TABLE 3 (cont'd.)

E.P. °C/%	312/99
Phenol	less 1%
Sulfur	less 0.1%
Cetane NO	45-50

The T.T.H. process using brown coal tar was the only combination of raw material and hydrogenation processes that was used to produce lube oils and waxes, as direct hydrogenation destroyed the paraffin constituents in the other coal substances processed. The Zeitz plant produced two grades of lube oils, a light 4° Engler (180 SSU) spindle oil and a heavy 6-8° Engler (215 SSU) machine oil. The yields were approximately two parts of the former to one of the latter. The waxes were also of two grades, a hard wax and a soft wax. The former, which had a melting point of 40-45°C, constituted one-third of the production, while the other two-thirds had a melting point of 52°C.

Fuel oil was produced in limited amount almost exclusively by the hydrogenation of pitch. As has been previously mentioned, the high asphalt content of the heavy residue oil from the sump phase distillation would have been difficult to hydrogenerate, if it had been recycled. Therefore, this product was sold for fuel oil. The yield of fuel oil was approximately twice that of gasoline. The Welheim plant (Ruhroel AG) was the only large unit to operate on pitch.

11. Operation and control of the Units.

(a) Introduction.

This section of the report deals with mechanical operation of the equipment. It will include a summary of operating sequence for starting up and shutting down the high pressure chambers, safety measures, an outline of control methods, and a brief discussion of instrumentation. Operations which are common to all chemical industry, such as

11. Operation and Control of the Units (a)(cont'd.)

operation of pumps and compressors will not be covered except for conditions peculiar to this particular process.

(b) Sump Phase Starting Procedure.

Before a new sump phase unit is first put into operation, all masonry and internal insulation must be dried. This ordinarily requires about two days at 120°C, during which time nitrogen is pumped through the system. Instrument checking and servicing requires about 3 hours.

The entire sequence of starting up is as follows:

(1) Purge system with nitrogen, after purging is complete, build N_2 pressure to 300 atm. and check for leaks. It is important that nitrogen be used, not only to avoid explosive mixtures within the equipment, but also to prevent fires in case there is leakage in the piping or equipment.

(2) Expand N_2 to 50 atm, using the excess for testing other equipment.

(3) Start gas circulators.

(4) Dry out preheater masonry.

(5) Check all meters and instruments.

(6) Blow down the nitrogen and fill the system with circulating gas at 300 atms.

(7) Put meters and instruments into service.

(8) Start heating the system with 12,000 m^3/hr of circulating gas, of which about 1/6 goes through the heat exchangers. The temperature at the inlet to the first converter is slowly increased at a rate of about 15 - 18°C per hour.

(9) When the temperature reaches about 300°C the slurry of oil and catalyst (30 percent of catalyst) is

11. Operation and Control of the Units (b)(cont'd.)

started to the preheater at a rate of 1000 liters/hr, rapidly increasing to 3000 liters. At the same time about 1000 liters per hour of oil are started through the heat exchangers.

(10) Start up the hot oil circulating system at 2000 liters/hour, increasing to 6000 - 7000 liters in about 30 minutes.

(11) When the separators are filled to the normal operating level, the expansion valves are used to hold this level.

(12) As the temperature rises, the use of fresh oil increases, and when it amounts to 6000 - 7000 liters/hour the catalyst slurry is turned through the heat exchangers.

(13) Turn cooling water on slowly.

(14) Control temperature out of the heat exchangers by increasing gas through-put.

(15) The rate of temperature rise is the system is slowed down as the normal operating temperature is approached.

(c) Shutting Down a Sump Phase Unit.

(1) Drop temperature at a rate of 15 - 18° per hour. Cut back on fresh feed, at the same time leaving the gas through-put and circulation unchanged.

(2) Stop catalyst addition at 425°C.

(3) Hold the cold separator temperature constant, unless the product becomes too viscous.

(4) When temperature reaches 250 - 220°C all heat can be removed from the preheater and the flue gas blower shut down. The combustion chamber is purged with nitrogen, and if the shut-down is to be of long

11. Operation and Control of the Units (c)(cont'd.).

duration the fuel lines are blanked off.

(5) All oil circulation and fresh feed is stopped at 165 - 185°C. The preheater is flushed with oil, then blown out with nitrogen. The chambers must remain hot enough to permit complete removal of the sludge.

(6) Gas circulation is contained for 10 minutes, then shut off. The liquid is blown out in sequence from the hot separator, the converters 4 to 1, and the regenerator. The gas is blown down through the expansion valve, taking care that the pressure difference across the entire unit does not exceed 15 atm.

(7) Purge the system with nitrogen for about 50 minutes until no more than 5 percent combustible is found in the outlet.

(8) Blank off all connections to the apparatus to be repaired.

(d) Starting a Gas Phase Unit with Electric Preheater.

(1) Test the electrical system with about 20 volts from the transformer, checking the resistance by comparison of voltage and amperage.

(2) Flush the system with nitrogen to 0.5 percent maximum oxygen.

(3) Build up nitrogen pressure, increasing the pressure from 1 to 20 atm in 1½ hours, and from 20 to 300 atm in 2 more hours. This slow buildup is necessary to protect the catalyst pellets from destruction caused by unbalanced pressures inside and outside the pores, and from damage by physical disturbance from excessive gas velocities. Check all piping for leaks. 30 atm. is sufficient pressure for this purpose if the unit has previously been in operation.

(4) The gas circulator cannot be operated with N_2 over 50 atm because of its greater density compared to

11. Operation and Control of the Units (d)(cont'd.)

H_2. Drop the pressure to 50 atmospheres slowly, then start the circulator.

(5) Blow off the nitrogen to other equipment, and fill with circulating gas. Care must be taken to prevent overheating of the catalyst as heat is released during adsorption of H_2. The pressuring velocity must not exceed that given above for nitrogen.

(6) Start the circulator when the pressure is up to normal, and circulate about 10,000 m^3/hr.

(7) Heat up the inlet to the first converter at a rate of about 10^o per hour.

(8) When the temperature is 220^o, open the cold hydrogen valves to the converters, and check the temperature at each inlet point to be certain that the lines are clear and the cold gas is actually entering the converter. Start the oil feed at a rate of 1 to 2 m^3 per hour per converter, checking its progress through the system with the temperature elements. Put water on the coolers.

(9) Control the temperature to the first converter by adjusting the cold hydrogen to the feed. If necessary, the current can be shut off of the preheater.

(10) Increase the oil feed by about 500 liters per hour to a value of about 7 m^3 per hour.

(11) If the temperature in the converter falls off much when the oil feed is started, cut the feed in half and increase the heat. Otherwise a sudden reaction may overheat the chamber with a high oil throughput.

(12) Hourly samples of the oil at the pump and at the separator should be taken for specific gravity determination. If the gravity increases suddenly the feed must be reduced or cut off until an explanation is found. Too heavy oil or water in the feed will permanently damage the catalyst.

11. Operation and Control of the Units (d)(cont'd.)

(13) As the converter temperature approaches the normal range, gradually increase the hydrogen and oil feeds to the unit to full volume.

(e) Shutting Down a Gas Phase Unit.

(1) Cut back the feed by 2-3m^3/hour, holding the catalyst temperature constant until all oil feed is stopped.

(2) Reduce the inlet temperature to the first converter by about 10-12°C per hour.

(3) Follow the procedure of liquid removal and gas expansion as outlined for the Sump Phase.

(f) Temperature Control in the Converter.

If the temperature rises in some part of a gas phase converter, the normal control is increase of the cold gas injection at that point. If the temperature shows signs of going out of control, and this condition is verified by the gas density recorder, as many of the following steps are taken in sequence as are necessary to bring the temperature under control:

(1) Heat is cut off of the preheater.

(2) Oil feed is shut off.

(3) Gas circulation temperature is decreased, and if this does not suffice, gas feed is cut off.

(g) Operating Safety.

Equipment or piping that has been removed from service is always carefully tested before operation is again undertaken. This testing includes hydrostatic tests of 1½ times the normal working pressure on equipment that has been repaired, leakage tests on heat exchangers, converters and piping systems. The entire system is tested with nitrogen at the working pressure of the unit before hydrogen is

11. Operation and Control of the Units (g)(cont'd.)

turned in. The flushing and purging procedure has been mentioned in the preceding pages.

The large high pressure equipment is installed in concrete cells enclosed on all sides, but with a removable door for installation or removal of equipment. The walls extend to the top of the vessels, and there is no roof. This arrangement has protected the operators and as far as is known no one has ever been hurt in the control room by an accident or explosion in the cell. However, the cells are so close to buildings on the opposite side from the control room, where the door is located, that on one occasion an explosion in the cell killed seven men in the coal paste pump building. A disadvantage of the cells enclosed on four sides is that explosions of gas in the cell are much more violent than if one side were open. On several occasions the converters have been blown or have fallen out through the door of the chamber. The open construction with only one main fire wall, as at Billingham England, appears to be much more desirable, but of course requires more space both for the cells and for the protective area in front of the cells.

An emergency blowdown system was installed in most of the plants. This arrangement was intended as a method of minimizing bomb damage, but is also useful in case of accident. The liquid is drawn off to an underground receiver from which the gas is vented to the air.

The following list of accidents in high pressure plants since 1930(38) shows only the major incidents, and is probably not complete, but it will give an idea of the causes and results of failures.

Plant	Year	Description
Leuna	1930	One chamber (cell) destroyed by failure of an S2 tube which had been installed in a hot location.

11. Operation and Control of the Units (g) (cont'd.)

Plant	Year	Description
Leuna	1935	Expansion machine cylinder failed, killing one man, shutting down the plant completely for two weeks and partially for three months
Scholven	1937	Dust explosion in the coal drying plant.
Nordstern	1940	Preheater fire, due to splitting of an N10 return bend.
Nordstern	1940	Preheater explosion due to plug on a return fitting blowing out.
Nordstern	1940	Fire in a sump phase chamber due to destruction of an emergency blowdown line following failure of a checkvalve and a blow down valve.
Nordstern	1940	Building explosion in the CO_2 absorption plant caused by operating failure of a level indicator on the absorber and breakage of a line to the expansion machine--Due to thawing out the equipment during extremely cold weather--One death.
Nordstern	1940	Leakage in the circulating system caused by foundation sinking.
Pölitz	1940	Gas was accidentally turned into a cell under repair. Explosion followed, killing three.
Pölitz	1941	Circulating gas line plugged with hydrate, causing overheating of the converter. Two converters were blown out of the chamber by the explosion.

11. Operation and Control of the Units (g)(cont'd.)

Plant	Year	Description
Pölitz	1942	Gas inlet line plugged off, overheating converters. The resulting explosion blew two converters out of the cell, and a flame 30 meters long followed. Seven men in the upper part of the coal paste pump house opposite the cell were killed.

It will be noted that most of the accidents occurred when the various plants were new, and could possible be traced to inexperienced operators.

Chart No. 1 on the following page shows the frequency of cell fires in Leuna from 1927 to 1940. An examination of this chart will show that the curve increases with the increasing size of the plant until 1929, when operating experience and development of improved alloys and better design brought a sharp decrease. The increase in 1934 is probably again due to plant expansion. The fires in the last several years amount to only about two a year for all units.

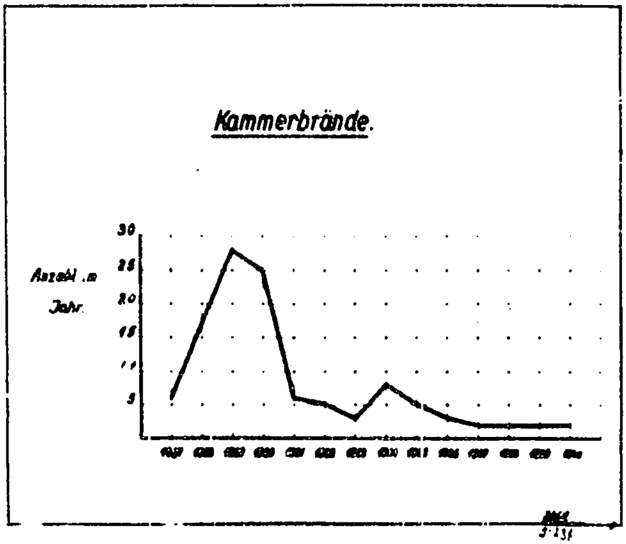
In conclusion it should be emphasized that widely spaced units with cells open on one side would prevent the majority of series accidents. Cell and preheater fires, while spectacular, rarely cause serious damage to equipment or injury to personnel in the Billingham plant of I.C.I. which uses this principle.

(h) Control and Control Instruments.

Free use was made of indicating and recording instruments in the coal hydrogenation plants, particularly of temperature instruments. Automatic control was much less extensive, and only in the last four or five years has it been used to any large extent. The automatic control instruments were all specially built and were usually of somewhat crude design when compared to industrial instruments of the United States. A brief description of the use and operating principle of the various instruments is given below.

RESTRICTED

CHART No. 1



Frequency of Cell fires in Leuna from 1927 to 1940.

11. Operation and Control of the Units (h) (cont'd.)

The "Ringwaage" (Fig. 1) was almost universally used for fluid flow indication or recording.[40] The primary element is an orifice plate or flow nozzle installed in the line in the usual way. The "Ringwaage" merely shows the differential pressure across the orifice, which is proportional to the square of the flow for any given installation. The Leeds and Northrup "Centrimax" and possibly other American makes operate in a similar manner. The instrument was used to record all gas and clean oil flows necessary for material balances or for control, such as fresh hydrogen, circulating gas, cold gas to each point, and the various product gases and liquids.

The coal paste charge quantity was determined by measurement of the stroke velocity of the pump. The instrument (Fig. 2) records an average velocity every three minutes. The "Eckhardt" piston displacement meter was used for some liquid measurements, and utilized a similar mechanism for recording the flow.

Coal feed to the paste mixer and catalyst feed to the slurry were weighed on "Dosierbandwaagen" (Fig. 3) which was a rather conventional automatic scale.

Pressure indication is by gauges with heavy steel spiral bourdon tubes, or by dead weight piston gauges. The piston gauges can be spring loaded or pendulum weight loaded. Accuracy of a fraction of an atm at 700 atm pressure was claimed for some of these instruments.

A differential pressure recorder (Fig. 4) was used to determine pressure drop across various parts of the system during operation. These were sometimes installed with remote oil-controlled valves so that they could be easily switched from one service to another. The photograph shows a piston instrument somewhat similar to the pressure instrument described above. Another type which was sometimes used operated with two steel coil bourdon tubes controlling air pressure through a moveable nozzle. The air pressure was proportional to the pressure difference and was recorded. A gas density instrument (Fig. 5) operating on the Bunsen law principle recorded two gas densities on

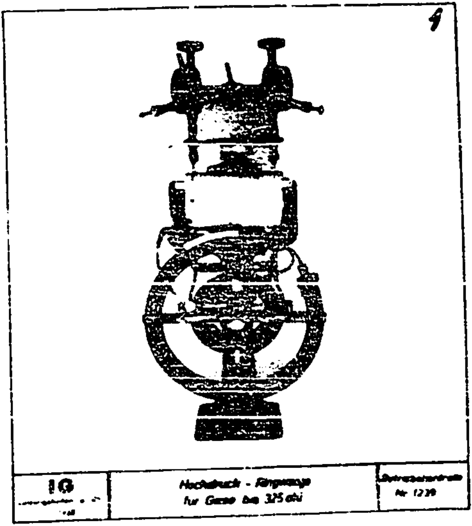


Fig. 1

2

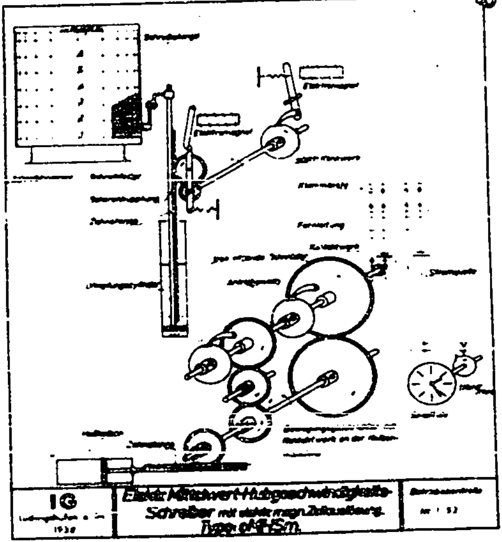
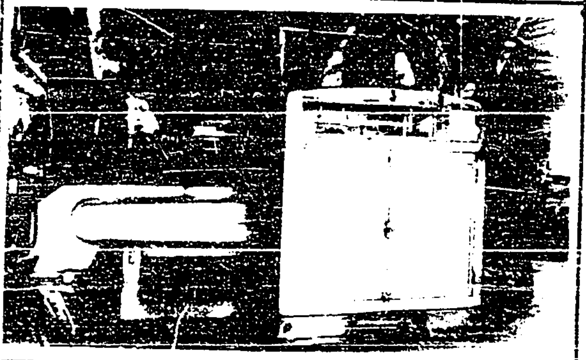


Fig. 2

Kolben-Differenzdruck-Schreiber



IG	Anordnung von IG-Dosierbandwaagen	
[illegible]	[illegible]	Nr. 39

11. <u>Operation and Control of the Units (h)(cont'd.)</u>

the same chart. It was usually applied to the inlet and outlet gas from the reaction chamber, thus giving the operator a picture of the condition of the process. A similar instrument was usually installed on all important metered gas flows where change in density might occur. These data are necessary for accurate measurements.

A combustion apparatus (Fig. 6) could be used for various purposes, such as determination of oxygen in the gas or combustible gas in the air. It contained a catalyst which caused oxidation and the amount of combustion is indicated by the recorded temperature of the outlet gas from the reaction.

A newer type of magnetic oxygen recorder (Fig. 7) has been developed which will indicate 0.01 percent oxygen in the gases. It makes use of the great magnetic susceptibility of oxygen by passing the gas mixture between the poles of a powerful electromagnet, then over a resistance differential thermometer which shows a temperature effect if oxygen is present. The only gas which has a magnetic susceptibility comparable to oxygen is NO, therefore this gas must be absent or the quantity must be accurately known. Further development envisions a permanent magnet instead of the 100 watt D.C. electromagnet.

An H_2S recorder (Fig 8) is operated on the conductivity change of a cell containing a solution of bromine or cadmium chloride. The scale could be made for any concentration of H_2S down to 10^{-5} or 10^{-6} volume percent. The reactions involved are shown on the figure. The second reactant ($CdCl_2$) is usable in the presence of unsaturated hydrocarbons, as the first (Br) is disturbed by the presence of HCN, NO, NH_3 or unsaturates. This instrument was used mostly in the gas separation units.

Ultra red absorption instruments(41) (Fig 9, 10) used to analyze for methane, CO, etc, operated on the small temperature difference between the methane-containing gas and pure methane when they were exposed to ultra red rays from the same source. This instrument is known in the U.S., and according to the I.G. staff the American type is much

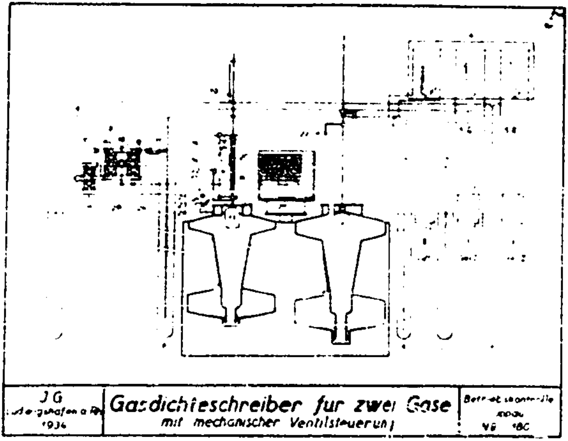

J G
1934
Gasdichteschreiber fur zwei Gase
mit mechanischer Ventilsteuerung

Fig. [illegible]

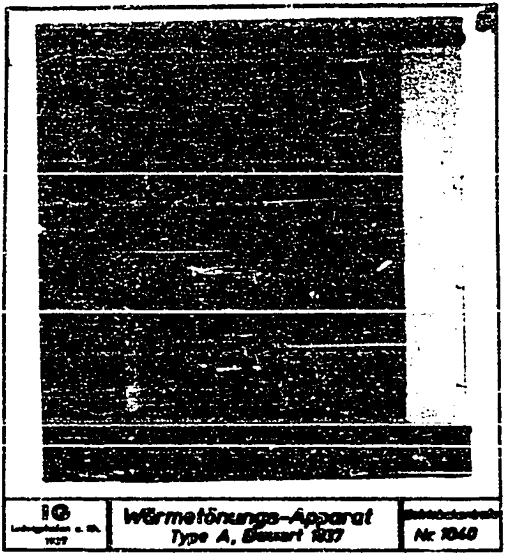
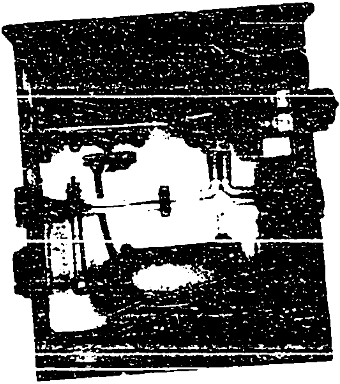


Fig. 6

7



IG .62	Magnetischer Sauerstoffschreiber Schrank I	Nr. 1285

Fig. 7

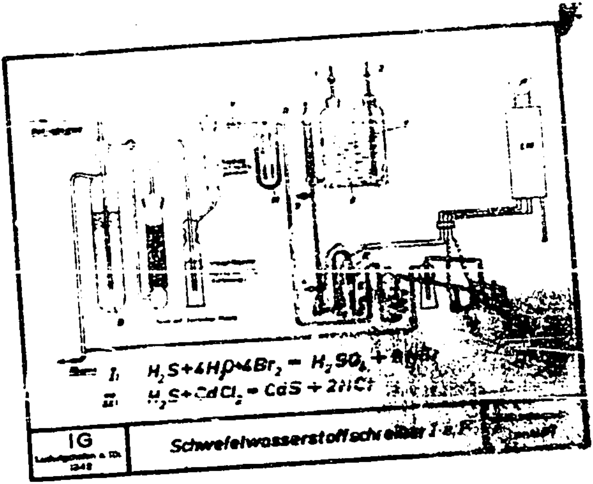
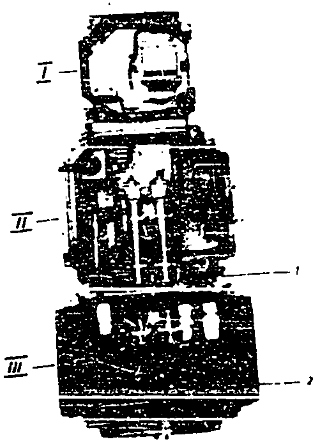

$H_2S+4H_2O+4Br_2 = H_2SO_4 + 8HBr$
$H_2S+CdCl_2 = CdS + 2HCl$
IG
Schwefelwasserstoffschreiber

Fig. 8



IG	Ultrarot – Absorptionsschreiber	Betriebskontrolle Nr 1150

Fig. 9

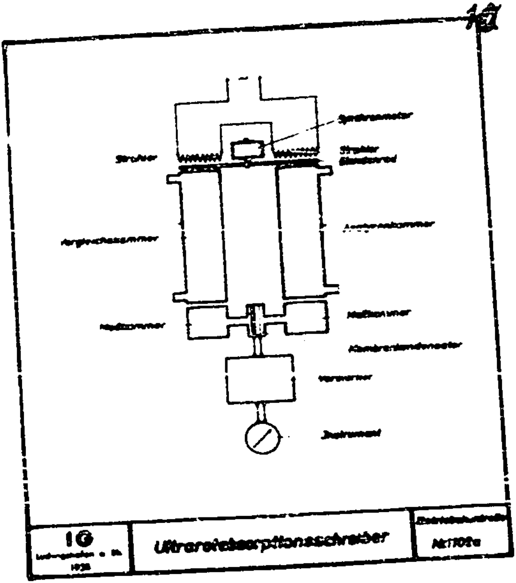


Fig. 10

11. Operation and Control of the Units (h)(cont'd.)

better than their own.

The most important class of instruments was probably that for indicating, recording or controlling temperature, and very free use was made of this type in the hydrogenation plant. The extent of this use can best be demonstrated by the following photostats of drawings 1675 and 1677 which show the pressure and temperature points of the chamber and the preheater, respectively, of Sump Phase Chamber 5 of the I. G. Leuna Plant.(42) In the coal chamber the elements were iron constantin thermocouples, and because the temperatures are often given in millivolts for this couple in the literature and documents, a conversion chart is shown here. The type of each preheater element depends on the temperature at the point of installation. The report entitled "Fortschritte auf dem Gebiete der Messung, Regelung und selbsttätigen Betriebüberwachung von Hochdruckanlagen" in the appendix describes briefly some of the German methods and experiments for increasing accuracy and speed of thermometric instruments. They claim an accuracy of ± 1 or 2°C in their measurements at 400° to 500°C, using photoelectric cell compensators.

The Pölitz and Gelsenberg plants use a temperature alarm instrument that covers 60 points in less than two minutes, but the older plants rely primarily on indicating instruments which are switched from one point to the other, with only a few points of major importance recorded automatically.

Liquid level instruments(43) are of two types, both of which are used in the U. S. One uses the differential pressure, recorded on a "Ringwaage", due to the hydrostatic pressure when very small flows of gas are bled into two tubes, one of which opens above the liquid surface and the other of which extends to a point near the bottom. This instrument was used for the hot separator of the sump phase, and for other services where heavy or dirty products were handled.

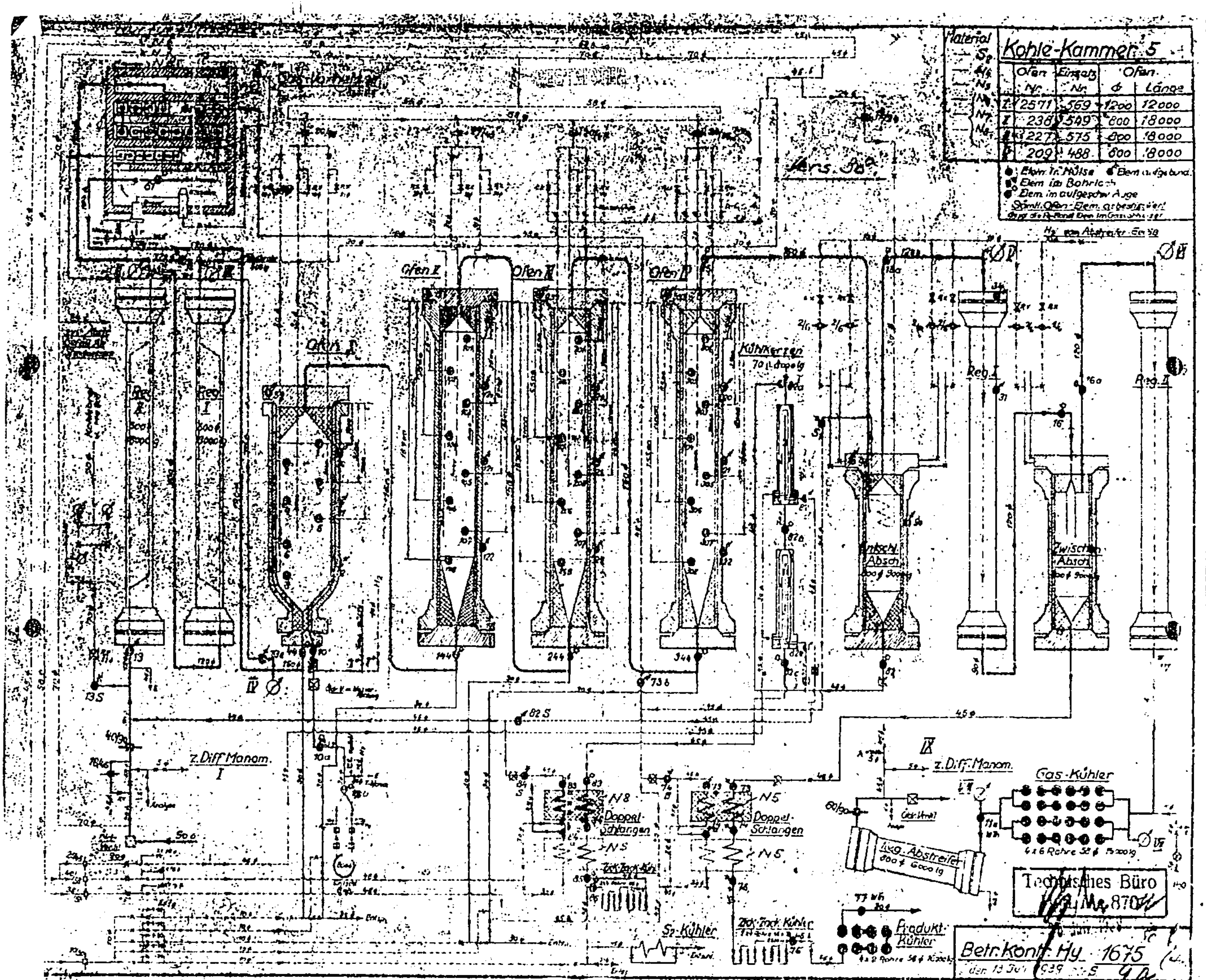
The second is the well-known displacement type where the buoyant effect of the rising level decreases the apparent

Tabelle für Eisen-Konstantan-Thermoelemente bezogen auf 40° C Klemmentemperatur.

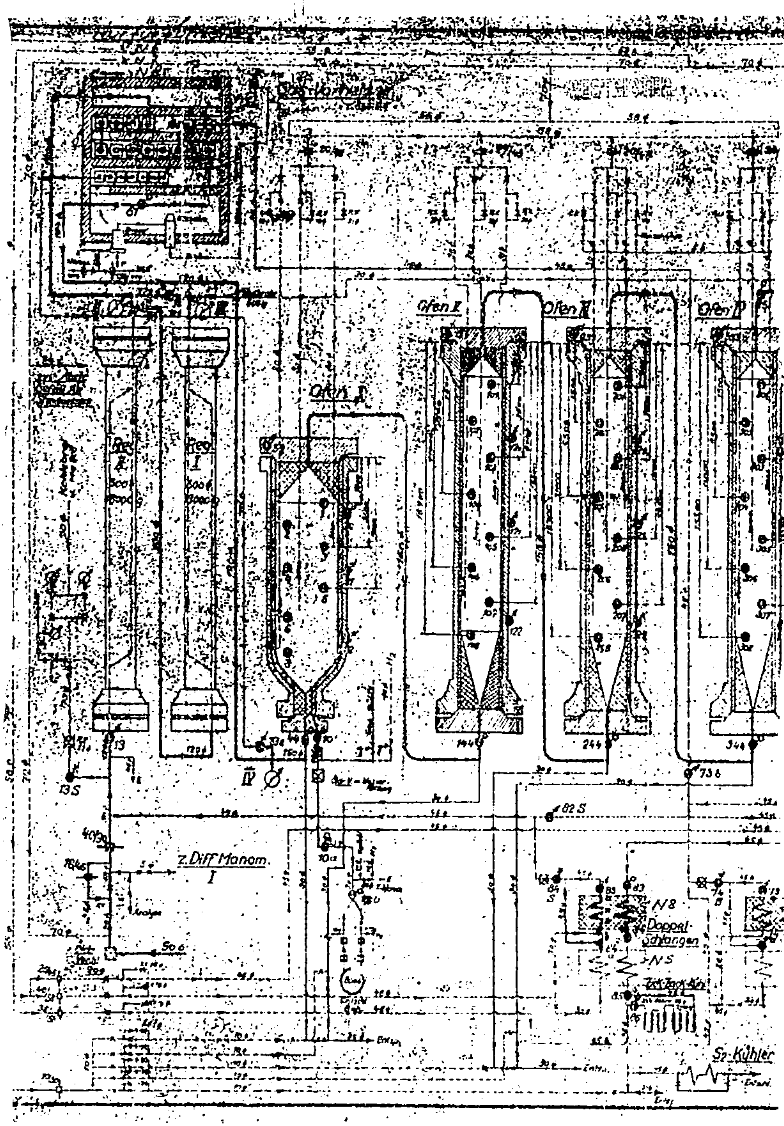
C°	Millivolt	C°	Millivolt	C°	Millivolt	C°	Millivolt	C°	Millivolt
— 195,8	— 9,78	58	0,94	120	4,37	182	7,86	244	11,49
— 78,5	— 5,77	60	1,04	122	4,48	184	7,98	246	11,61
0	— 2,07	62	1,15	124	4,60	186	8,10	248	11,73
2	— 1,97	64	1,25	126	4,71	188	8,21	250	11,85
4	— 1,87	66	1,36	128	4,82	190	8,33	252	11,96
6	— 1,77	68	1,47	130	4,93	192	8,45	254	12,08
8	— 1,66	70	1,58	132	5,05	194	8,57	256	12,20
10	— 1,56	72	1,69	134	5,16	196	8,68	258	12,31
12	— 1,46	74	1,80	136	5,27	198	8,80	260	12,43
14	— 1,36	76	1,91	138	5,38	200	8,92	262	12,55
16	— 1,25	78	2,02	140	5,50	202	9,03	264	12,67
18	— 1,15	80	2,13	142	5,61	204	9,15	266	12,78
20	— 1,05	82	2,25	144	5,72	206	9,27	268	12,90
22	— 0,95	84	2,36	146	5,83	208	9,38	270	13,01
24	— 0,84	86	2,47	148	5,95	210	9,50	272	13,12
26	— 0,74	88	2,58	150	6,06	212	9,62	274	13,24
28	— 0,63	90	2,69	152	6,17	214	9,73	276	13,35
30	— 0,53	92	2,80	154	6,28	216	9,85	278	13,47
32	— 0,42	94	2,91	156	6,40	218	9,97	280	13,58
34	— 0,32	96	3,03	158	6,51	220	10,09	282	13,70
36	— 0,21	98	3.14	160	6,62	222	10,21	284	13,82
38	— 0,11	100	3,25	162	6,73	224	10,32	286	13,93
40	0,00	102	3,36	164	6,84	226	10,44	288	14,05
42	0,10	104	3,47	166	6,96	228	10,56	290	14,16
44	0,20	106	3,59	168	7,07	230	10,67	292	14,28
46	0,31	108	3,70	170	7,18	232	10,79	294	14,39
48	0,41	110	3,81	172	7,29	234	10,91	296	14,51
50	0,52	112	3,92	174	7,40	236	11,02	298	14,62
52	0,62	114	4,04	176	7,52	238	11,14	300	14,74
54	0,73	116	4,15	178	7,63	240	11,26	302	14,85
56	0,83	118	4,26	180	7,75	242	11,38	304	14,97

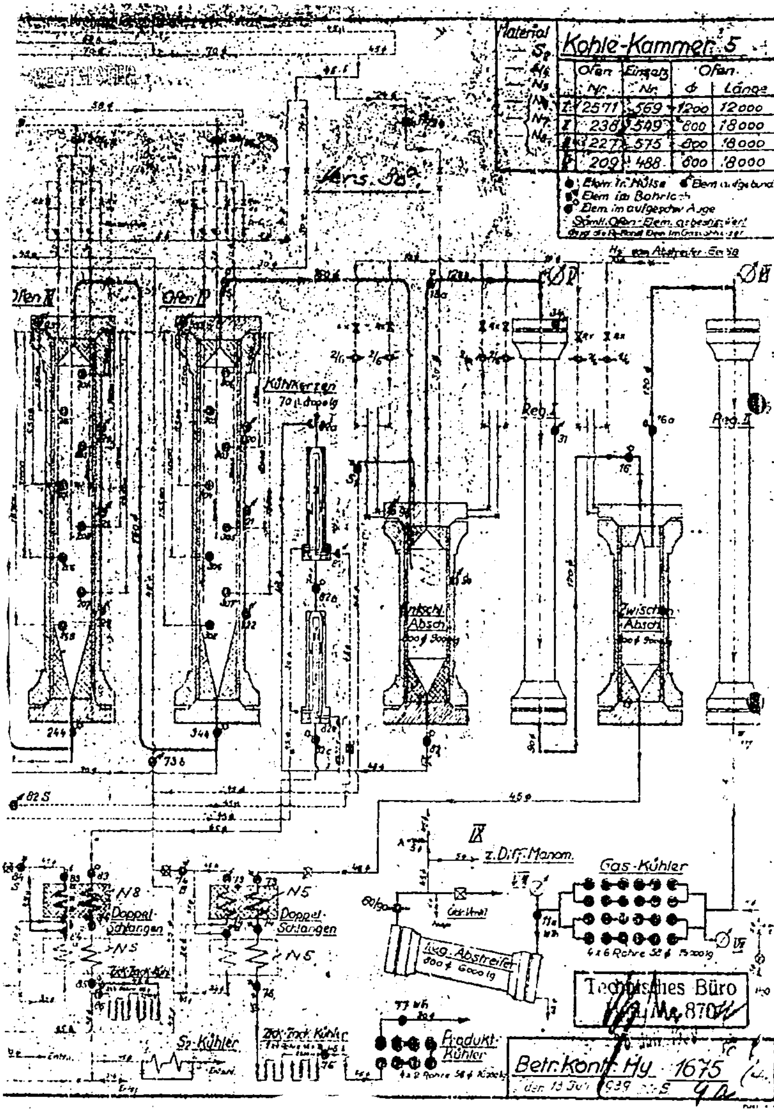
377

C°	Millivolt	C°	Millivolt	C°	Millivolt	C°	Millivolt	C°	Millivolt
306	15,08	368	18,67	430	22,29	492	25,94	554	29,64
308	15,20	370	18,78	432	22,40	494	26,06	556	29,76
310	15,31	372	18,90	434	22,52	496	26,18	558	29,88
312	15,43	374	19,02	436	22,64	498	26,30	560	30,00
314	15,54	376	19,13	438	22,75	500	26,42	562	30,12
316	15,66	378	19,25	440	22,87	502	26,54	564	30,24
318	15,77	380	19,37	442	22,99	504	26,65	566	30,36
320	15,89	382	19,48	444	23,10	506	26,77	568	30,48
322	16,00	384	19,60	446	23,22	508	26,89	570	30,60
324	16,12	386	19,72	448	23,34	510	27,01	572	30,73
326	16,23	388	19,83	450	23,46	512	27,13	574	30,86
328	16,34	390	19,95	452	23,57	514	27,25	576	30,99
330	16,46	392	20,07	454	23,69	516	27,37	578	31,11
332	16,57	394	20,18	456	23,81	518	27,48	580	31,24
334	16,69	396	20,30	458	23,93	520	27,60	582	31,36
336	16,81	398	20,42	460	24,05	522	27,72	584	31,49
338	16,92	400	20,53	462	24,17	524	27,84	586	31,61
340	17,04	402	20,65	464	24,28	526	27,96	588	31,74
342	17,15	404	20,77	466	24,40	528	28,08	590	31,86
344	17,27	406	20,88	468	24,52	530	28,20	592	31,99
346	17,39	408	21,00	470	24,64	532	28,32	594	32,11
348	17,50	410	21,12	472	24,76	534	28,44	596	32,24
350	17,62	412	21,24	474	24,88	536	28,56	598	32,36
352	17,74	414	21,35	476	25,00	538	28,68	600	32,49
354	17,85	416	21,47	478	25,11	540	28,80	602	32,61
356	17,97	418	21,59	480	25,23	542	28,92	604	32,74
358	18,08	420	21,70	482	25,35	544	29,04	606	32,87
360	18,20	422	21,82	484	25,47	546	29,16	608	32,99
362	18,32	424	21,94	486	25,59	548	29,28	610	33,12
364	18,43	426	22,05	488	25,70	550	29,40		
366	18,55	428	22,17	490	25,82	552	29,52		

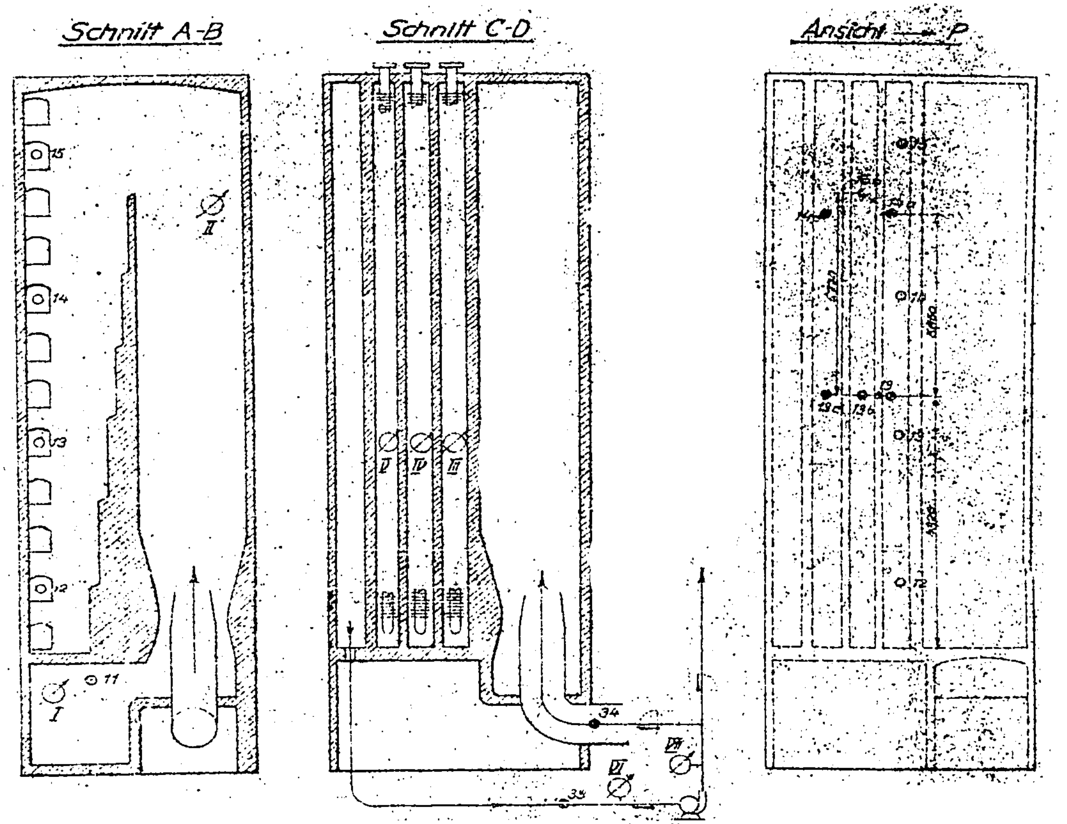


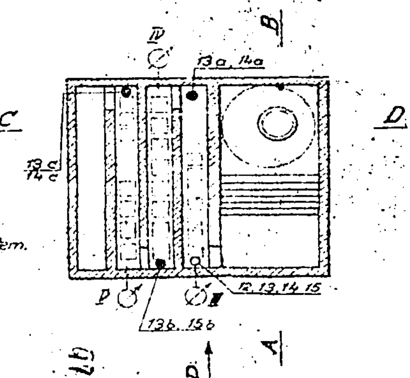
Kohle-Kammer 5
Material
Ofen Nr.
Einsatz Nr.
Ofen ⌀
Länge
2571
569
1200
12000
238
549
800
18000
227
575
800
18000
209
488
800
18000
Elem. in Hülse
Elem. aufgebund.
Elem. im Bohrloch
Elem. im aufgeschw. Auge
Vers. 98a
Ofen I
Ofen II
Ofen III
Ofen IV
Reg. I
Reg. II
Kühlkerzen
Zwischen Absch.
z. Diff. Manom.
Gas-Kühler
4 x 6 Rohre
Doppel-Schlangen
N8
N5
Sp-Kühler
Produkt-Kühler
Technisches Büro
Betr. Kont. Hy. 1675


z. Diff Manom.
I
Doppel-Schlangen
Sz-Kühler
13 S
82 S
73 b
144
244
344



Kohle-Kammer 5
Material
Ofen Nr. | Einsatz Nr. | Ofen Ø | Ofen Länge
I 2571 | 569 | 1200 | 12000
II 238 | 549 | 800 | 18000
III 227 | 575 | 800 | 18000
IV 209 | 488 | 600 | 18000
Elem. in Hülse
Elem. aufgebund.
Elem. im Bohrloch
Elem. im aufgeschw. Auge
Sämtl. Ofen-Elem. abgestimmt
Vers. 30a
Ofen II
Ofen IV
Kühlkerzen
70 l. Doppelg
Reg. I
Reg. II
Zwischen-Absch.
Zwischen-Abscheider
z. Diff.-Manom.
Gas-Kühler
4 x 6 Rohre 58 Ø
Flüss. Abstreifer
800 Ø 6000 lg
N8
Doppel-Schlangen
N5
Doppel-Schlangen
N5
Sz-Kühler
Produkt-Kühler
4 x 2 Rohre 58 Ø
82 S
73 b
Technisches Büro
Nr. 870
Betr. Kontr. Hy 1675
den 13 Juli 1939





Bemerkungen

- ○ Platin - Element
- ○ ChromniBi - Thermom. Elem.
- ● Eisen - Konst. Elem.
- ⌀ Druckmeßstellen

Gasvorheizer Ka 5

— Thermoskizze —

11. <u>Operation and Control of the Units (h)(cont'd.)</u>

weight of a suspended cylinder.(44) The Germans supported the cylinder on a coil spring so that an iron rod attached to the top of the cylinder rises into an induction coil with rising level. The increasing insertion of the iron rod changes the inductance of the coil which can be determined and recorded as proportional to the liquid level. The Brown electric flow meter in the U. S. uses a similar principle, one advantage of which is the complete absence of any packing or stuffing box. This instrument was used for cold separators, column kettles, etc. where the liquid was fluid at atmospheric temperature and was comparatively free from sediment or suspended matter.

Automatic control of variables in the high pressure coal hydrogenation plants is in its infancy in Germany, and as mentioned before, the design of the instruments is somewhat crude when compared to American practice. Electric, pneumatic, and combinations of the two types of control are utilized. The controlled valves are usually of the diaphragm type, often using balanced pressures on both sides of the diaphragm rather than spring loading in one direction. A crude form of valve positioner was developed to overcome the stuffing box friction which is invariably present in high pressure control valves. Some hydraulic valves were observed, but these were usually remote manual control valves rather than automatic. Solenoid valves were also built, but their use in large sizes was not frequent.

The automatic controls in the Leuna plant were as follows:

(1) Liquid level, cold separator.
(2) Temperature into first converter.
(3) Temperature into second converter.
(4) Liquid level, intermediate separator.
(5) Temperature control of descending first sump phase converter.
(6) Liquid level, hot separator.
(7) Liquid level, oil feed storage.

Not all of these instruments were used on all units,

11. Operation and Control of the Units (h)(cont'd.)

and they are arranged in order of decreasing numbers. A description of the controls and their applications is included in the appendix in a report "Selbsttätige Regelung der Sumpf-und Gas phase Kammern in der Hydrierung des Leunawerkes."

(i) High Pressure Joints and Closures.

There are no indications that any novel or improved joints or closures for high pressure vessels or piping have been used in the German High Pressure Coal Hydrogenation Industry. The flanged piping joints used screwed, through-bolted flanges, and the gaskets were lens rings very similar to standard American high pressure practice. The lens rings were usually made of the same material as the pipe, and the joint was a line contact between the ring and the ground end of the pipe itself.

No self-sealing gasket designs were found. The large vessel closures all had the bolts in tension, and the entire gasket load was taken by the bolting. The bolts and nuts were usually made from one of the S steels for 325 atm. service, and from one of the K steels for 700 atm. service. Bolts were studded into the end of the vessel and passed through drilled holes in the forged head, with the nuts on top of the head.

Two general types of large vessel closures were found. The simpler type had no separate gasket, but the tapered end of the head entered the vessel and wedged the stainless steel liner of the vessel against the vessel wall. This design is shown on drawings C-1, C-5, and C-10 in the appendix to this report. The second type of closure uses a separate steel ring which has a facing of 1mm thick pure aluminum on the seating surfaces. The seating surfaces are bevelled at a 30^{o} angle so that the aluminum is wedged into a recess in the head on the head seating surface and between the head and the vessel wall on the other surface. A detail of this construction is shown on drawing C-4 in the appendix, and the equipment shown on drawings C-7, C-11, and C-14 uses the same general design. Apparently

11. Operation and Control of the Units (i)(cont'd.)

the separate gasket is used only when the vessel has no stainless steel liner.

12. Materials of Construction.

One of the great problems in high pressure high temperature processes of any kind is the development or selection of construction materials that will stand up under the operating conditions. For coal hydrogenation the primary requirements of materials for the high pressure equipment are:

(1) Hydrogen resistance at high temperature and with high hydrogen partial pressure.
(2) High tensile and creep strength at the operating temperature.
(3) Resistance to H_2S and Cl_2 corrosion.

A great amount of experimental and development work has been done in Germany in an effort to produce suitable steels, but at best a compromise must be made between the various properties listed above. Their problems were multiplied by wartime shortages of molybdenum, tungsten, and chromium. Austenitic 18-8 Cr-Ni steel was fairly satisfactory, but the tonnage were so great that Germany could not supply the high chromium and nickel requirements.

The early experimental work on the hydrogenation process was done with vessels of various carbon steels, but hydrogen attack was severe and the vessels failed in a very short time.(31) Low alloy chrome nickel steels were then used, but nickel appeared to decrease the hydrogen and H_2S resistance of the alloy. Molybdenum was then substituted for nickel, and gave better alloys with less alloying metal. About 0.5 percent Mo gave high temperature strength properties equivalent to 2.0 percent Nickel. Krupp P469 (N6) was the first steel of this type. In chronological order, steels N6, N8, V 2AED, and N10 were developed, each being an improvement over previous material for high temperature high pressure hydrogen service. Then, during the war, N 8V and N9 were used to save critical alloys, but were not nearly so resistant to corrosion as the earlier steels.