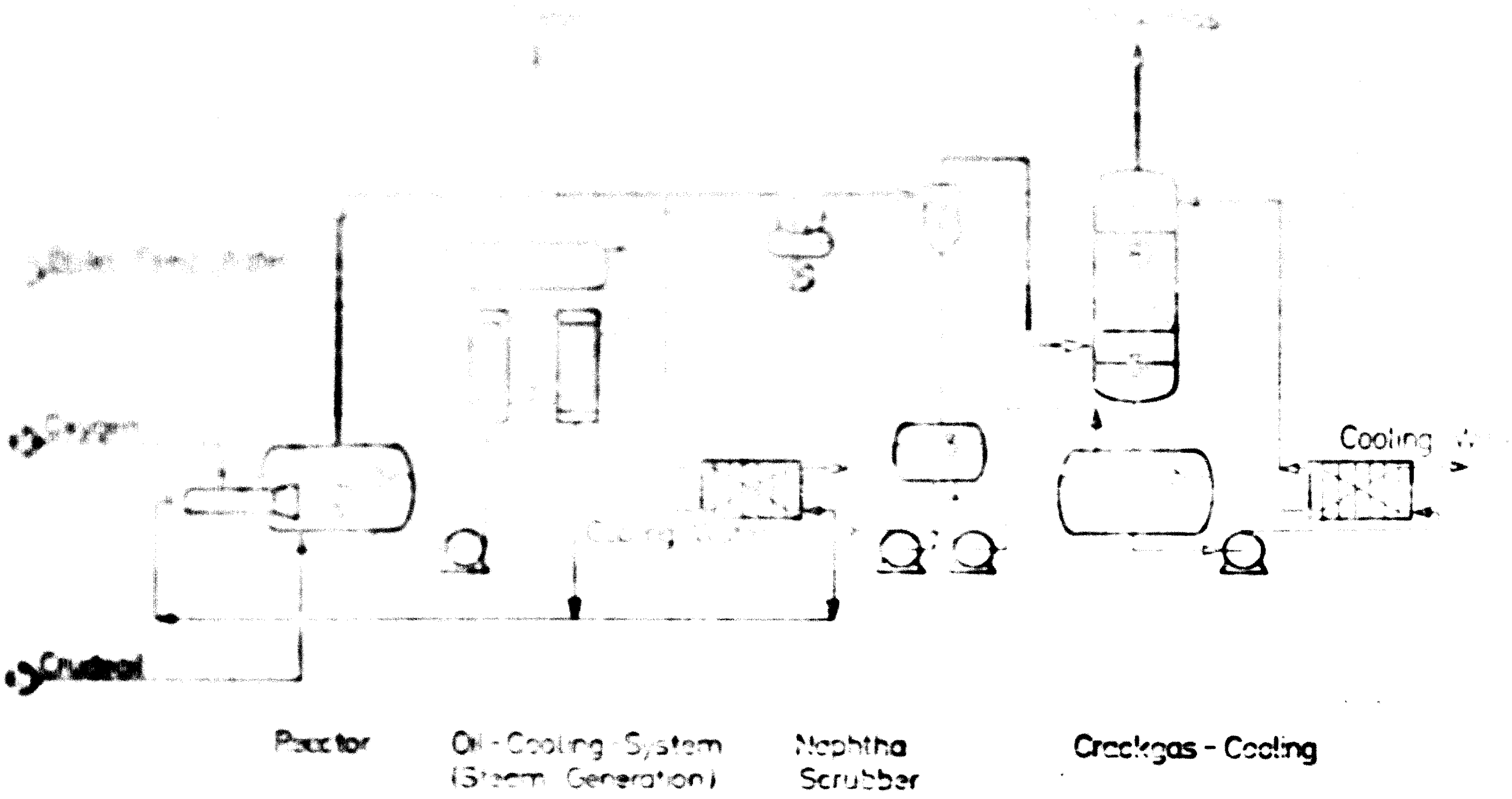
[illegible] generation

[illegible]

[illegible]

[illegible] process (see Fig. 3) takes place in an oil bath [illegible], with [illegible] supplies the necessary energy.

[illegible] quickly quenched by the oil surrounding [illegible] equilibrium [illegible] is frozen. [illegible] an oil bath temperature of [illegible]



Submerged Flame Process

Fig. 4

Cracking-Section (BASF)

There are two kinds of gas separation processes for the production of acetylene [illegible] as can be seen from the fig. 5. As regards investments [illegible], there is very little difference between [illegible] employ a scrubber immediately following the crack [illegible]. Thus, the major part of the CO_2 and all the H_2S are [illegible].

In [illegible] I, first the remaining CO_2 is removed with sodium [illegible] and the gas subsequently dried with methanol. By [illegible], the [illegible] and higher hydrocarbons are condensed and [illegible] CO, H_2 and CH_4. The [illegible] hydrocarbons by distillation [illegible], the acetylene [illegible] the C_2-distillate [illegible] concentrated in a countercurrent column and separated [illegible].

[illegible] II, the [illegible] scrubber is followed by an N-methyl-[illegible] which the acetylene together with the C_3- and [illegible], concentrated in a counter-current column [illegible]. [illegible] pure acetylene, [illegible] which the C_3- and [illegible] in the acetylene following its concentration, [illegible] remains in the lean gas in the [illegible]. The ethylene is removed from it by partial [illegible] temperature plant and recovered by distillation.

2.2 Some technical data (Feedstock: crude oil)

a. Purity of the products

	Acetylene % by vol.	Ethylene % by vol.
Purity	99.8-99.9	99.9

b. Investment (based on conditions in the Federal Republic of Germany)

Total cost of a plant for 30,000 metric tons/yr. acetylene (turn key, but without site development and auxiliary plants such as air separation, power station, tank farm, etc.)

DM 40,500,000.-- (US $ 10,125,000)

c. Consumption of utilities per 100 kg acetylene

Separation process		I	II
Electricity	kWh	280	277
cooling water (25° C/Δ t = 12°)	m³	29.5	30.5
Boiler feed water	metric ton	0.97	0.96

d. Consumption of materials per 100 kg acetylene

[illegible]		I	II
Crude oil	kg	827	827
Oxygen	m^3	510	510
Scrubbing agent for CO_2 removal	kg	0.4	0.4
NaOH ([illegible]) calculated as 100 %)	kg	4	4
Na [illegible]	kg	0.3	0.3
Acetone	kg	0.15	-
[illegible]	kg	-	0.5-1
Scrubbing [illegible] for C_3-, C_4-[illegible]	kg	-	0.35
Compressed air	m^3	25.	25
Nitrogen	m^3	50	50

e. By[illegible] per 100 kg acetylene

[illegible]		I	II
Steam (more than [illegible]0 atm. gauge)	metric ton	0.85	0.5
Ethylene	kg	118	118
Lean gas [illegible] value [illegible]	m^3	1135	1135
(CO, H_2, [illegible] CH_4, <0.3 ppm S)			
C_3- and C_4-hydrocarbons	kg	92	92

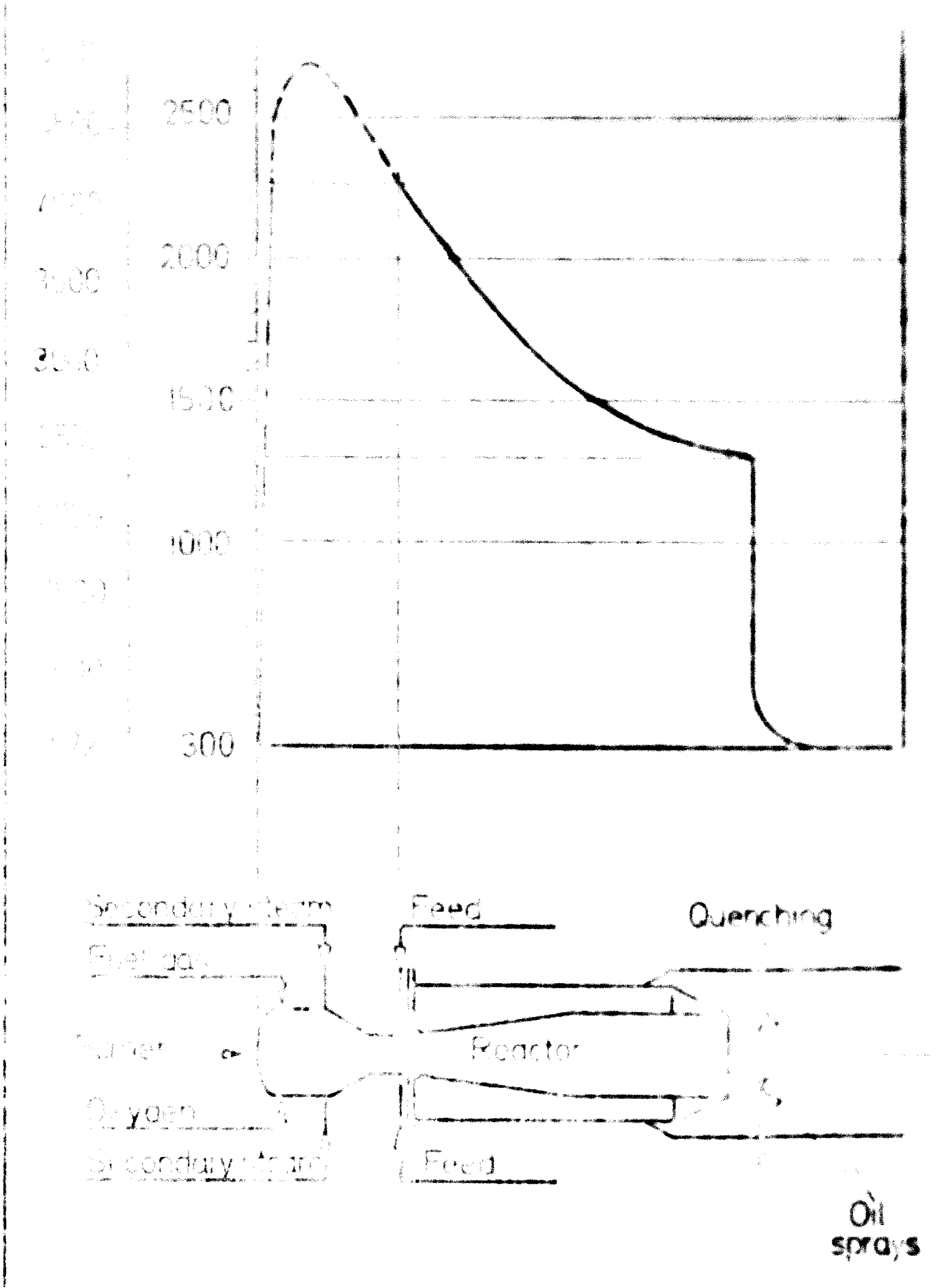
Literature references:

H. Kröger, [illegible] Hintz, Erdöl und Kohle 16, 537/540 (1963);

Proc. 6th World Petroleum Congress, Vol. 4, 289/294 (1964);

Proc. 7th World Petroleum Congress, Vol. 5, 233/247 (1967);

H. Fris, Chemie-Ingenieur-Technik 40, 995/1004 (1968).



Temperature profile of the
HTP-Reactor System (Hoechst)

Fig [illegible]

[illegible]

[illegible]

[illegible]

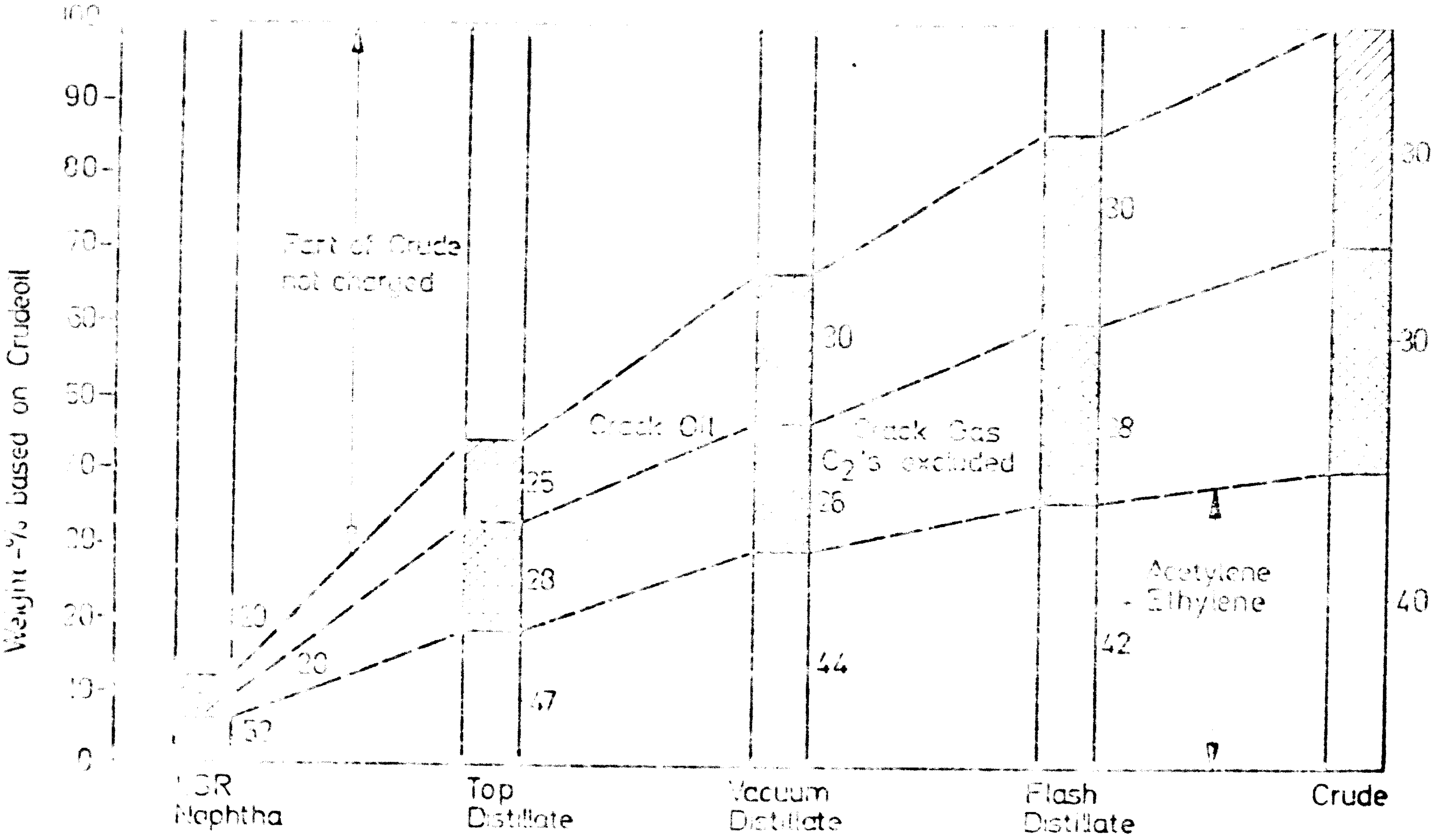
[illegible]

[illegible] other reaction conditions being equal, the more the [illegible] is shifted in favour of acetylene. A final cracking [illegible] correspond approximately to a weight [illegible].

[illegible] temperature the cracking equilibrium is [illegible] from the process and the cracked [illegible]). A [illegible] [illegible] only [illegible] three thous- [illegible] reaction tube. The [illegible] of the tail gas [illegible] the [illegible] clearly [illegible] virtually [illegible] precisely [illegible]. These condi- [illegible] which operates [illegible].

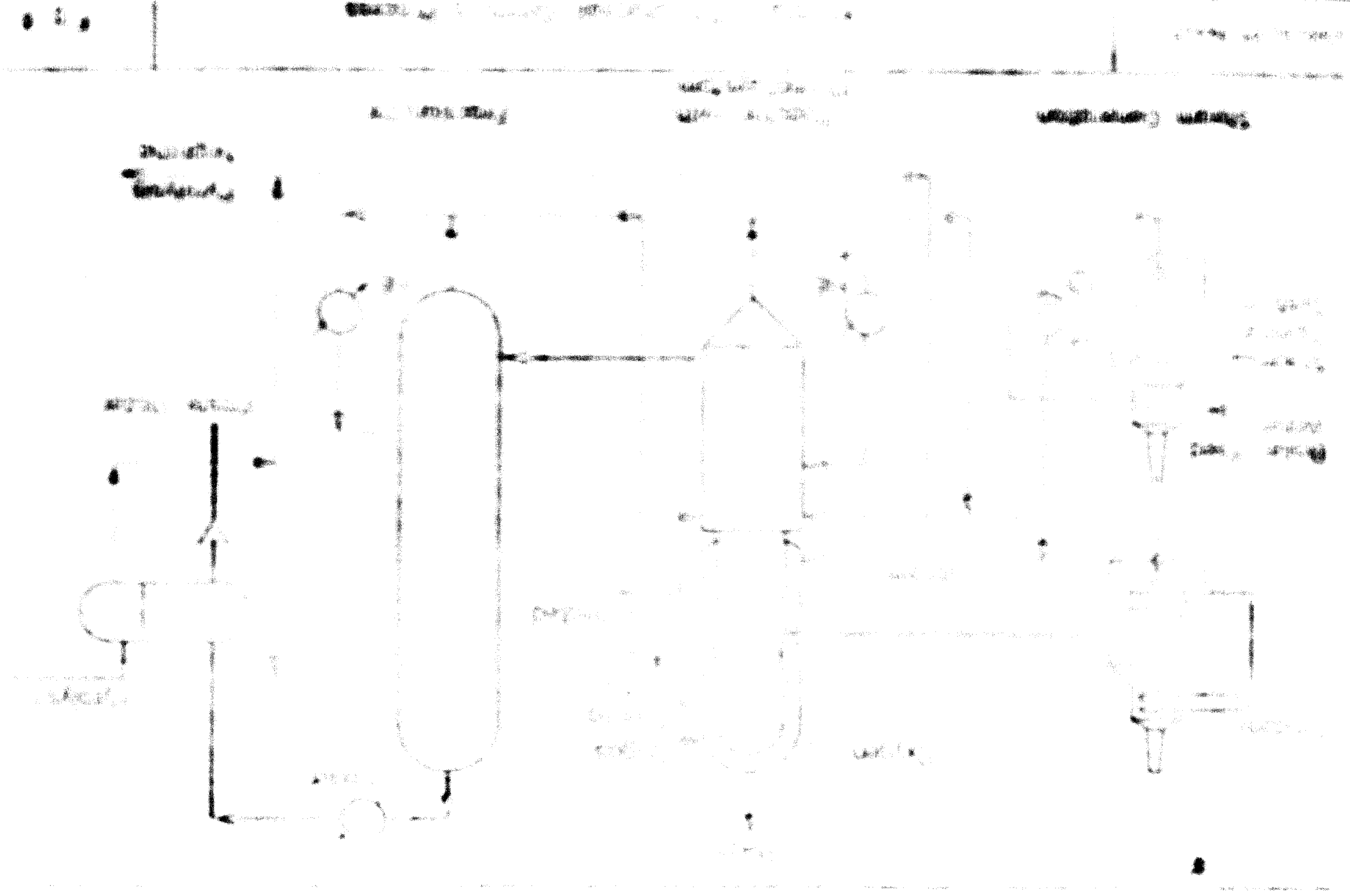
[illegible]

[illegible] number of crude- [illegible] represented [illegible] naphtha, [illegible] atmospheric [illegible] from which [illegible] (Aramco). The [illegible] by weight [illegible] cuts. The [illegible]



Pattern of Products obtained by the Cracking of Crudeoil and its Fractions (Hoechst-HTP-Process)

Fig 7



[illegible] by the [illegible] The [illegible] This [illegible] operation [illegible] the [illegible]

[illegible] for [illegible] in [illegible]

[illegible]

[illegible]

[illegible] 000

[illegible] 000

[illegible]

[illegible]	A	B	C	
[illegible]	[illegible]	[illegible]	[illegible]	DM/metric tons
[illegible]	[illegible]	[illegible]	40	DM/1000 kWh
[illegible]	4	20	40	DM/1000 m³
[illegible]	[illegible]	7	7	DM/[illegible] m³

[illegible] DM/1000 kg total [illegible]

[illegible]	A	B	C
[illegible]	[illegible]	17.38	17.38
[illegible]	[illegible]	[illegible]	0.83
[illegible]			
[illegible]	[illegible]	4.70	[illegible].27
[illegible]	0.10	0.10	0.10
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	[illegible]
[illegible]			
[illegible] deducted [illegible]	-[illegible].04	- 1.64	- 1.64
[illegible]	[illegible]	[illegible]	[illegible].69

[illegible] DM 30/1000 kg the following prices thus result for

[illegible]			
[illegible]	46.37	50.30	[illegible]4.22
([illegible] cents/[illegible])	5.3	5.7	6.2

Literature references:

H.K. Kamptner 6th World Petroleum Congress, Sect. IV - Paper 12-PD 8. 1963
H.K. Kamptner Chemical Engineering
W.R. Krause Feb. 28, 1966
H.P. Schilken
B. Schnack Symposium Acetylene-Ethylene DECHEMA-Frankfurt, March 1968

1. The BASF Process for the manufacture of acetylene by partial oxidation of hydrocarbons

2. Principle of the process:

Gaseous or completely vaporizable hydrocarbons (natural gas, liquified gas or light gasoline) are preheated and mixed in the gaseous state with oxygen [illegible] the hydrocarbons. The [illegible] channels, and [illegible] by means of [illegible] chamber, [illegible] flame zone, [illegible] a few milliseconds, are quenched to about 80°C [illegible] The cracked [illegible] soot from [illegible] is [illegible] soot formed [illegible] passes into [illegible] water [illegible] is separated by flotation. The [illegible] is separated from the soot-free gas, following compression [illegible] by scrubbing selectively with N-methylpyrrolidone and by fractionated desorption from the solvent.

The main by-product formed is a gas consisting essentially of carbon monoxide and hydrogen which, for example, can be used for ammonia synthesis.

The acetylene produced has a purity in excess of 99 [illegible] which, if required, can be increased to 99.5 [illegible] by [illegible] unit.

4.2 [illegible]

At present, [illegible] metric tons/year [illegible] produced from natural [illegible] process, [illegible] acetylene from [illegible]

[illegible]

[illegible]	[illegible] per shift	[illegible] per shift
[illegible]	[illegible] m³	[illegible] m³
[illegible]	[illegible] kcal/m³	[illegible] kcal/m³

[illegible]

[illegible] to obtain the [illegible] to [illegible] temperature [illegible] to [illegible] on [illegible]

[illegible] of [illegible] Further [illegible]

[illegible]

6.

temperature it is possible to vary within certain limits the ratio of acetylene/ethylene in the cracked gas according to requirements.

A carrier gas, [illegible] hydrogen, is heated to above 3000°C in an [illegible] injected into the heated [illegible]. It is consequently a two-step process:

[illegible] (plasma)

[illegible] the plasma, cracking, [illegible]

The cracked gas [illegible] being [illegible]

[illegible]

[illegible]

and down, this is brought about by [illegible] and appropriate shaping of the electrodes.

[illegible] appropriate when [illegible] gives the [illegible] stock.

[illegible] constituents [illegible]

Table 11

[illegible]

[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
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[illegible]	[illegible]	[illegible]
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[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
	[illegible]	
	[illegible]	

[illegible]

[illegible]	0.1% by vol.	-
[illegible]	[illegible] % by vol.	62.[illegible] % by vol.
[illegible]	-	0.[illegible] % by vol.

[illegible]

[illegible]

[illegible] mainly [illegible] gradually [illegible] [illegible] [illegible] [illegible].

[illegible] Recovery of pure propylene from pyrolysis C_3-cut

[illegible] consists almost wholly of propylene [illegible] Besides propane [illegible] methylacetylene and propadiene. [illegible] practi- [illegible] with [illegible] of propylene [illegible] applica- tion [illegible] of this method with the [illegible] of high purity [illegible] propylene from the pyrolysis [illegible] which [illegible] from [illegible] hydrogenation [illegible] catalyst [illegible]

[illegible]

[illegible]

Column	[illegible]	2	1
	I.D. mm	1,100	1,400
	[illegible] meter	250	130
Reflux ratio		12.9	10.0
Utilities: electricity	kWh/h	700	50
[illegible]	[illegible]	610	60
Investment	[illegible]	1,000,000 4,300,000	[illegible]
Purity [illegible]	percent	10	0.01

b) [illegible] extraction

[illegible] hydrocarbons [illegible] refined [illegible] hydro- [illegible] quantities [illegible] temperature [illegible] treated [illegible] content [illegible]

Data on analysis of fresh feed and product, on hydrogenation conditions, on utilities' consumption and investment are given in table 2.

Table 2

Full hydrogenation of pyrolysis C_3-cut

Reaction temperature	°C	30 —— 60
Pressure	atm. gauge	35
Fresh feed space velocity *	m.t./m^3 catalyst.hr	7
Recycle: fresh feed		2 : 1

Analysis before and after hydrogenation

Component	mol % Feed	mol % Product
C_2	0.14	0.14
methylacetylene + propadiene (C_3H_4)	4.31	< 20 ppm
propylene	91.19	0.62
propane	4.17	98.94
C_4^+	0.19	0.30
	100.00	100.00

Fresh Feed	metric tons/yr	100,000
Utilities: H_2 (80 Vol %)	m^3/hr	9,300
: Refrigeration energy for the removal of heat of reaction	10^6 kcal/hr	8
: cooling water	m^3/hr	60
: electricity	kWh/hr	50
Investment	US $	205,000
Catalyst charge	m^3	1.8

6.2 Hydrogenation of minor amounts of Butadiene remaining in C_4-raffinate

The [illegible] C_4-[illegible] rich in butadiene so that in general pure butadiene [illegible] therefrom. The remaining C_4-cut (raffinate) still [illegible], mostly in the range of 0,5–1,0 %. Also C_4-cut [illegible] – i.e. the [illegible] often contain butadiene [illegible] range. For [illegible] further chemical treatment this [illegible] disadvantageous so that it is recommended to [illegible] butadiene. [illegible] hydrogenation has proven [illegible]. The mode of operation is the usual one for the cold [illegible], but especially adjusted to the [illegible] to be fulfilled here. [illegible] It can be seen that the butadiene in the feed is nearly [illegible] hydrogenated, but that practically [illegible] hydrogenated to butane.

By [illegible] it is also possible to combine the [illegible] hydrogenation with nearly complete isomerization of n-butene-1 into n-butene 2. In performing the process in this way a distillative separation between isobutene and n-butene-2 becomes possible: in distillation [illegible] product [illegible] together [illegible] the non-isomerized [illegible] together with n-butane are taken as bottoms. Thus a large part of the butene in the original feed is realized as butene-2 for further chemical processing.

Table 3

Selective [illegible]

Reaction temperature	°C	25 — 60
Pressure	atm. gauge	12.6
Fresh feed space velocity	m.t./m³ catalyst.hr	20
Recycle: fresh feed		0 : 1

Analyses [illegible] hydrogenation

Component	parts by weight inlet	 outlet
Butanes	9.2	9.7
Isobutene	45.5	45.5
n-butene-1	27.6))44.9	25.3))44.8
n-butene-2	17.3)	19.5)
Butadiene	0.4	200 ppm
	100.0	100.0

[illegible]	[illegible]	[illegible]
[illegible]	m^3/hr	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	6.5
[illegible]	[illegible]	6.5
Investment	[illegible]	[illegible],750
[illegible]	[illegible]	0.[illegible]5

[illegible]

After [illegible] the pyrolysis [illegible] there [illegible] of this hydro[illegible] -isobutene (TIB) [illegible] used for the [illegible] for further processing [illegible] full hydrog[illegible] The hydro[illegible] hydrogenation technique. Tabe 4 shows [illegible] procedure.

The [illegible] pure isoparaffins, absolutely free from [illegible] of great advantage in the use of special solvents, mainly [illegible] of the production of [illegible] The hydrogenated products [illegible] high octane number and [illegible] added to hydrogenated pyrolysis [illegible] by [illegible] raising the motor octane [illegible]. [illegible] the hydrogenated [illegible] can be used as motor gasoline [illegible].

Table [illegible]

Conditions of hydrogenation [illegible]

Reaction temperature	°C	150 — 220
Pressure	atm. gauge	40
[illegible] velocity	m^3/m^3 catalyst.hr	2
Recycle [illegible]		1 : 1

Analyses before and after hydrogenation

Specification	Units	DIB (C 8) feed	DIB (C 8) hydrogenated product	TIB (C 12) feed	TIB (C 12) hydrogenated product
bromine number	g/100g	[illegible]	0.005	16[illegible]	0.01
content of aromatics	% wt	0	0	0	0
initial boiling point	°C	98	98.5	174	169
50 Vol% distilled	°C	100.5	99.5	178	175
final boiling point	°C	101	100.5	181.5	179
Octane numbers RON, unleaded		99.8	99.1	99.8	100.6
+1.5 cc TEL/US-gal		100.4	101.2	100.3	102.2
MON, unleaded		89.2	97.0	88.8	98.0
+1.5 cc TEL/US-gal		91.1	101.0	89.8	102.4

Steam [illegible] kg/h		[illegible]	[illegible]
[illegible]		[illegible]	[illegible]
[illegible]		[illegible]	[illegible]
[illegible]		[illegible]	[illegible]
[illegible]		[illegible]	[illegible]
Investment	[illegible]	[illegible]	[illegible]
Cat[illegible]		[illegible]	[illegible]

The [illegible] hydrogenation [illegible] plants in operation, [illegible] about [illegible] metr. tons/yr.

[illegible] ion.

a. [illegible]

[illegible]

[illegible]

[illegible]

[illegible]

7. The [illegible] for the recovery of butadiene from [illegible]

Butadiene [illegible] by-products formed during the prod[illegible] the cracking [illegible]. For the manufacture of [illegible] the following "poly-cis quality" [illegible] total acetylene under 50 ppm [illegible] ppm. For producing [illegible] butadiene from [illegible] allene containing pyrolysis C_4-cuts, a process

[illegible]

[illegible]

[illegible] exchanger ([illegible] methyl[illegible] [illegible] pure [illegible] still [illegible]

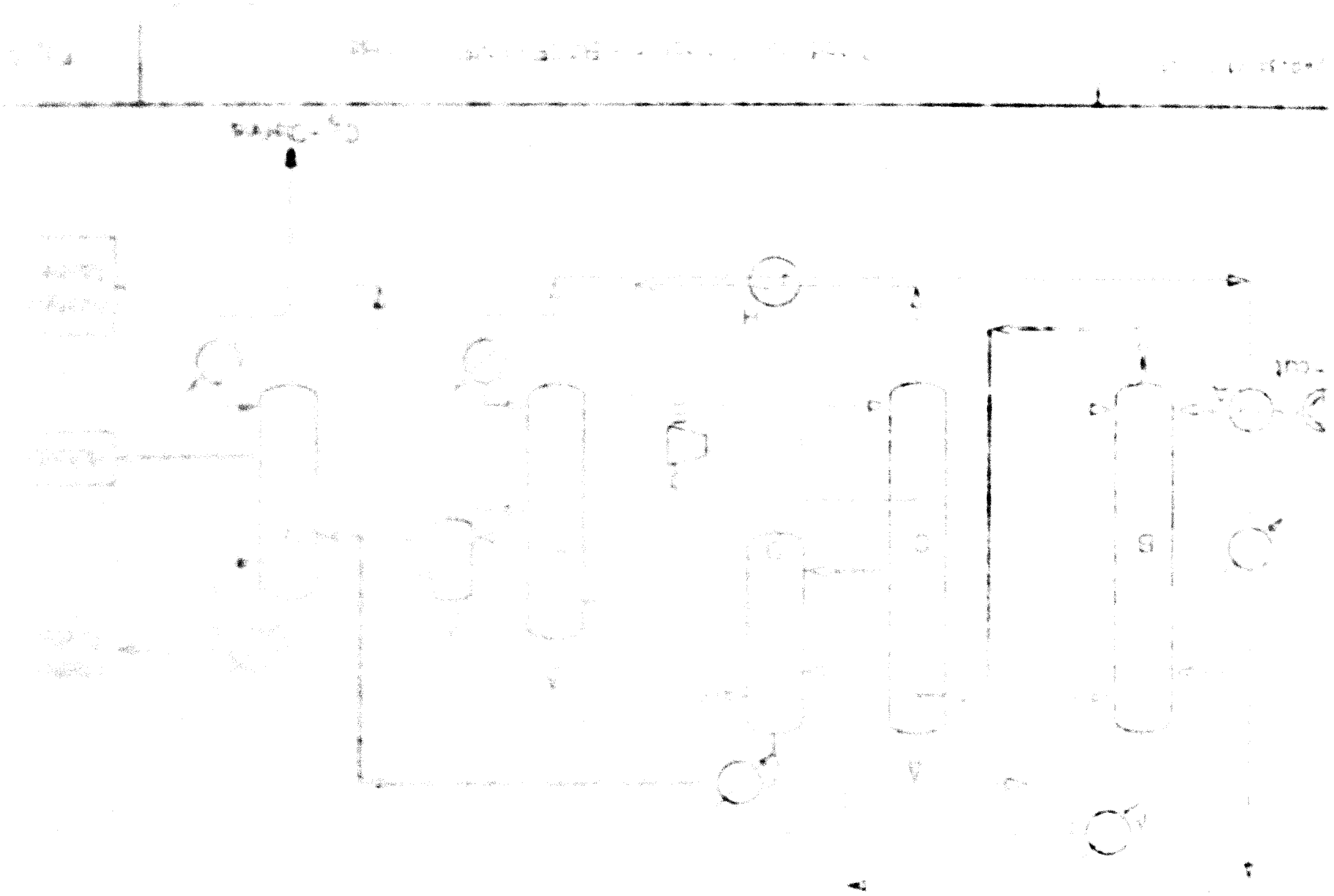
[illegible] from the [illegible] column (D). [illegible] and [illegible] considerably [illegible] outlet of [illegible] methylacetylene [illegible] of these two [illegible] there-

[illegible]

[illegible] vessel.

[illegible] separation and [illegible]

[illegible] about [illegible]. The operating temperatures [illegible] depend on the [illegible] pressures and are above the critical temperature of the hydrocarbon. The process can



[illegible]

[illegible]

[illegible]

[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	0.8	[illegible]
[illegible]	[illegible]	
[illegible]	[illegible]	[illegible]

[illegible]

25. I. 72

2 OF 2

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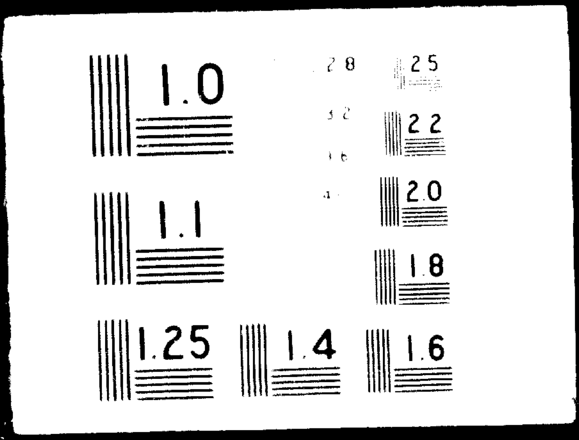


[illegible] Petroleum Congress 1963,

[illegible] IV [illegible]

[illegible]

[illegible] 93

[illegible]

[illegible] hydrocarbon

[illegible]

[illegible] high-grade [illegible] starting [illegible] the

[illegible]

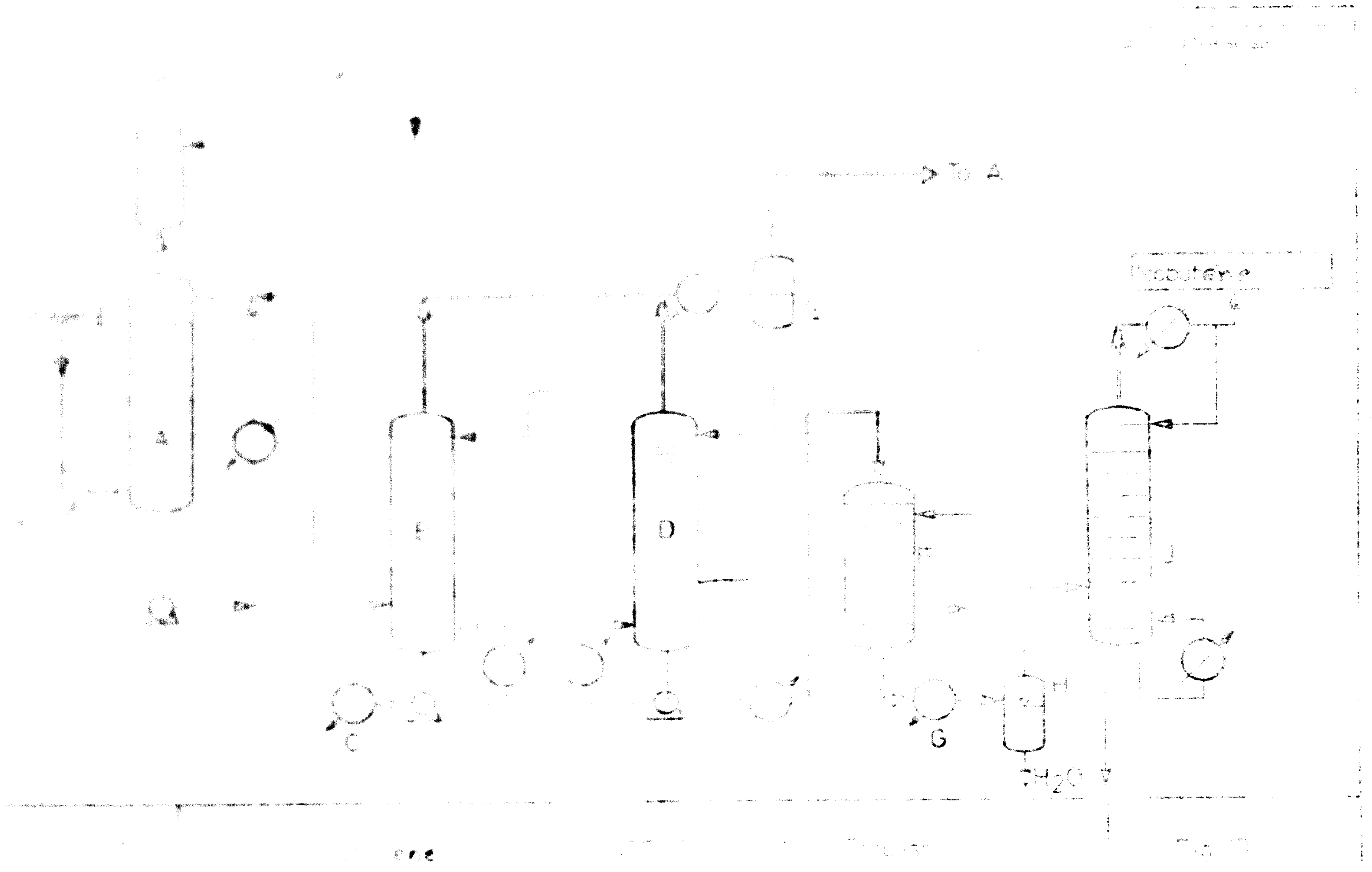

Isobutene
To A
A
B
C
D
E
G
H2O

Table 1 - continued

Butadiene-1,3	43.7	- 4.41°C
Others	1.1	-

The process developed by BASF for the recovery of isobutene makes it possible to use all isobutene - containing pyrolysis C4-cuts as feedstock and on the other hand produces an isobutene that satisfies the highest known purity requirements (99.9 % isobutylene). The yield is 90 - 95 %, very little diisobutene is formed (less than 1 % calculated on the isobutene).

The isolation of isobutene from butane-butene-butadiene mixtures such as those from cracking light naphtha pyrolysis, is not possible by normal distillation because of the proximities of the boiling points (see Table 1).

Since no highly selective solvents for the economical separation of isobutene [illegible] are known there is only one possibility of isolating [illegible] utilizing the great chemical reactivity of isobutene [illegible].

Of [illegible] reactions, the most suitable chemical reaction for the isolation [illegible] by the addition of water to tertiary [illegible] product.

[illegible] process

The [illegible] recovery of isobutene (Fig. 10) can be divided [illegible] absorption, isolation and if necessary, distillative purification of the tert.-butanol, dehydration and rectification of the isobutene. The isobutene is selectively absorbed from the gaseous butene mixture at about 30°C in a bubble column A with the aid of the sulphuric acid-containing absorption solution. Tert.-butanol is being formed. The absorption solution flowing from the absorber passes into a distillation column B in which the tert.-butanol formed is distilled at reduced pressure as an azeotrope (with water). The absorption solution is recycled to the absorber after cooling in C.

The absorption solution flowing into the distillation column contains a small amount of physically dissolved n-butenes. The overhead vapors of the column therefore contain, besides the tert.-butanol-water-azeotrope small amounts of n-butenes which are separated by distillation in a second, smaller column. These n-butenes as well as small amounts of isobutylene formed in the bottoms of column B by dehydrolysis of the tert.-butanol, are separated from the tert.-butanol-azeotrope in D and recycled to A. The tert.-butanol-azeotrope is drawn off from the bottoms of D and introduced into a heated dehydrating furnace F; dehydration takes place in the presence of activated aluminium oxide at 250°C. The reaction mixture condensed in condenser G is separated in separator H into the two reaction products isobutene and water. The isobutylene is then freed from traces of higher boiling contaminants (diisobutene, tert.-butanol) by fractionating in column J.

8.2 Investment, consumption figures and manufacturing costs

Investment (Inside Battery Limits)

DM 4,500,000 (US$ 1,125,000)

(Price in Federal Republic of Germany, without taxes, engineering fee)

Feedstock:

Butene mixture (50 wt % isobutene)	13,320 metric tons/yr
Operating hours per year:	8 000

Product

Product isobutene:	6 000 m.t./yr
Isobutylene:	99.9 %
n-butenes:	< 0.05 %
tert.-Butanol + diisobutene:	< 0.01 %

Yield:

Consumption figures (per ton isobutene)

Steam (4 atm. gauge)	2.5 m.t.
Electricity:	500 kWH
Natural gas:	140 m³
Cooling water: (25°C)	400 m³

Operating costs calculation	DM per m.t.
Steam (DM 6/m.t.)	15.00
Electricity (DM 4/100 kWh)	20.00
Natural gas (DM 6.5/100 m^3)	9.10
Cooling water (DM 2/100 m^3)	8.00
Chemicals	2.00
Operating costs	DM 54.10/m.t. isobutene US $ 13.525)

Capital costs (4 year capital return) DM 1,125,000/yr

i.e. DM 187.50/m.t. isobutene

Maintenance (4 % of investment costs/yr): DM 180,000/yr

i.e. DM 30 / m.t. isobutene

Personnel (2 men per shift) DM 10 /hr

DM 13.35 /m.t. isobuten

Feedstock (isobutene in the butene mixture):

DM 100 /m.t.

Manufacturing costs: DM 385 /m.t. isobuten

(US $ 96.25)

Isoprene Extraction [illegible] Process

Following [illegible] on the recovery of butadiene from pyrolysis C_4-cuts it was [illegible] whether the [illegible]-process is also suitable for the recovery of isoprene from pyrolysis C_5-cuts. It was found that isoprene of high purity and in a good yield can be recovered from C_5-cuts (Table 1). It was found to be advisable to place an NMP liquid-liquid extraction step before the NMP counter-current gas scrubbing step.

Table 1

Typical analysis of a cyclopentadiene-containing pyrolysis C_5-cut

	wt %
[illegible]-hydrocarbons	0.9
[illegible]	26.1
[illegible]	24.1
[illegible]	4.2
[illegible]	11.8
[illegible]	13.7
[illegible]	1.6
[illegible]	0.1
[illegible]	8.8
[illegible]	7.5
[illegible]	1.2

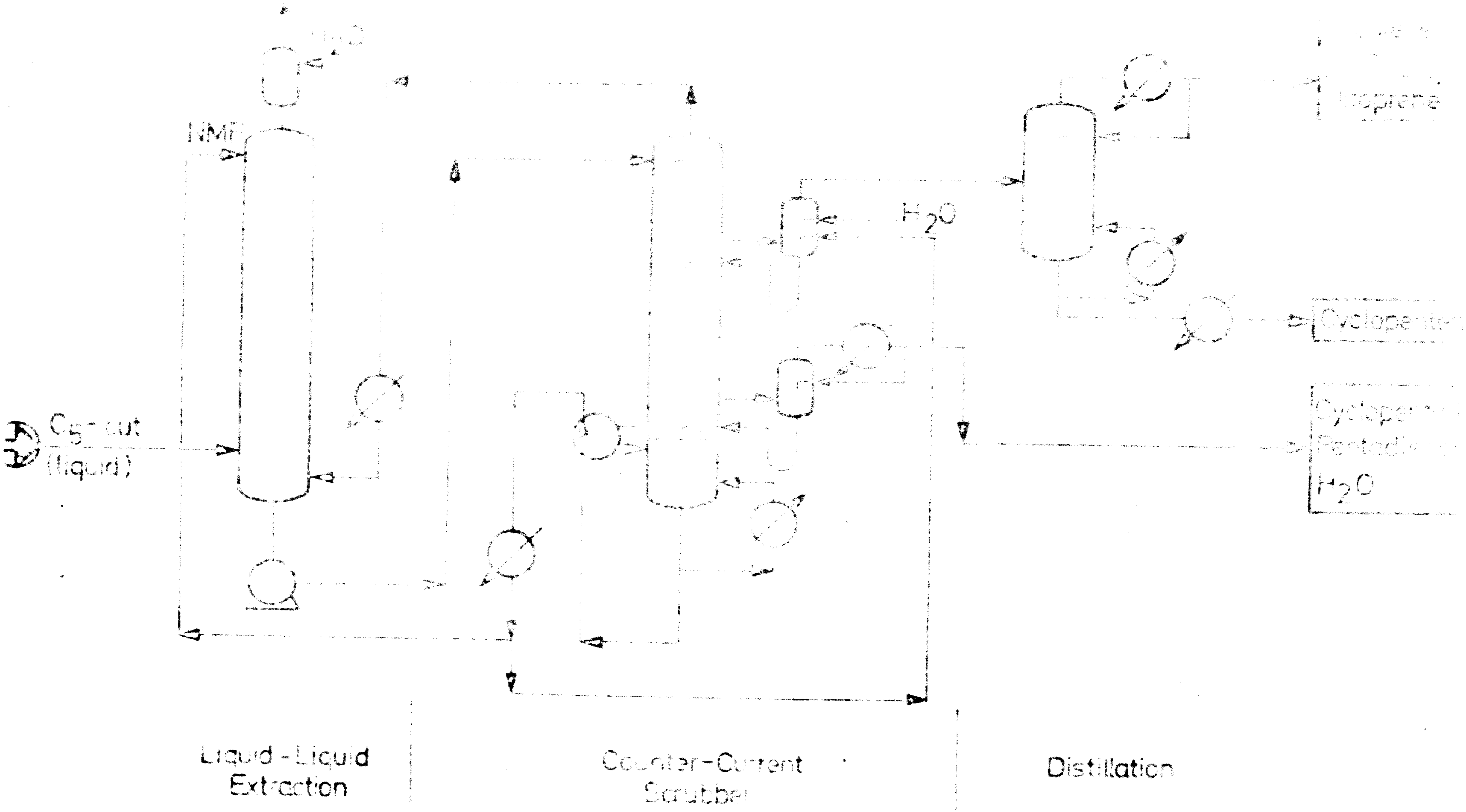
[illegible]

[illegible] of the pyrolysis C_5-cut [illegible] to 14 % on average - [illegible] to recover the isoprene economically from [illegible]

[illegible] counter-current gas scrubber - [illegible] in the case of this [illegible] - the required amount of circulated solvent, corresponding to the isoprene concentration, is dependent on the amount of C_4-cuts to be used as feedstock. In the case of isoprene recovery, therefore, a relatively large plant would be necessary in relation to the moderate isoprene amount. In addition, the entire C_5-cut would have to be vaporized which in turn would require a large amount of steam for heating. In comparison, however, to separate the pentanes which are contained in amounts between 40 and 50 % in the C_5-cut, a considerably small amount of solvent would be needed for a liquid-liquid extraction by the use of water-containing N-methylpyrrolidone. This makes it possible economically to absorb the total isoprene including the other C_5-diolefins in N-methyl-pyrrolidone by means of such a liquid-liquid extraction. A proportion of the pentenes is dissolved in the N-methylpyrrolidone at the same time.

The N-methylpyrrolidone which contains the isoprene, the other C_5-diolefins and part of the pentenes, as shown in the flow sheet (fig. 11), is freed from the dissolved hydrocarbons by introducing it into the head of a distillation column, heating and boiling in the usual way in the bottoms of this column. Thus in this column, under the conditions of countercurrent scrubbing, the isoprene is separated from the other accompanying components. This separation process, in contrast to separation of the pentanes, requires a relatively large number of trays, but this is quite feasible in a distillation column.

The amount of solvent was considerably reduced by removal of the pentanes in the preceding liquid-liquid extraction. The mixture of pentenes and a small amount of isoprene collected as overhead of the column is condensed



NMP-Process (BASF)

Fig. 11

and recycled at a suitable level into the liquid-liquid extraction column. Corresponding to their solubility, the C_5-hydrocarbons accumulate at [illegible] levels in the column from which they are drawn in the usual way as side streams.

The isoprene is removed as the uppermost side stream. If the isoprene [illegible] contains excessive amounts of pentadienes, cyclopentadiene or C_5-acetylenes, i. e. compounds with higher solubility, these are removed from the isoprene in an additional scrubber, as has already been shown analogously for the removal of acetylene in the butadiene recovery.

[illegible] is not sufficiently selective for separation of the system isoprene cyclopentene. Since the boiling point difference of these two hydrocarbons is, however, 10°C, i. e. the relative volatility is high, a separation of the accompanying component from the diolefin is possible without any difficulty, like in the system butadiene ethylacetylene.

The studies aiming at employing this process on an industrial scale have not yet been completed. However the results obtained so far indicated that the costs for the recovery of isoprene of high purity from cracked [illegible] with the [illegible] are very favorable.

[illegible] Refining process of Pyrolysis Naphtha-Liquid-Phase "Cold Hydrogenation"

If one regards the pyrolysis of naphtha as a severe thermal reforming, then the pyrolysis naphtha is a thermal reformate which has to be refined to meet motor gasoline specifications. Thus indeed the use of the pyrolysis naphtha as motor gasoline blending component is a common one.

From the general view points, given above, it is quite understandable that the conditions [illegible] for the refining of the [illegible] is achieved [illegible] indefinite [illegible] they start [illegible] operation. [illegible] pyrolysis [illegible] the [illegible] below [illegible] hydrogena- tion [illegible] residual material [illegible] com- [illegible] ion.

[illegible] pyrolysis [illegible] pyrolysis [illegible] periodical regeneration [illegible] possible.

Table [illegible] figures for the [illegible] of pyrolysis naphtha [illegible] the figures [illegible] hydrogenated [illegible] resistant [illegible] hydrogenation, [illegible] hydrogenation, the content of aromatics [illegible] constant.

For re[illegible] from the hydrogenated product the correspondin fraction [illegible] to be hydrogenated [illegible], in order to remov

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]	°[illegible]	[illegible]
[illegible]	atm. gauge	[illegible]
[illegible]	[illegible]/m^3 catalyst/[illegible]	[illegible]
[illegible]		[illegible]

[illegible]

[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]		[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]		[illegible]	[illegible]
[illegible]		[illegible]	[illegible]
[illegible]		[illegible]	[illegible]
[illegible]			
[illegible]		[illegible]	[illegible]
[illegible]		[illegible]	[illegible]

[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]		
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]

[illegible] start-up. After leaving the reactor, [illegible] mentioned, and in an [illegible] and freed in a [illegible] refrigerators.

[illegible] this is ne- [illegible] the earliest. Pure [illegible] less than 50 % by volume [illegible].

[illegible] be carried out [illegible] without intermediate [illegible] circulation [illegible] cooler. The waste gas [illegible].

[illegible] weight.

[illegible] to hydroaromatics less [illegible]

[illegible]

[illegible] of the feedstock, [illegible]

c) Consumption of utilities and materials:

Per one metric ton of pyrolysis naphtha employing combined air and water cooling

Electricity	20 kWh
Steam	0.1 m.t.
Cooling water	5 m^3
H_2-consumption	40 - 80 m^3
Catalyst consumption	0.02 kg

11.3 Economy

The calculation is based on a plant for an annual throughput of 100,000 tons of pyrolysis naphtha which is connected to a selective hydrogenation unit operating by the BAYER process (Federal Republic of Germany conditions)

Investment for the key-turn plant		DM	2,600,000
		US $	650,000
Interest and amortization	15 %		48,75
Maintenance	3 %		9.75
Personnel (1 man per shift) and supervision			7.50
Hydrogen (less caloric value credit for waste gas)			44.00
Utilities and other operating materials			32.00
Total manufacturing costs		DM/hr	142.00
		DM/m.t.	11.35
		US $/m.t.	2.34

Literature references:

O. Reitz, "Treating Aromatic By-Product Light Oils from Pyrolytic conversions by the L.G.-Scholven-Process for Light Oil Refining", Proceed. 5th World Petrol. Congress, Vol. III, Paper 17 (1959).

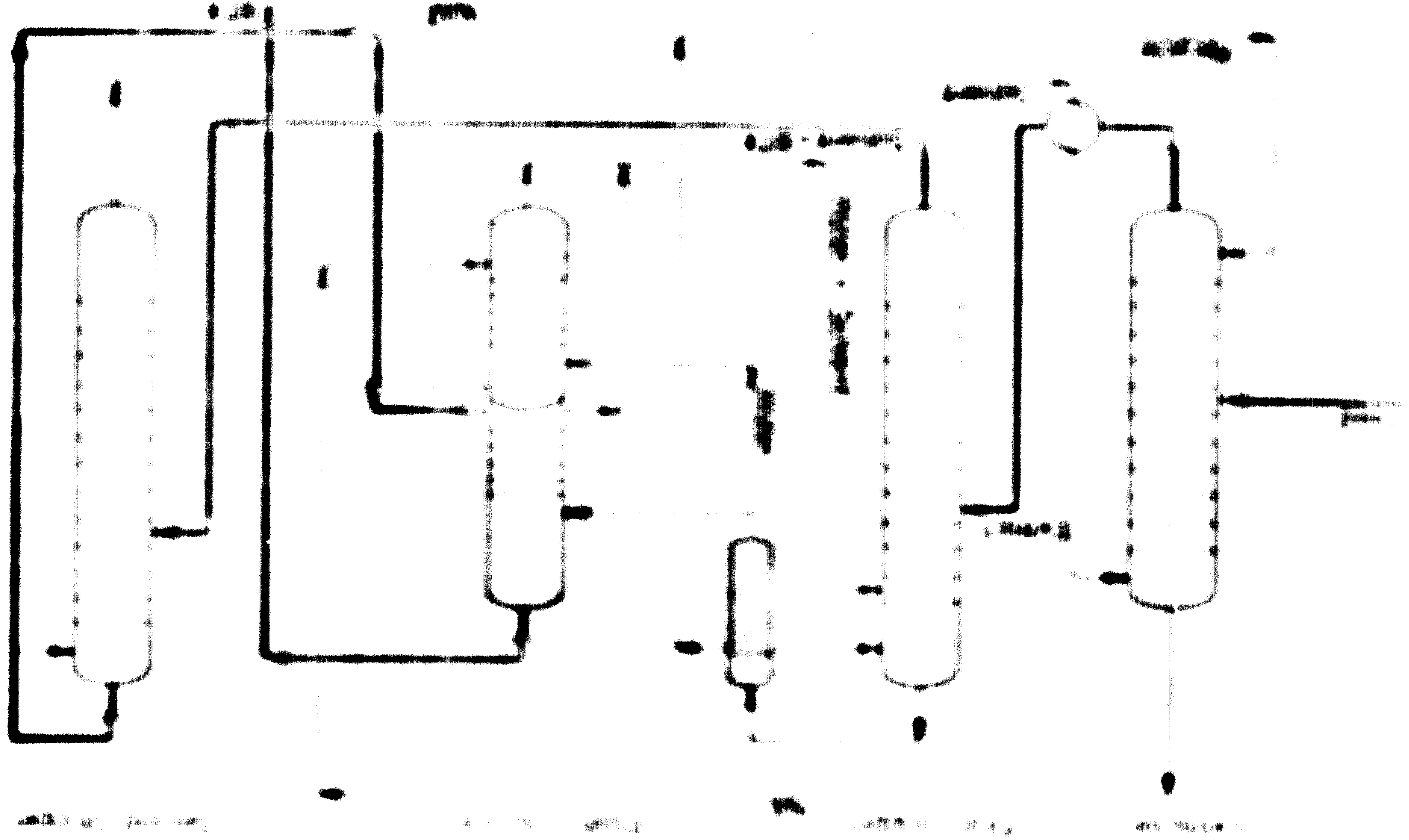
12. Pure Aromatics Extraction-Lurgi Arosolvan Process

The pyrolysis products of naphtha, compared with coal-tar light oil rich in aromatics, contain a much larger proportion of nonaromatics whose sharp separation from the aromatics by simple distillation would be uneconomical and impossible.

For this purpose Lurgi has developed the Arosolvan process by means of which, with the aid of NMP (N-methylpyrrolidone) as the solvent, aromatics of the highest purity are obtained in optimal yields and requiring the lowest power consumption.

The Arosolvan process was developed for the extraction of aromatics with NMP/water as the solvent mixture. Improvement of the selectivity of the solvent by the addition of water as the mixing component made it possible to produce aromatics in a high degree of purity. However, it was found that distillative separation of the solvent/aromatic mixture by water distilling over as the azeotrope with the aromatics causes a relatively high steam consumption.

Further development of the process resulted in using glycol as the mixing component. The choise of the solvent mixture NMP/glycol not only reduced



[illegible] accordingly, the [illegible] as [illegible]

[illegible] the [illegible]

[illegible]

[illegible]

[illegible] the [illegible] as in the [illegible]

[illegible] and [illegible]

[illegible]

[illegible]

[illegible]

[illegible] further treatment being necessary, the pure aromatics can be separated into the individual components by distillation. The nonaromatics [illegible] off at the top of the extractor column. They [illegible] removed by scrubbing in a column with [illegible] which contain some solvent, are drawn off as [illegible] transferred to the 1st stripper column. [illegible] separated in a separator into water and [illegible] flows into the scrubbers, the hydrocarbons flow [illegible]

[illegible]

[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible].860–0.870
[illegible]	[illegible]	[illegible]	–
[illegible]	[illegible]	[illegible]	–
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]

[illegible]

[illegible] pure aromatics in the extraction unit, based on a typical reformate or pyrolysis naphtha:

Steam (1[illegible]-16 [illegible] gauge): 0.3 m.t.

or equivalent amount of heat in the form of fuel oil or fuel gas.

Cooling water	20 m^3
Electricity	15 kWh
[illegible]	0.1 kg

The steam consumption for the fractionating of 1 m.t. of pure aromatics in a following simple distillation plant depends on the ratio in which the individual aromatics are present in the feedstock; it is known from experience to be between 0.[illegible] and 0.6 metric tons.

[illegible]

[illegible] economy, a calculation is given for a plant with an [illegible] metric tons. The amount of aromatics [illegible] metric tons/yr ([illegible]00 operating hours).

Investment [illegible]	DM 9,000,000 (US$ 2,250,000)
[illegible] ([illegible]ion)	15 [illegible]

[illegible] MT/hr

[illegible]

[illegible] = 11.[illegible] MT/hr

[illegible] = [illegible] /hr

[illegible]

[illegible] = [illegible] /hr

[illegible] = [illegible] /hr

[illegible] = 23.[illegible] /hr

[illegible] kg = [illegible] /hr

[illegible]

[illegible] = [illegible]

[illegible] price of the [illegible] for the credit for [illegible]

[illegible]

[illegible] aromatic component [illegible] be recovered, [illegible] the [illegible] below the [illegible] this [illegible] aromatics are [illegible]

[illegible] aromatics are separated again [illegible]

[illegible] from the extractive distillation column. [illegible]

Technical data:

Recovery of benzene from coaltar light oils with 1-5 % nonaromatics:

Yield:	95 - 98
Crystallization point:	5.45 - 5.5° C

Utilities per one metric ton of pure benzene:

Steam	0.8 m.t.
Cooling water	40 m^3
Electricity:	7.6 kWh
Solvent:	0.1 kg

[illegible]

[illegible] "Production of pure aromatics by means of azeotropic distillation and extraction"

Proceed. 4th World Petrol. Congress, Vol. 4, [illegible]/43 (1955)

[illegible] "Aromatics extraction with solvent combination"

Proceed. 5th World Petrol. Congress, Vol. 4, 13/20 (1959)

13. Hydrodealkylation [illegible]

Dealkylation in the presence of hydrogen has as its objective the splitting off of side chains and even saturated rings, from aromatic hydrocarbons in order to obtain from substituted compounds of which there is an excess, the required lower substituted compounds or preferably the unsubstituted basic compounds. Of the greatest importance today is the production of benzene from toluene and naphthalene from mono and dimethyl naphthalenes.

Work was already started by BASF in the early nineteen-forties on the catalytic dealkylation of benzene homologues (DBP 861.238, 1943). These studies which originally were directed especially toward the production of toluene besides benzene from xylenes, were taken up again when a surplus of toluene was created through the petrochemical manufacture of benzene.

An industrial process was evolved which, in a first plant with an annual throughput of 120,000 metric tons toluene is to go on stream at Scholven-Chemie AG in the middle of 1969. The process can also be employed for the dealkylation of naphthalene homologues; it is briefly described in the following by the example of the production of benzene from toluene.

13.1 Description of the process

Excess gas from catalytic reforming can be used to advantage as fresh gas for dealkylation instead of pure hydrogen. The fresh toluene together with the unreacted toluene and circulation gas is heated by heat exchange and in a pre-heater, and passed to the reaction zone which consists of two in-line reactors with regenerable catalyst. To limit a rise in temperature as a result of the high heat of reaction, quenchoil is added.

To remove the methane formed during dealkylation, part of the circulation gas, and in the case of reformer gas being used, the fresh gas itself, is conveyed to a hydrogen concentrating unit.

First the liquid reaction products are freed in a stabilizing column at elevated pressure from the constituents that boil at a lower temperature than benzene. In a subsequent column at normal pressure the pure benzene is separated from unreacted toluene. This recycle toluene contains small amounts of higher boiling products, especially diphenyl, which [illegible] topping. If the diphenyl fraction can be used, for example, as heat carrier oil, it can be recovered as an additional product. The valuation of this fraction is possibly higher than [illegible] by taking it out, plant and operating costs of dealkylation are lowered somewhat.

13.2 Technical data

a) Yields

(in the case of a toluene with 2 % weight C_8-aromatics and 0.2 % by weight nonaromatics):

81.5 % by weight pure benzene

1.5 % by weight diphenyl fraction

b) Purity of benzene

Crystallization point	+	5.5° C
Bromine consumption	under	0.01 g/100 g
Total sulphur	under	1 ppm
Toluene content	under	[illegible] ppm

c) Utilities and consumption of materials (for the dealkylation of 1 metric ton toluene with combined air and water cooling including distillation):

[illegible]	[illegible]
[illegible]	[illegible]
[illegible]	[illegible]
[illegible]	[illegible]
[illegible]	[illegible]
[illegible]	[illegible]
[illegible]	[illegible]

[illegible]

[illegible]

[illegible]

[illegible]

[illegible]	[illegible]
[illegible]	[illegible]
[illegible]	[illegible]
[illegible]	[illegible]
[illegible]	[illegible]

[illegible]

[illegible]

[illegible] for the production of [illegible] developed [illegible] Federal [illegible] for the [illegible] plants.

25.

1. 72