ATTACHMENT XV

Manufacture of an Ethylene-Containing Gas by Thermal Cracking of Ethane or Propane and Processing of the Cracked Gas to Pure Ethylene in a Linde Gas Separation - Plant.

(I. G. Leuna Report)

(Spot Translation)

The ethylene used for the manufacture of SS oils must be at least 95% pure and shall not contain O_2, CO and CO_2 in quantities larger than 0.01%. The water content shall not be higher than 0.3 g per cubic meter.

The gas processing equipment consists of Cracking Plant No. 388 and Linde Plant No. 387, and is designed to produce ethylene of the above degree of purity from ethane, and eventually propane (resulting from hydrogenation processes).

An ethylene-containing cracked gas is produced in the so-called "Small Furnace" (Klein-Ofen) from ethane (or propane) to which oxygen and some nitrogen is added. The gases are heated to 600 and 400°C, respectively. A crude cracked gas is produced containing on the average: 30% ethylene, 19% ethane, 8.5-10% CO, 0.4% CO_2, 1-1.5% O_2, 1.5% higher olefins, and about 0.5% acetylene, the remainder being methane, hydrogen and nitrogen.

The cooled gas is then passed through a purification plant consisting of two alkali wash towers, acetylene hydrogenating units, 2 oil-, 4 carbon-, 2 "alkazit"- and 4 additional alkali wash towers.

Linde Plant

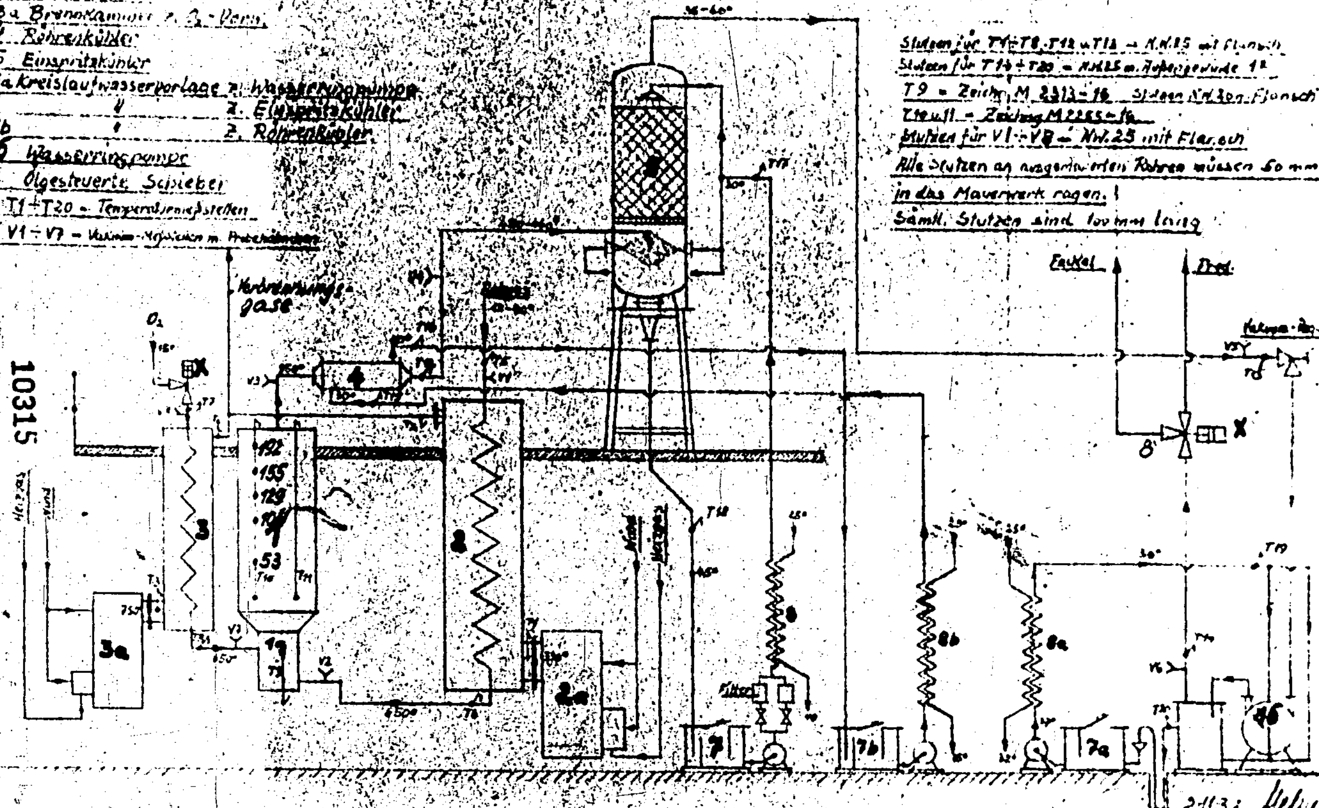
The purpose of this plant is to produce pure ethylene by fractional distillation of the cracked gas.

The plant consists of 2 units, compressors and counterflow condensers, and ammonia refrigerators. The entire apparatus is inclosed in a large heat-insulated housing of sheet iron in which 3 columns, counterflow heat exchangers and ammonia

Schema zur Spaltanlage Me 388
ohne Wärmeregeneration

1 Ofen m. Mischkopf
2 Rohgasvorwärmer
2a Brennkammer z. Rohgasvorw.
3 O_2-Vorwärmer
3a Brennkammer z. O_2-Vorw.
4 Röhrenkühler
5 Einspritzkühler
7a Kreislaufwasservorlage z. Wasserringpumpe
7 " z. Einspritzkühler
7b " z. Röhrenkühler
46 Wasserringpumpe
X Ölgesteuerte Schieber
T1 ÷ T20 = Temperaturmeßstellen
Y V1 ÷ V7 = Vakuum-Meßstellen m. Probenahme

Stutzen für T1 ÷ T8, T12 u. T13 = N.W. 25 mit Flansch
Stutzen für T14 ÷ T20 = N.W. 25 m. Rohrgewinde 1"
T9 = Zeichn. M 2313-16 Stutzen N.W. 30 m. Flansch
T10 u. 11 = Zeichng. M 2265-16
Stutzen für V1 ÷ V8 = N.W. 25 mit Flansch
Alle Stutzen an ausgemauerten Rohren müssen 50 mm in das Mauerwerk ragen.
Sämtl. Stutzen sind 100 mm lang



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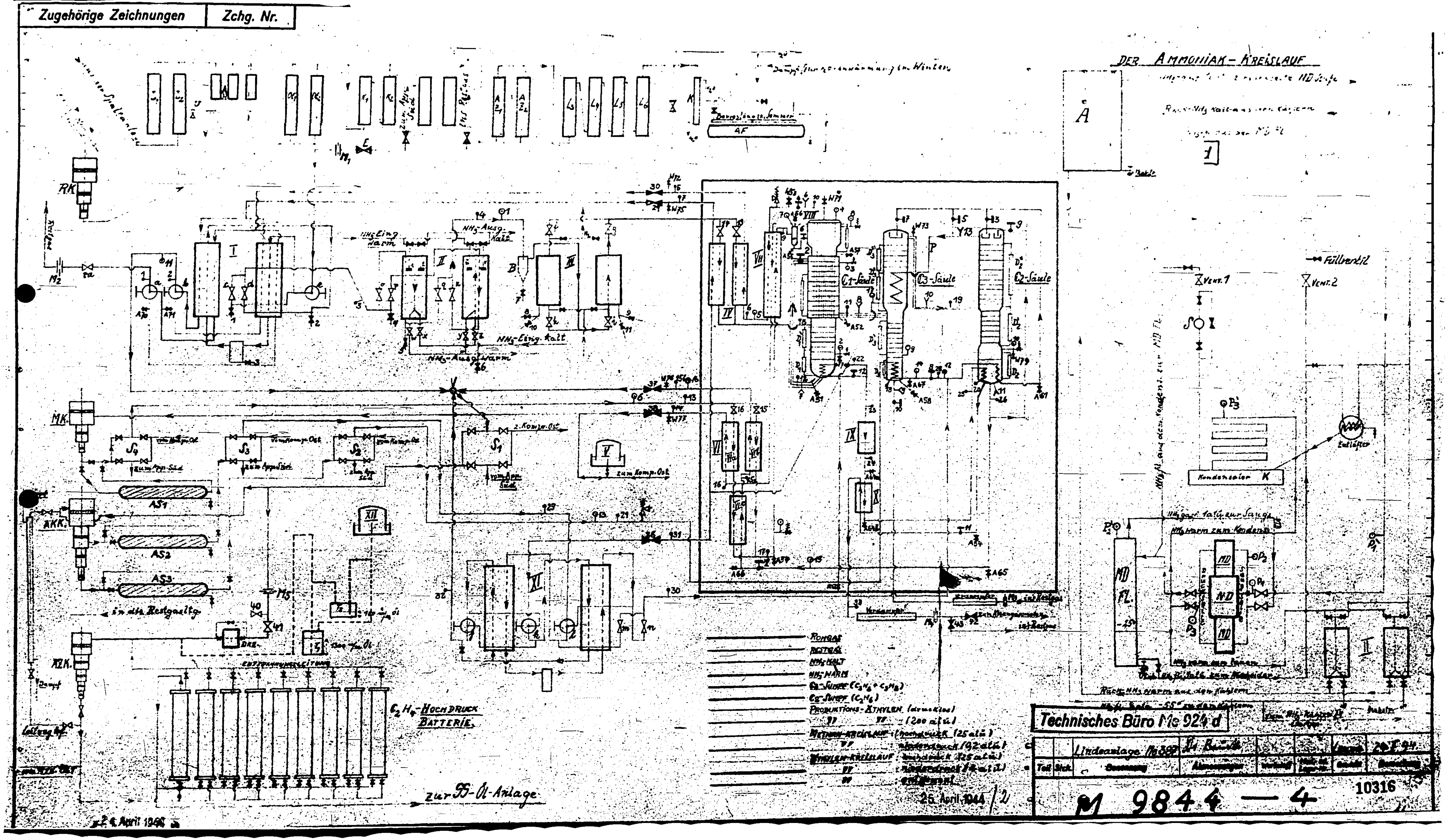

Zugehörige Zeichnungen
Zchg. Nr.
DER AMMONIAK-KREISLAUF
RK
MK
AKK
AS1
AS2
AS3
in die Restgasltg.
C1-Säule
C3-Säule
C2-Säule
NH3-Eing. kalt
NH3-Ausg. warm
NH3-Eing. warm
NH3-Ausg. kalt
zum Komp. Ost
2. Komp. Ost
Füllventil
Vent. 1
Vent. 2
Kondensator K
Entlüfter
ND
MD
FL.
C2H4-Hochdruck Batterie
zur SS-Öl-Anlage
Rohgas
Restgas
NH3-kalt
NH3-warm
C2-Sumpf (C_2H_6 + C_3H_8)
C2-Sumpf (C_2H_6)
Produktions-Äthylen (drucklos)
" " (200 atü)
Methan-Kreislauf: hochdruck (25 atü)
" : niederdruck (92 atü)
Äthylen-Kreislauf: hochdruck (25 atü)
" : niederdruck
" : entspannt
25. April 1944
Technisches Büro Me 924 d
Lindeanlage Me 389
Teil
Stck.
Benennung
M 9844 — 4
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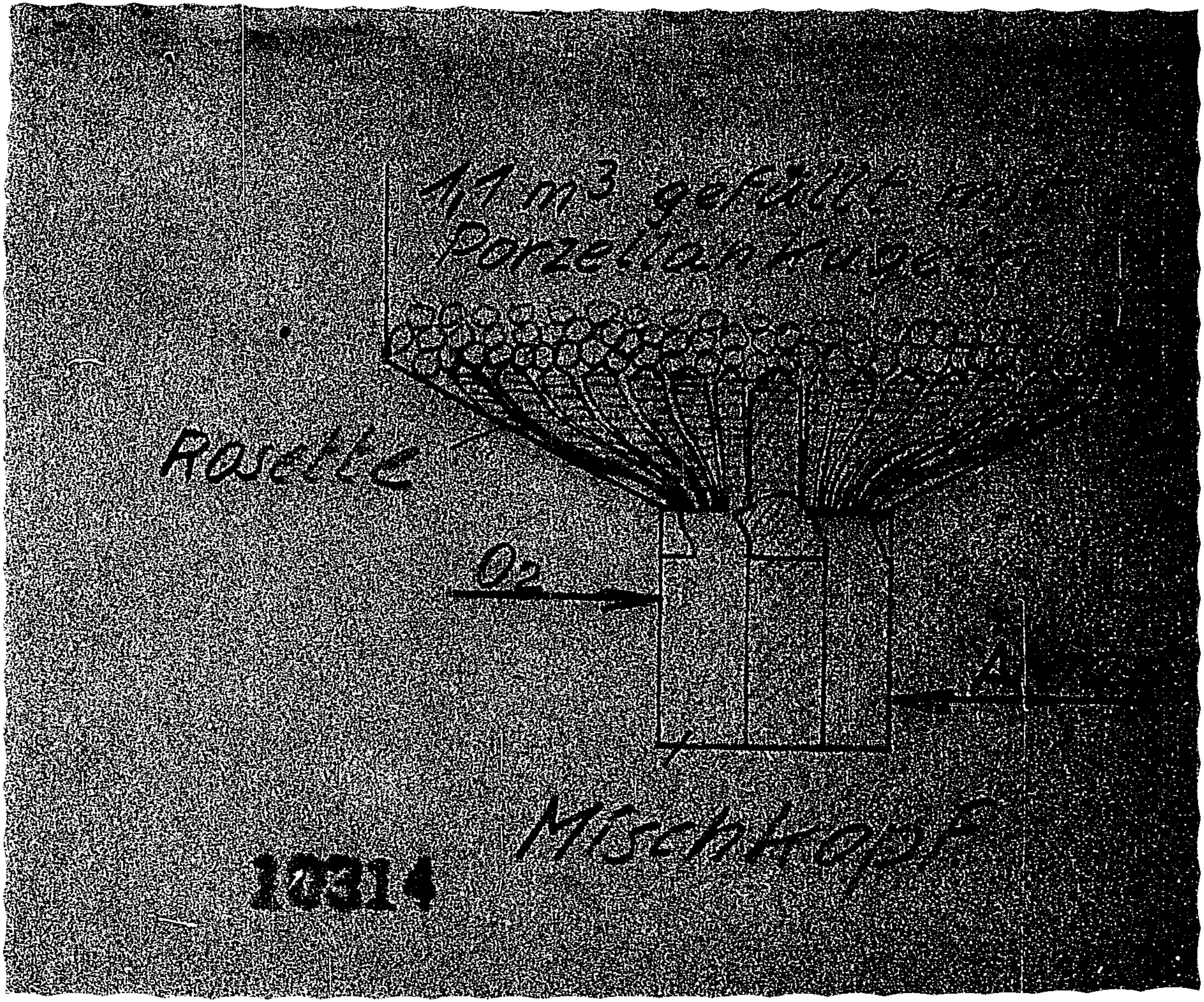
1,1 m3 gefüllt
Rosette
O2
A
Mischkopf
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refrigerators are located.

The gas is compressed to 16.5 atm. gauge pressure and passes through the various washing units in which undesirable contaminants, such as aromatics, more unsaturated higher olefins, acids, carbon dioxide, etc. are removed.

After this preliminary purification the gas — now called crude gas — is passed through counterflow heat exchangers and ammonia refrigerators, is cooled to a temperature of -55°C, and enters the first column C_1, the methane column. In this column methane and inert gases are removed under a pressure of 13.5 atm. C_2 and C_3 remain as bottoms.

In the C_2 column the ethylene is separated from ethane and is recovered as overhead and compressed to 200 atm. in a battery of steel cylinders which are shipped to the SS Oil manufacturing plants. The low temperature required for distillation is produced partly by expanding the C_1 bottoms through the crude gas condenser into column C_3 but principally by the methane, ethylene and ammonia auxiliary circulating units.

<u>The methane circulation unit</u> in which methane is cooled to -100°C under 25 atm. gauge pressure and released into the methane condenser is located on top of column C_1 where the pressure is released to 0.2 atm. gauge pressure.

<u>The ethylene circulation unit</u> operates in four stages at pressures ranging from 4 to 25 atm. In the C_1 and C_3 bottoms high pressure ethylene is used for heating; in the C_2 bottoms high- and low-pressure ethylene is used as heating medium, at -64 to -74°. It is then introduced as reflux in the C_2 and C_3 columns after which it is used as a cooling medium in counterflow heat exchangers.

The ammonia refrigerators (II, IX) are continuously fed with liquid ammonia. The warm (gasified) ammonia is recompressed and recondensed.

The Cracking Plant

consists of four identical units made up of: the so-called "small furnace" (1) preheaters (2 and 3) with firing boxes (2a and 3a) for the feed gas and for the oxygen, one tubular and one jet condenser (4 and 5), a water vacuum pump (6) for passing the gas mixture through the furnace at the reduced pressure required, and the circulating cooling units (7a, 7b, and 7c) for the two condensers and the pump. Auxiliary blowers for the feed gas and blowers for heating gas and air are additional accessories.

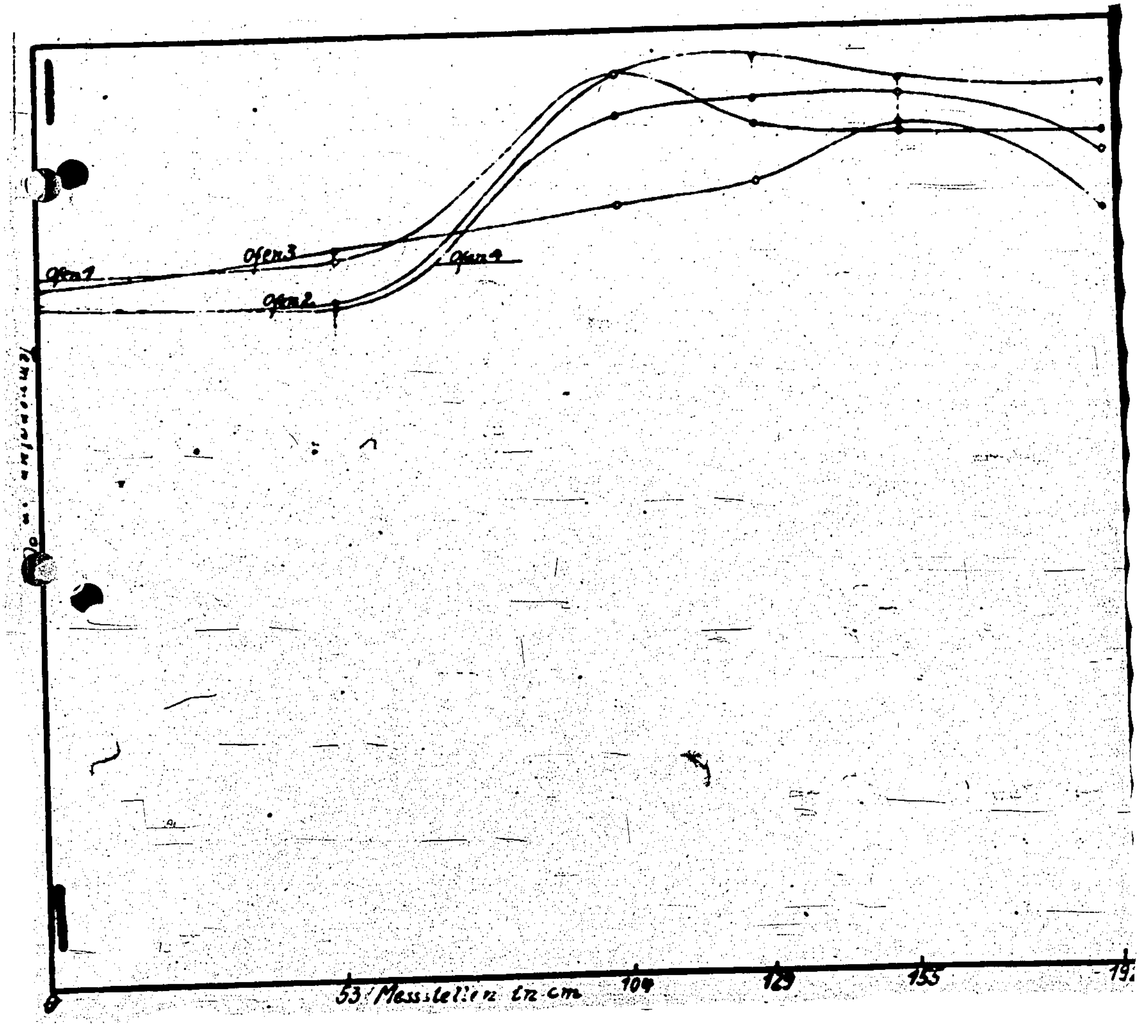
The Small Furnace (1) is an iron shell lined with fire brick, 1.75 m high and 0.9 m in diameter. Ethane and oxygen + nitrogen preheated to 600° C and 400° respectively are introduced into a mixing head. The gas preheater is an iron shell lined with fire brick into which another iron shell (5 m long, 1 m diamter) filled with kieselgur is fitted. The ethane is heated to 600° (point 6) and the oxygen to 400°C (point 8). The gas mixture is then introduced into the 1.1 m^3 capacity furnace which is filled with porcelain balls, resulting in partial combustion of the ethane with simultaneous cracking of the remainder.

The hourly throughput is 780 m^3 ethane and 220 m^3 oxygen to which 20 m^3 nitrogen is added.

Nitrogen is added to minimize explosion hazards; the furnace is operated under a reduced pressure of 412-413 mm Hg (measured at the point V_2) which is produced by a water vacuum pump and adjusted through a vacuum regulating valve.

Correct reading of the reduced pressure, with reference to the barometric pressure, is imperative since the actual reaction zone shifts with the pressure in the furnace and satisfactory operation depends thereon. Measurements are taken at 6 temperature points and, as will be seen from the diagram, range from 820 to 860° in the upper half of Furnaces 1, 2 and 4. When a furnace operates well (good mixing, no carbon deposit on the porcelain balls) as in Furnace 3, the temperature is about 800°C in the upper third.

Furnace		1	2	3	4
Measuring point	0	670°	640°	660°	645°
" "	53	680°	640°	690°	635°
" "	104	850°	850°	740°	810°
" "	129	860°	865°	745°	820°
" "	155	790°	840°	795°	825°
" "	192	785°	830°	710°	765°



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0 designates the lowest point (furnace bottom). The other points are distances in cm from the bottom .

1,340 m^3 cracked gas, produced from about 1000 m^3 feed gas, passes to the tubular condenser (4) and the jet condenser (5) in which the gas is first cooled to 400°C (point 12) and finally to 50° (point 13).

The cooled gas then passes to the suction side of the water vacuum pump from which it goes to the Linde plant at about 180 mm Hg. abs. pressure.

Operating Trouble

Trouble may be experienced when

1. the vacuum is unsatisfactory, or
2. ignition occurs at the bottom of the furnace.

The furnace is shut off by discontinuing the supply of oxygen and replacing it with nitrogen.

For a density of 0.82-0.84 the cracked gas has the composition given on page 1.

Cooling Units. Three units are provided (7a, 7b, and 7c), 7a for the jet condenser, 7b for the tubular condenser and 7c for the water vacuum. Since water in 7a and 7c comes in direct contact with the gas it is rendered slightly alkaline with 10% NaOH to neutralize acidic constitutents in the cracked gas.

Starting of the Furnace

1. The cooling units are started,
2. 350 m^3 of nitrogen is passed through the ethane preheater and 100 m^3 nitrogen through the oxygen preheater.
3. The heating gas is ignited in the fire boxes
4. The water vacuum pump is started and the pressure adjusted to 400-500 mm Hg by means of the regulating valve.
5. Both preheaters are raised 20° per hour until point 6 of the ethane preheater shows 600° and point 8 of the oxygen preheater 400°. The point 0 then shows about 580°C.
6. Ethane and oxygen are introduced (quantities according to load, say 680 m^3

ethane + 220 m^3 oxygen). Nitrogen is then introduced, about 10% of the oxygen used (20 m^3).

The Linde Plant

The Linde Plant consists of two identical units which may be operated independently or disconnected.

Each unit is composed of:

1. Four gasometers and 9 200 atm. high-pressure vessels for storing the ethylene.
2. Six compressors.
3. Vessel for separating NaOH.
4. Crude gas counterflow-heat exchanger.
5. Ammonia condenser.
6. Silica gel tower.
7. Ethylene counterflow heat exchanger.
8. Gas-liquid separator.
9. Apparatus (Apparat)
10. Manifold
11. Oil separating vessels in the methane and ethylene pressure stages.
12. Bottoms pressure release and quick release.

1. Gasometers

Gasometers are provided (a) for crude gas, (b) for ethane, (c) for methane, (d) for ethylene.

2. Compressors

a. The crude gas compressor (Sürth) is a 3-stage compressor of 2,100 m^3/h capacity, 550 stroke.

1st Stage	1.6 atm. gauge pressure
2nd "	6 " " "
3rd "	16 " " "

All other compressors are 2- to 5-stage compressors operating at 1.5 to 200 atm. gauge pressure. Directions are given of how to start and operate them.

Ice Machine

Directions are also given for starting and operating the ice machine.

6. The Silica Gel Tower (III) consists of two units. Its purpose is to remove the last traces of moisture from the crude gas coming from the ammonia refrigerator. The silica gel is regenerated with nitrogen. (This unit was later discontinued as non-essential).

7. The ethylene heat exchanger (XI) is a two stage unit. Exchange between pressureless ethylene from the apparatus and recycled ethylene brings the temperature of the latter to -64°C (temperature point 30)

8. Gas-Liquid Separator

This unit is provided with a safety valve and a rupture disc. Gas-liquid separation is important and must be carefully supervised to prevent condensate from being entrained, which might cause clogging of condensers and lines and cause trouble in the operation.

9. Apparatus

In a Linde plant this designates the portion of the plant in which the actual separation of gases takes place.

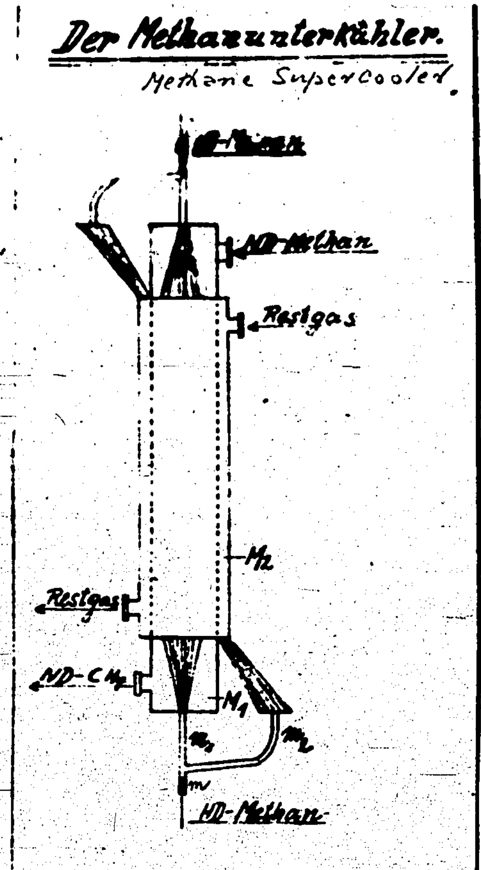
It consists of three columns, C_1, C_2 and C_3 (named for the carbon content of the gases), a crude gas counterflow heat exchanger in two stages (IV), a methane counterflow unit (VI) (called methane liquifier), a simple methane refrigerator (VII), a methane separator (VIII), an ammonia refrigerator (IX) and a methane counterflow unit (X) (called ethane vaporizer).

These units are indicated and described on Chart M 9844-4.

Methane Supercooler (VII)

This unit serves to liquify high pressure methane (from the methane liquifier (VI)) which passes counterflow to the residual gas leaving the C_1 column at -140°C (temperature point 10) and low pressure methane from the methane condenser.

Da an diesem Gegenströmer eine Reparatur vorgenommen werden musste, konnte er beim Ausbau angesehen werden und ist im Folgenden skizziert:



Der Gegenströmer besteht aus einem Kupferblechmantel von ca. 2,1 m Länge und 30 cm im Ø, über dem ein zweiter Kürzerer mit ca. 35 cm Ø liegt. Durch den inneren Mantel strömt Niederdruckmethan aus dem Methankondensator kommend durch den äußeren, das Restgas aus der C 1-Säule kommend.

Das aus dem Methanverflüssiger in der Leitung m ankommende Hochdruck-Methan teilt sich in die Zweige m1 und m2, die sich dann weiterhin beim Eintritt in die Mäntel (M 1) bzw. knapp vorher (m 2) in eine große Zahl von verzinkten Kupferröhrchen aufteilen, in denen das Hochdruckmethan dem Niederdruckmethan bzw. dem Restgas entgegenströmt und so verflüssigt wird.
Es ist anzunehmen, dass die Röhrchen sowohl im Inneren als auch im Äußerne Mantel Spiralen liegen.

Der Grund, warum der Gegenströmer ausgebaut werden musste, war ein ca. 4 cm langes Stück geplatzter Lötnaht des unteren Deckels vom inneren Mantel. Beim Ausbau konnte bemerkt werden, dass beide Mäntel auffallend schwach und keinerlei grösseren Druckbeanspruchungen gewachsen sind.

Der Methanabscheider (VIII) stellt eine gewöhnliche Hochdruckflasche von ca. 60 cm Länge vor. In der Mitte mündet die Zuführungsleitung des Flüssiggasmethans das unten abgeht und über Ventil 6 in den Methankondensator entspannt wird. Oben sitzt eine Sicherungsventilleitung, die Manometerleitung 7 und eine Ausblasleitung

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The unit consists of a copper jacket about 2.1 m long and 30 cm in diameter, surrounded by a second cylinder about 35 cm in diameter. Low pressure methane flows through the inner jacket from the methane condenser; residual gas from the C_1 column flows through the outer jacket (see diagram).

10. Manifold

Switching members (S_1, S_2, S_3 and S_4) serve to exchange the ethylene and methane circulation compressors. This largely facilitates shutting off compressors for repair. The operation is easily understood from the drawing.

11. Oil Separating Vessels in the Methane and Ethylene Primary Pressure Stages.

These units were inserted for separating entrained oil from the compressors because the standard equipment was found inadequate.

12. Pressure Release for Bottoms and Quick Pressure Release.

On shutting down the plant all bottoms and liquified gases in the lines are discharged into a copper tank of about 400 liter capacity; during air raids a copper tank (about 3 m long and 1.5 m in diameter) large enough to hold all products is used.

Flow of Crude Gas and Ethylene

The crude gas from the cracking plant passes through the parallel caustic soda wash towers (L_1, L_2) to the suction side of the crude gas compressor (RK). These towers are 13 m high and have a diameter of 1.1m. They operate at 0.5 atm. gauge pressure and are fed with about 3 m^3 per hour of 10% NaOH which is withdrawn after saturation with CO_2.

The gas then passes to the acetylene hydrogenation step (AH) where it is heated to 160° by high pressure steam, and then in parallel through three contact furnaces, leaving these at 220°, whereupon it is cooled in spray condensers to 30° C. This step may be omitted, but as the oxygen content is simultaneously reduced, the operation is being continued, particularly since it seems to have a certain "filtering action". From the crude gas compressor the gas goes to oil towers (OE_1, OE_2) under a pressure of 60.5 atm. These towers are 13 m high, 0.8 m diameter, and operate under 20 atm. gauge pressure. Two charcoal towers (K_1, K_2) connected in series follow the oil towers. The gas then passes into the "alkazit" wash towers which have the same dimensions as the oil towers. The "alkazit" towers are fed with 6 m^3 per hour "alkazit" (M-liquor) which is recirculated continuously after regeneration. Alkazit is potassium-N-methyl- α -amino propionate:

$$2CH_3 - \underset{\displaystyle NH.CH_3}{\overset{|}{CH}} - COOK + H_2O + CO_2 \rightleftharpoons \underset{\displaystyle NH\ CH_3}{CH_3.CH.COOH} + K_2CO_3$$

The reaction is reversible; CO_2 is driven off by boiling the solution which is thus regenerated.

Alkali washing towers L_3, 4 and 5 and 6 follow the alkazit wash to remove the last traces of CO_2.

Complete removal of CO_2 is imperative since a content in excess of 0.01% renders the gas unfit for use. The gas then passes through a separator for removing entrained alkazit solution and is brought by the crude gas heat exchanger I to a temperature of -25°C (temperature point 3); it then flows to the ammonia refrigerator II which it leaves at -45°C (point 4) and passes through the separator B and the silica gel tower III into the so-called "apparatus". Here methane and inert gases leave as overhead (1300 m^3/h for 2,400 m^3/h feed, representing 54%) at a temperature of -140°C (point 10). Liquefied crude gas constituents return to the bottoms which are heated by high pressure ethylene (T=30°, temp. point 21) from the ethylene cycle. The bottom temperature is one of the most important temperatures and must be maintained carefully at -32 to -33°C. A drop of two degrees prevents the bottoms from being entirely free of CO so that the ethylene which is recovered as overhead in the next two columns may contain an excessive quantity of CO, rendering it unfit for SS Oil production (limit 0.01% CO).

The bottoms (t = -32°), freed from C_1 and inert gases, is now passed through the crude gas condenser. The pressure is reduced though Valve 2 from 13.5 to 0.5 atm. gauge pressure and the gas enters the column C_3 at -82° (temperature point 11).

In this column C_2 and C_3 are separated, ethylene and methane passing over head into the C_2 column. All C_3 and higher gases remain as bottoms and pass to the residual gas through Valve 10. The temperature (point 18), which is maintained at -50 to -60°, depends on the composition of the bottoms and must therefore be controlled. Reflux in the C_3 column is pure ethylene from the low pressure stage (4 atm.), reduced to 0.2 atm. gauge pressure by Valve 7 after having been used to heat the bottoms in the C_2 column.

The overhead product of the C_3 column now passes at a temperature of -93° (temp. point 19) into the C_2 column. Here pure ethylene is finally distilled off while ethane and in certain cases entrained higher hydrocarbons remain as bottoms and are withdrawn through Valve 11 as residual ethane. The bottom temperature is about -82° (temp. point 20) and depends on the composition of the bottoms and the purity of the distilling ethylene.

The bottoms are heated in two different ways, by high pressure and low pressure ethylene. In practice the heating is regulated in the high pressure system alone by controlling the ammonia in the ammonia refrigerator 1, since the amount of bottoms supplied to the ethane vaporizer X is constant.

Residual Gas

Residual gas (about 54% of the crude gas) consists chiefly of methane, hydrogen and carbon monoxide together with small amounts of oxygen and nitrogen. It escapes as overhead from the C_1 column at -140° (temp. point 10) through Valve 4 into the methane supercooler VII and refrigerates the high pressure methane, thus attaining a temperature of -100° (temp. point 6) and then passes through the crude gas counterflow exchanger IV, which it leaves at -50°, and enters the crude gas counterflow exchanger I, passing therefrom at + 16° into the residual gas line.

Residual Ethane

Residual ethane is the bottom product of the C_2 column and must contain not more than about 1% ethylene. It represents about 18-20% of the quantity of crude gas being processed. From the bottoms it passes in counterflow to the high pressure leg of the ethylene cycle through the ethane vaporizer X, leaves the "apparatus" and passes through a steam-heated vaporizer (so-called Leuna evaporator) which it normally leaves at + 60° and flows to the ethane storage tank.

The Methane Cycle

The methane cycle serves the purpose of condensing the last portions of ethylene and ethane from the overhead residual gas of the C_1 column. The gas comes from the third stage of the compressor through the oil separator to the manifold S_4 and passes at a temperature of + 15 (temp. point 13) under a pressure of 25 atm. into the "apparatus".

The Ethylene Cycle

This cycle operates in four stages, the second stage at 4 atm. and the fourth stage at 25-37 atm. gauge pressure and serves to heat the bottoms in the three columns, supply reflux to the C_3 and C_2 columns and refrigerate the methane cycle.

The high pressure ethylene comes from the fourth stage at 25 atm., passes through the oil separator and manifold S_2 into the "apparatus" at a temp. of + 30° (temp. point 21). A small branch stream passes into the C_3 column to heat the bottoms. This branch stream is regulated by Valve 8. The bottoms (temp. point 18) are kept at -50 to -60° C. After release of pressure all the recycled ethylene goes into the C_2 column except that portion of the processed ethylene passing through Valve I.

Ammonia Cycle

This cycle serves to cool the crude gas in the ammonia refrigerator II from -24° (temp. point 3) to -44 to 50° (temp. point 4) and the high pressure ethylene in the refrigerator IX from + 20 to -24° (temp. points 23, 24).

Shutdown and Cleaning of the "Apparatus".

Special directions are given for safe handling of the "apparatus".

Starting the "Apparatus"

After shutdowns and cleaning the apparatus is usually dry in 60-72 hours.

The temperature should be about 25-40° and the nitrogen used for flushing must come out dry. Directions are given for operating the valves in the various lines, including the crude gas line, the methane cycle and the ethylene cycle. These steps are illustrated in tabular form (text pages 34 to 36).

Safety Valves and Manometers

Safety valves are located at the principal points. Pressures indicated by gauges in the various units are tabulated and are given in atmospheres at the various locations.

Analyses

1. In the cracking plant a 100 cc sample of the processed gas is taken every 4 hours. The composition should correspond approximately to the one given on page 1.

2. Linde Plant

Acetylene hydrogenation ;

a. The gas from the cracking plant is passed over a catalyst(not specified), heated to 160°, for the purpose of hydrogenating the acetylene. The average acetylene content is 0.4-0.2% before hydrogenation and not more than 0.2% after passing over the catalyst.

b. Separation of Gases.

1. Analysis point 1: Higher olefins and ethylene are determined every2 hours in the crude gas before reaching the counterflow exchanger I. In normal operation the gas should contain 30-31% ethylene and 1% higher olefins.

2. Analysis point 6: Oxygen and total olefins are determined every 2 hours in the residual gas coming from the C_1 column (before Valve 4). In normal operation the gas should contain 0.4-1% O_2, olefins 0.0%.

3. Analysis point 7: The content of higher olefins and ethylene is determined every 4 hours in the C_1 bottoms. Contents: 1.2% higher olefins, 59% C_2H_4.

4. Analysis point 9: Higher olefins and ethylene are determined every 4 hours in the C_3 bottoms (V9) Contents: 36% higher olefins, 2.6% C_2H_4.

5. Analysis point 11: The ethylene content is determined hourly in the

C_2 bottoms (Y 11). Normal content 0.4-1%

6. Analysis point 13: the overhead product (pure ethylene from the C_2 column (Y 13)) is tested every half hour for purity by determining the ethylene content: C_2H_4 = 99-99.6%.

The CO content in the pure ethylene is determined once every day and the molecular weight of all gases hourly in the molecular weight balance.

This balance operates by determining the difference in weight of two gas columns of equal height under identical pressure and temperature. (See sketch). In normal operation the following values were found.

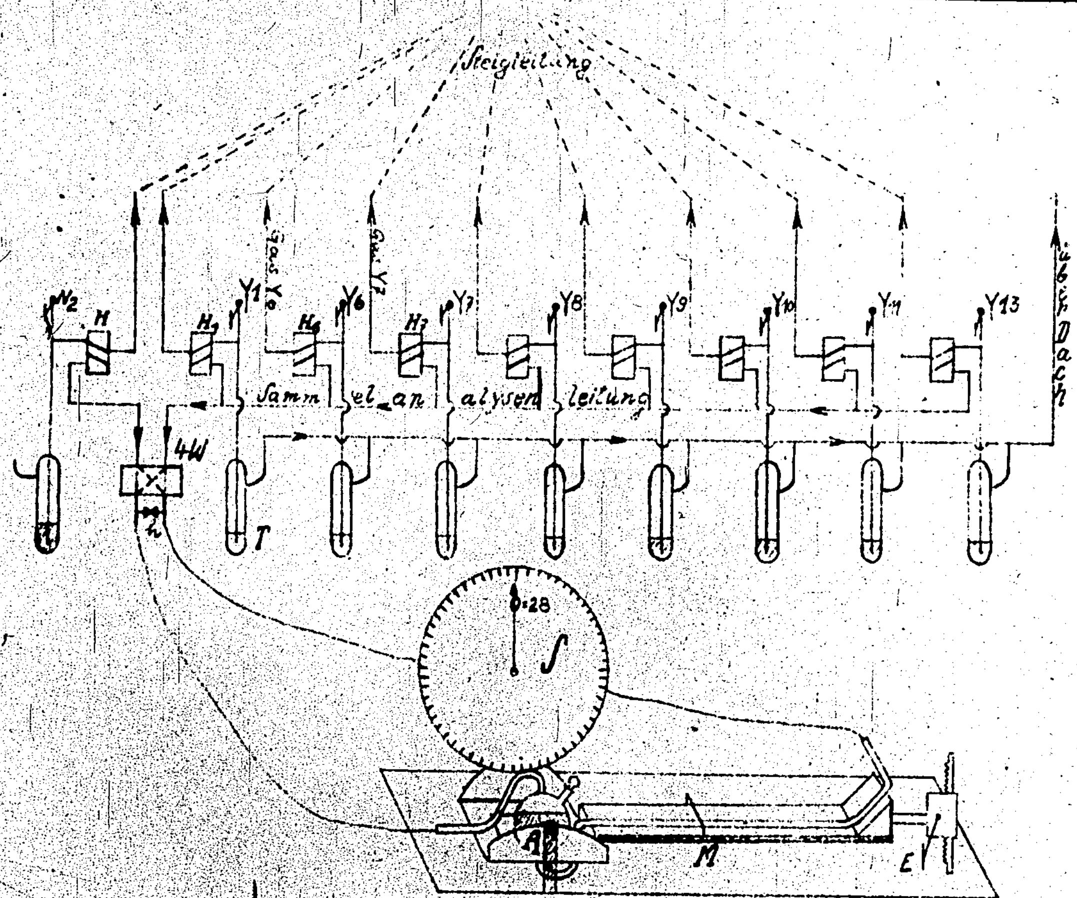
Analysis point	1	20.4	Analysis point	9	Above	42
" "	6	10.8 - 11	" "	10		29
" "	7	29.6 - 30	" "	11		30.2
" "	8	29.6 - 30	" "	13		28.2

Procedures:

CO_2 is determined with 30% KOH, 1 min. shaking. Oxygen is determined with chromous chloride prepared by electrolysis of a solution of 200 g chromic acid in one lit. conc. HCl. CO is analyzed with acidic cuprous chloride or with hemoglobin. Higher olefins are determined in 80% H_2SO_4.

C_2H_2 is determined by the mercury cyanide method or by the Ilosvay colorimetric method, using a solution of $CuSO_4$, ammonia and hydroxylamine. (The method is described in detail).

Additional tests include the determination of COS and mercaptan (CH_3SH). COS is determined by passing 40 lit. of gas through wash bottles containing an aqueous solution of isobutylamine and Cd-acetate, and subsequent titration with N/10 I-solution (for COS) and absorption in an aqueous solution of $AgNO_3$ and H_2SO_4 (for CH_3SH).



DIE MOLEKULARGEWICHTSWAAGE.

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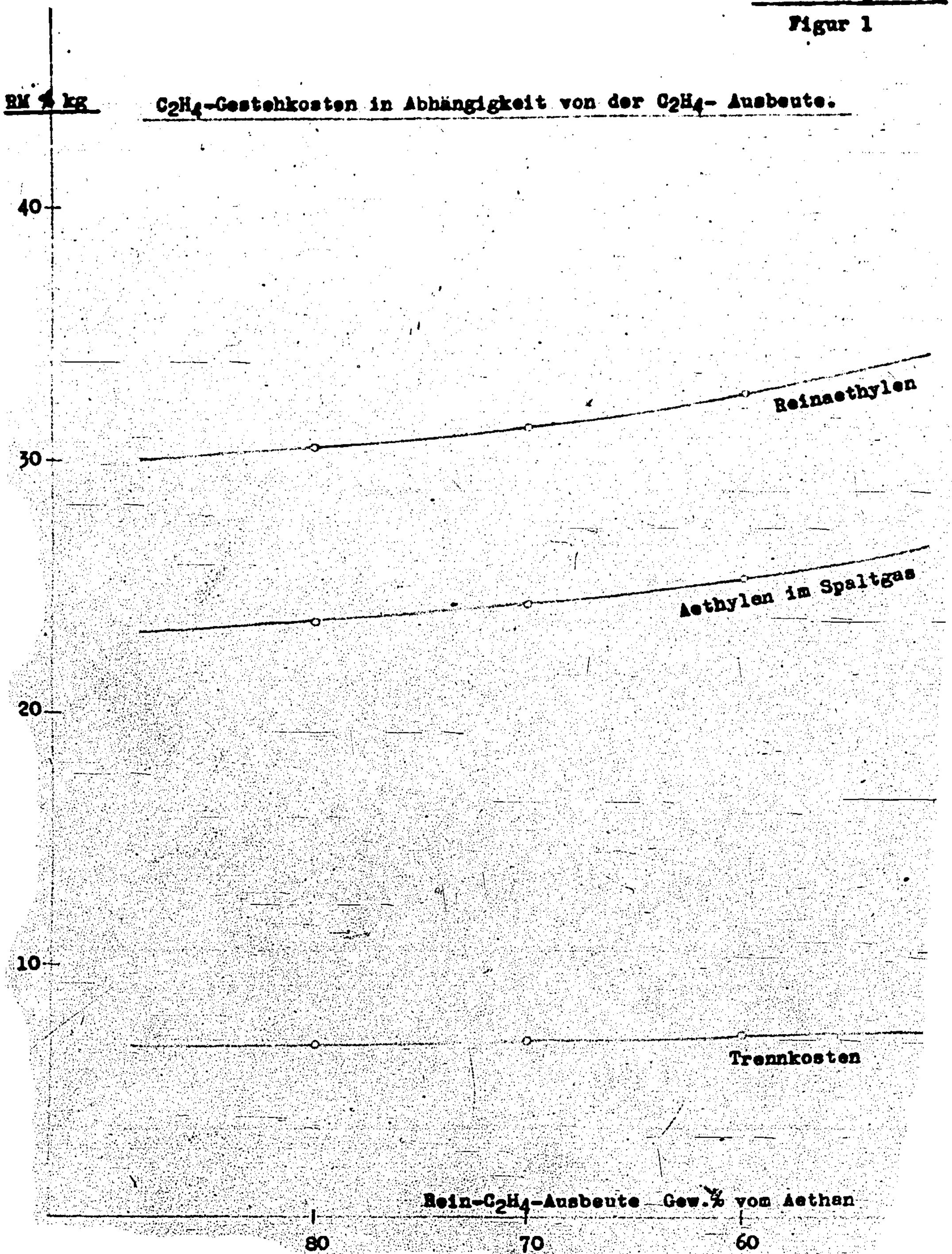
Anlage 2
Figur 1

C_2H_4-Gestehkosten in Abhängigkeit von der C_2H_4- Ausbeute.



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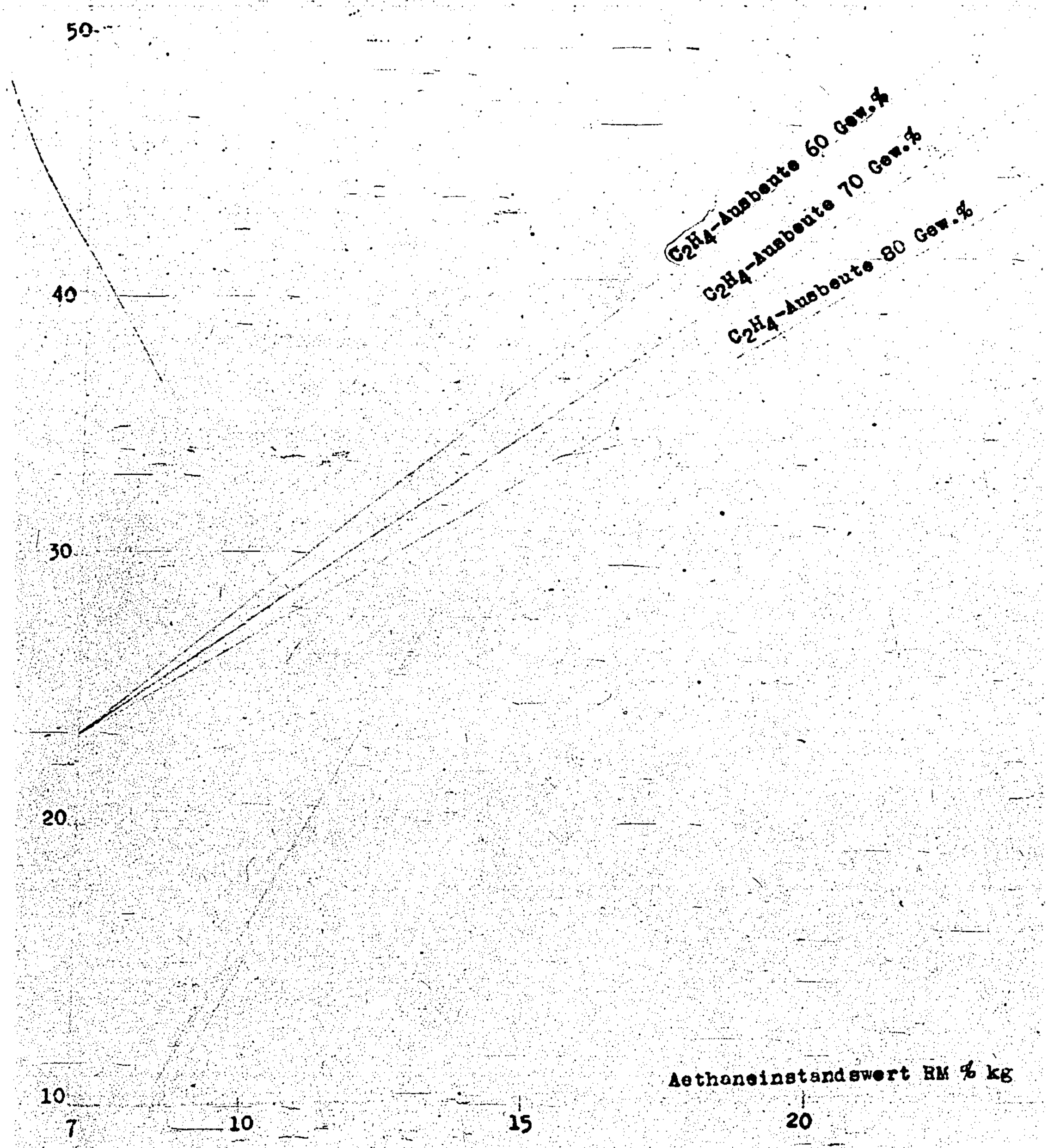
Anlage 3

Figur 2

C_2H_4-Gestehkosten in Abhängigkeit vom Aethan-Einstandswert bei verschiedenen C_2H_4-Ausbeuten.



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