

Alkazid Process Data

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Item 22a

Application of Disintegrators for the Extraction of H_2S from Gases by Means of Alkazid-solution

In the early days of the development of the Alkazid process, it was experienced that H_2S is more rapidly absorbed by the Alkazid solution than CO_2 . But despite the difference of the velocity with which the gases are absorbed, it is not possible to extract chiefly H_2S from H_2S and CO_2 -containing gases by the application of packed scrubbers (Raschig-ring-packing) and Alkazid solution as an absorbing agent. In order to secure the desired purification of the gases from H_2S , such a long period of contacting time between gas and solution must be maintained that larger volumes of CO_2 are extracted also. In the average, during the purification of hydrogenation-return-gases by means of scrubbers, with every 100 parts of H_2S , 60 parts of CO_2 are extracted simultaneously. The absorptiveness of the solution is but partly utilized for the H_2S -extraction.

In connection with other experiments, when the solution as a result of overloading of the scrubber had become foaming, it was observed that the absorbing velocity of the solution for H_2S could be increased many times if the solution was widely spread over a large surface area and brought into an extremely intimate contact with the gas to be scrubbed. The described observations seemed to indicate that the desired better utilization of the absorptiveness of the solution against H_2S could be reached if such scrubbing devices are

employed which secure the shortest contact time of the gas with the widely dispersed solution. At first preparations were made to extract the gases by means of an atomized solution. Since it was almost impossible to supply nozzles which had such a large capacity as required, it was decided to employ disintegrators for the intended experiments.

A normal Zochocke disintegrator, 600 x 250 mm, was mounted (volume of the cage 100 liters) which was operated in the normal way with refreshed solutions and crude gas. The results, which are represented by table 1, show that even if no satisfactory extraction was obtained (column 4), a high H_2S -absorption could be realized (column 11). The most important observation was that by the application of a disintegrator no CO_2 was extracted from the gas. It could be expected that with a smaller gas volume to be treated or by operating the disintegrators in series, a high H_2S -concentration in the spent solution and an efficient H_2S extraction would be obtainable. No better results could be obtained by operating packed scrubbers in series. The H_2S concentration of the solution was always kept constant, whereas the CO_2 absorption of the solution was increased. It happened sometimes that the previously absorbed H_2S was removed by the following absorption of CO_2 .

The aim of the following experiment was to prove that, unlike a scrubber, a disintegrator was able to increase the H_2S content of a solution which had already been saturated during its passage through a scrubber. A disintegrator was operated with a solution which had already been saturated in a scrubber and crude gas was fed to the apparatus. Table 2 contains the results of the experiments. It is easily seen that a further saturation of the solution took place. Comparing columns 9 and 12, it can be observed that during its passage through the disintegrator the H_2S -gas value increased from 12-14 to 17-21 indicating an increase of 5-7 $m^3 H_2S$ per 1 cu. m. of the applied solution. The H_2S content of the hydrogenation gas was simultaneously reduced. No considerable volumes of CO_2 were extracted.

The disintegrator was arranged in such a manner that it provided for a pretreatment of gases before the "North" scrubber. The gas was fed to the disintegrator and then to the following scrubber whereas the solution, applying a countercurrent flow, was pumped over the scrubber and then fed to the disintegrator. By the application of that method it was now possible to treat 1,400 m^3 hydrogenation-gas instead of the previously extracted 1,100 m^3 . The H_2S content of the extracted gas was 7-8 g/ m^3 . The test was run for 8 days, the results obtained are represented by table #3. Columns 11 and 13 show that the H_2S -absorption of the solution was about 2.3 to 3.5 $m^3 H_2S$ per m^3 solution higher than that which was obtained in the "South" scrubber which was operated under the same conditions except that no disintegrator was employed. The higher H_2S absorption corresponds with a lower CO_2 -absorption for the disintegrator experiment. (Compare columns 12 and 14). But the absorptiveness of the solution is still not fully utilized for the H_2S extraction due to the volume of CO_2 which is absorbed in the final stage of the process.

Finally 2 disintegrators were operated in series whereby a countercurrent flow of the liquid and the gas was applied. With such a method a satisfactory purification of the gas and a considerable H_2S absorption of the solution was obtained. The H_2S absorption was as high as 17-20 cu. m. per 1 m^3 solution compared with 11-13 cu. m. H_2S per m^3 solution when a scrubber was used. (Table 5, col. 11, table 4) The result was that the hydrogenation gases which contained

90-100 g H₂S per m³ could be purified to 6-8 g H₂S with only 3.5 m³ of the solution whereas 4.6-4.9 m³ of the solution were required to bring down the H₂S content of the gas to 12-13 g/m³ if a scrubber was used.

The spent solution of the disintegrator process was separately recirculated. The stripping process consumed 100 kg. steam per 1 m³ solution which is the same volume as consumed by the scrubber process. Obviously the steam consumption decreases correspondingly with the decreasing volume of the solution. The quantity of steam which is consumed for the extraction of 1 metric ton of H₂S-sulfur amounts to 5.25 tons if a scrubber is employed, the corresponding figure for the disintegrator process is as low as 3.75 tons.

Table 1

Experiments using a refreshed solution

Arrangement: The disintegrator is operated with refreshed and crude gas

Hy-gas m ³ /hr.	Sol. m ³ /hr.	H ₂ S		CO ₂		Gas values of the Sol.					
		crude g/m ³	finished gas g/m ³	crude gas %	finished gas %	Total	Before dis.	behind d.	H ₂ S	CO ₂	H ₂ S
1	2	3	4	5	6	7	8	9	10	11	12
1,250	6	66.0	15.2	4.4	4.0	-	-	-	-	-	-
1,000	5	66.4	11.8	5.3	4.6	16.0	4.8	11.2	23.0	12.7	11.1
1,000	4	71.0	18.4	4.7	4.1	16.0	4.5	11.5	24.4	13.4	11.1
1,000	3	71.0	25.4	4.6	4.2	16.7	4.8	11.9	26.4	15.7	11.1
1,000	2	70.0	31.0	4.3	4.4	16.2	4.3	11.9	28.8	28.8	11.1
570	2	71.0	15.3	-	-	-	-	-	-	-	-

Table 2

Experiments with saturated solution (solution has been saturated in a separate scrubber)

Procedure: The disintegrator serves as initial H₂S-extractor before the Hy-gas is fed to the scrubber "North". The saturated solution is withdrawn from the scrubber "South" and transferred to the disintegrator. The scrubber "North" is operated with a separate cycle of the solution.

Hy-gas m ³ /hr.	Sol. m ³ /hr.	H ₂ S		CO ₂		Gas values of the Solution							
		before disintegrator g/m ³	behind disintegrator g/m ³	before disintegrator %	behind disintegrator %	total	Before	behind	Total	H ₂ S	CO ₂	H ₂ S	CO ₂
1	2	3	4	5	6	7	8	9	10	11	12	13	14
1,000	4	65.0	34.0	4.6	4.4	29.8	14.3	15.5	34.7	18.7	16.1		
1,250	5	59.0	35.6	5.1	4.8	30.8	15.2	15.6	34.7	19.8	14.1		
1,250	3.5	62.1	35.1	5.1	5.1	29.8	13.6	15.2	35.4	20.1	11.5		
1,250	3.5	65.0	35.8	4.5	4.6	29.0	13.7	15.3	35.0	19.9	15.1		
1,250	3.5	63.4	33.0	4.6	4.7	27.0	12.9	14.1	33.0	19.9	15.1		
1,250	3.0	63.7	42.6	4.9	4.4	23.6	14.1	14.5	35.2	21.1	14.1		
1,250	3.0	66.0	40.3	4.6	5.2	29.0	14.1	14.9	35.0	20.1	14.1		
1,250	4.2	67.5	46.0	4.6	4.6	28.8	11.9	16.9	34.0	17.4	16.1		

Table 3

Experiments with a saturated solution, counter current flow operation.

Test run April 16-21, 1934

Procedure: 1,400 m³/hr. Hy-gas are led through the disintegrator and later on through the scrubber "North", 5 m³/hr. refreshed solution (gas value 15) are fed to the scrubber "North". The solution leaving scrubber "North" is sprayed into the disintegrator. The saturated solution is pumped to the stripping column. The scrubber "South" (parallel test) is operated with 1,400 m³/hr. Hy-gas and 5 m³/hr. solution.

Date	Hrs.	H ₂ S in g/m ³ crude gas	H ₂ S in g/m ³ behind disin.	behind "North" scrubber	behind "South" scrubber	
4/17/34	7	66.5	44.4	7.2	5.8	
4/18/34	7	67.5	34.1	5.1	5.4	
4/19/34	7	87.2	44.3	7.7	8.6	
4/20/34	7	82.6	52.0	8.1	8.9	
4/21/34	7	77.7	42.4	8.6	7.0	
4/22/34	7	76.8	44.8	8.5	7.3	
4/23/34	3	84.9	49.0	7.0	8.4	
4/24/34	5	66.0	44.0	7.7	5.9	
	(1)	(2)	(3)	(4)	(5)	(6)

(continued below)

Absorbed gas vol. in the disintegrator		in the scrubber "North"		Disintegrator + scrubber "North"		in the scrubber "South"	
H ₂ S	CO ₂	H ₂ S	CO ₂	H ₂ S	CO ₂	H ₂ S	CO ₂
7	8	9	10	11	12	13	14
4.40	0	7.20	4.75	11.60	4.73	9.33	7.60
6.55	0	5.70	4.38	12.25	4.60	9.58	5.10
8.40	0	7.17	1.36	15.57	1.36	12.13	3.30
6.00	0	8.60	3.37	14.60	3.37	11.35	4.62
6.91	0	6.61	3.50	13.36	3.80	10.85	4.30
6.36	0	7.00	4.29	13.36	4.20	10.65	5.29
5.60	0	5.54	2.60	12.14	2.60	10.71	5.60
4.80	0.9	7.60	5.00	12.70	3.90	9.26	4.40
%	0						

Table 4

Gas-absorption of the Alkalic solution in the 6,000 m³ scrubber

Date	Gas Vol.	Sol. per 1000 gas	H ₂ S g/m ³ crude gas	CO ₂ % final gas	Total steam consumed kg/m ³ gas	Absorbed gas vol. of the sol. m ³ gas						
1943	Vol. m ³ /h	m ³ /h	g/m ³ gas	% gas	kg/m ³ gas	H ₂ S CO ₂ H ₂ S+CO ₂						
	1	2	3	4	5	6	7	8	9	10	11	12
5/4	2,900	10	61.0	5.1	1.5	100	12.5	8.0	20.3			
5/12	2,500	10	70.0	5.3	1.70	100	11.2	6.5	17.7			
5/12	2,500	10	70.0	5.7	1.7	100	12.9	8.0	20.9			
5/14	3,000	14	67.3	7.0	1.2	100	9.0	6.0	15.0			
5/15	2,200	10	70.0	4.6	1.3	100	11.5	6.0	17.5			
5/20	2,600	12	77.3	5.7	1.3	100	11.0	6.0	17.0			
5/21	1,700	8	70.0	4.3	1.3	94	10.6	6.8	17.4			
5/23	2,300	10	70.0	4.4	1.5	97	11.6	7.8	19.4			
5/26	3,250	16	108.2	12.1	3.0	100	13.6	4.7	18.3			
5/26	3,500	16	93.0	13.4	3.4	100	12.2	4.3	16.5			

Table 5

Gas absorption of the Alkazid solution when 2 disintegrators are operated in series
Test period May 11-27, 1934

Test No.	Gas Vol. m ³ /h	Alk. sol. m ³ /h		H ₂ S g/m ³ crude gas			CO ₂ %	
		1,000m ³ gas	1,000m ³ gas	behind disint. 1	behind disint. 2	crude gas	final gas	
0	1	2	3	4	5	6	7	8
1	1,500	5.0	3.3	95.5	55.1	11.4	4.8	5.2
2	1,000	3.5	3.5	104.5	47.1	10.9	5.0	5.6
3	1,000	3.5	3.5	111.55	56.6	10.9	5.1	5.5
4	860	3.0	3.5	108.2	48.0	8.7	5.2	5.8
5	860	3.0	3.5	95.7	44.8	8.4	5.7	6.2
6	570	2.0	3.5	92.4	42.6	6.1	6.3	6.1

(continued below)

Steam consumpt. kg/m ³	Temp. of the sol. at the entr. of the stripper	Gas absorption of the Alk. sol. m ³ gas per m ³ solution	
		H ₂ S	CO ₂
9	10	11	12
120	86°C.	17.7	zero
100	87"	18.8	"
106	89"	20.0	"
100	90"	20.0	"
100	88"	17.5	"
100	86"	17.2	1.0

The "Feed-Scrubber" and its Applicability for the H₂S-extraction from Hydrogenation-return-gases by Means of Alkazid-solution

If a very short contact time between gas and Alkazid-solution is maintained and simultaneously the solution is distributed over a very large surface area, it is possible to preferentially extract H₂S from H₂S and CO₂-containing gases. As shown in a previous report, it was possible to absorb 18 m³ H₂S per 1 m³ Alkazid solution and to get a satisfactory purifying effect by the application of disintegrators as a scrubbing agent. The Walther-feld scrubber which uses a centrifuge force for the distribution of the scrubbing liquid was especially interesting due to its low energy consumption and its particular design. The desired surface-low time-scrubbing is repeated eight times by scrubbing groups which operate in series.

At first we tried to construct some centrifugal acting groups. But since we were fortunate enough to obtain the test scrubber from the Feld company, Essen, we abandoned our efforts to construct our own scrubber. The beginning of the intended test runs was delayed because it was necessary to repair the pilot scrubber before it was ready for the experiments.

Description of the Scrubber

The inside diameter of the scrubber was 800 mm, its height from the bottom to its cover 3,160 mm. Seven rotating conical frustrums were arranged at the axle. The trays of liquid were fixed to the walls of the scrubber. The sixth group from the top was omitted. The pictures 1 to 4 and sketch 1 show the design of the scrubber. The area of a group which is sprayed by the liquid is framed by green lines (picture 2). That part of the sprayed area through which the gas passes, the scrubbing area is hatched (red hatching). According to picture 2 the scrubbing area of a group is 25 liters, the total scrubbing area is 175 liters.

With 300 r.p.m. of the axle, the average peripheral velocity of the edge of the conical frustrum was 8 m per sec. The axle of the commercial scrubbers can be raised or lowered ± 25 mm, thus altering the volume of the sprayed liquid. Since the pilot washer was not equipped with such an installation, the volume of the sprayed liquid was controlled by the revolution of the axle.

It is to be assumed that the liquid is nowhere near as widely dispersed as in a disintegrator, but the volume of the dispersed liquid is extremely large. According to information received from the Feld Company 90 liters of liquid are sprayed per sec. per cone if the scrubber has a diameter of 3,000 mm. and an output of 50 m³ liquid per hour, whereby the peripheral velocity equals that of the pilot scrubber. The eight groups of the pilot washer should disperse 2,600 cu. m. of liquid per hour.

ExperimentsHydrogen sulfide absorption and gas volume treated by the scrubber

Since the time period during which liquid and gas are in intimate contact is of decisive importance for the H₂S-absorption of the solution if H₂S- and CO₂ containing gases are treated, the efficiency of the scrubber is determined for various gas volumes to be treated (gas volumes 1,000 cu. m. per hour). For each gas volume the minimum volume of the solution was determined which was required to purify the gases to about 5 g/m³ H₂S. Each test was run over such a period of time that it was made sure that a state of equilibrium was obtained.

A refreshed solution which was withdrawn from the cycle of system I of the commercial plant was employed for the tests. After enrichment in the test scrubber the solution was returned to system I. The solution together with that of the commercial system was refreshed in stripper I whereby 100 kg. steam were consumed per m³ solution. With a determined gas value of 13-14, the refreshed solutions were of excellent quality. If comparisons must be made between the gas volume which were absorbed in the Feld scrubber and those which were observed in the disintegrator tests, it must be born in mind that by the formerly used stripping columns the gas value of the refreshed solution was still 16. To obtain proper results 1-2 points should be deducted from the present H₂S concentration or 1-2 points must be added to the formerly obtained results.

Table 1 represents the obtained results. It is easy to be seen that by a Feld-scrubber very large volumes of H₂S are absorbed with simultaneous good purification effect: With outputs of 375-750 cu. gas per hour 17-21 cu. m. H₂S are absorbed by 1 m³ of solution. The H₂S absorption in the Feld-scrubber is at least as good as that which was obtained by the disintegrator system (18 m³ H₂S per 1 m³ solution).

Table I
Influence of the gas output on the gas absorption of the solution
(Experiments carried out in October and November 1934)

Test No.	Test carried out hr.	Gas m ³ /hr	Spl. m ³ /hr.	Energy consumpt. kwh	H ₂ S in g/m ³ crude final	CO ₂ % vol. final	CO ₂ gas	CO ₂ gas	Absorption ³⁾ calc. on the scrubbing area	Time passage	
											gas
1	9	100	0.33	2.7	85.2	27.3	4.5	1.2	12.3	10.0	6.30 sec.
2	9	100	0.33	2.5	82.0	28.5	3.8	1.5	11.4	7.0	" "
3	7	250	0.83	2.8	85.2	17.0	3.8	1.6	14.3	6.6	2.5 "
4	13	375	1.00	not det.	88.5	10.8	5.1	2.8	13.6	5.8	" "
5	8	375	1.25	2.9	86.5	9.1	4.0	2.4	16.2	4.8	1.68 "
6	3	375	1.41	not det.	90.0	7.2	5.4	2.9	15.4	6.6	" "
7	6	375	1.16	" "	83.4	8.4	4.9	2.8	17.0	6.8	" "
8	6	375	1.16	" "	89.4	9.4	5.0	3.4	18.1	5.2	" "
9	7	500	2.00	3.2	85.1	2.8	not	det.	14.4	not det.	1.26 "
10	16	500	1.52	not det.	88.6	4.9	5.5	4.2	19.4	4.3	" "
11	17	500	1.36	" "	86.8	8.3	5.2	3.7	20.2	5.5	" "
12	9	500	1.35	" "	84.9	9.9	4.7	4.0	19.8	2.6	" "
13	19	500	1.35	" "	88.4	7.1	4.6	3.7	21.0	3.3	" "
14	9	750	2.50	3.2	93.5	4.6	not	det.	18.6	not det.	0.84 "
15	14	750	2.50	Not det.	82.7	3.6	4.9	3.8	16.6	3.3	" "
16	7	750	2.50	" "	82.3	3.0	4.7	4.0	16.8	2.1	" "
17	35	750	2.00	" "	79.6	7.4	4.8	4.3	18.9	1.9	" "
18	21	750	2.30	" "	90.0	5.1	5.0	4.4	19.5	2.0	" "
19	4	1,000	2.50	3.4	84.1	25.7	3.6	3.1	16.3	2.0	0.63 "
20	8	1,000	3.33	3.2	83.2	19.4	4.2	3.3	15.9	1.5	" "

1) Average peripheral velocity of the outside of the conical frustrum 8 m/sec.

2) m³ H₂S per 1 m³ solution

3) m³ CO₂ per 1 m³ solution

Table II
Gas Absorption of the Solution depending on a various output of the Scrubber

Properties of the solution	Output m ³ Hydrogenation-gas per hour					
	total gas H ₂ S	+) 100++) total gas H ₂ S	250 total gas H ₂ S	375 total gas H ₂ S	500 total gas H ₂ S	750 total gas H ₂ S
Refreshed sol.	14.6	55.0	13.2	4.0	14.0	4.0
behind group 1	15.0	4.8	14.6	4.2	14.0	4.2
" " 2	16.2	4.9	15.6	4.5	14.8	4.4
" " 3	17.0	4.8	16.4	4.7	16.6	5.4
" " 4	18.2	5.3	16.6	5.0	19.3	7.8
" " 5	22.4	8.0	19.4	5.2	24.2	11.4
" " 6	30.8	12.3	23.6	8.7	28.7	16.2
" " 7	34.8	15.4	29.6	15.6	32.2	19.6
Feed scrubber exit	54.8	17.4	32.2	17.5	34.0	20.1
Total H ₂ S absorpt.		12.4		13.5		16.1
Total CO ₂ " +++)		7.8		5.5		4.0

+) vol. gas per 1 vol. solution
 ++) " H₂S " " "
 +++) " CO₂ " " "

Table 3
Influence of Various Factors on the Solution Absorption

Test No.	Test carried out hrs.	Gas m ³ /hr	Sol. m ³ /hr	Energy consumpt. kWh	H ₂ S in crude gas	H ₂ S in final gas	CO ₂ content of gas	CO ₂ in crude gas	CO ₂ in final gas	Absorption H ₂ S	CO ₂	Average peripheral velocity of the cone
a) Greatest CO ₂ content of gas												
21	10	750	2.5	3.2	82.7	3.6	4.9	3.8	16.6	3.3	8 m/sec	
22	10	750	2.5	3.2	78.7	5.5	10.5	8.5	15.3	6.0	"	
23	14	750	2.5	3.2	74.8	13.7	14.9	12.4	12.8	7.5	"	
b) Change in the speed of the shaft												
10	16	500	1.52	3.0	88.6	4.9	5.5	4.2	19.4	4.3	8 m/sec.	
24	4	500	1.35	6.5	84.4	3.2	5.2	3.7	21.0	5.6	11 m/sec.	
25	9	500	1.35	1.5	88.1	7.1	4.6	3.7	21.2	3.3	5.5 m/sec.	
26	3	500	1.40	1.5	84.0	6.4	5.0	3.9	19.4	3.9	5.5 m/sec.	
27	11	750	2.44	1.6	89.7	5.1	4.7	4.0	18.2	2.1	5.5 m/sec.	
c) Change in the solution												
Solution with 14 total gas and 4 H ₂ S (System 1)												
27	11	750	2.44	1.6	89.7	5.1	4.7	4.0	18.2	2.1	5.5 m/sec.	
Solution with 16 total gas and 9 H ₂ S (Circular disintegrator)												
28	7	750	2.64	1.4	88.0	7.2	4.6	3.8	16.2	2.5	5.5 m/sec.	
29	12	750	2.35	1.5	87.1	9.7	5.1	4.1	17.3	3.2	5.5 m/sec.	
30	15	750	2.20	1.5	81.4	9.1	5.3	4.3	-17.2	3.4	5.5 m/sec.	

FIGURE 1

COURSE OF THE H₂S ABSORPTION DEPENDING ON VARIOUS OUTPUTS OF THE SCRUBBER

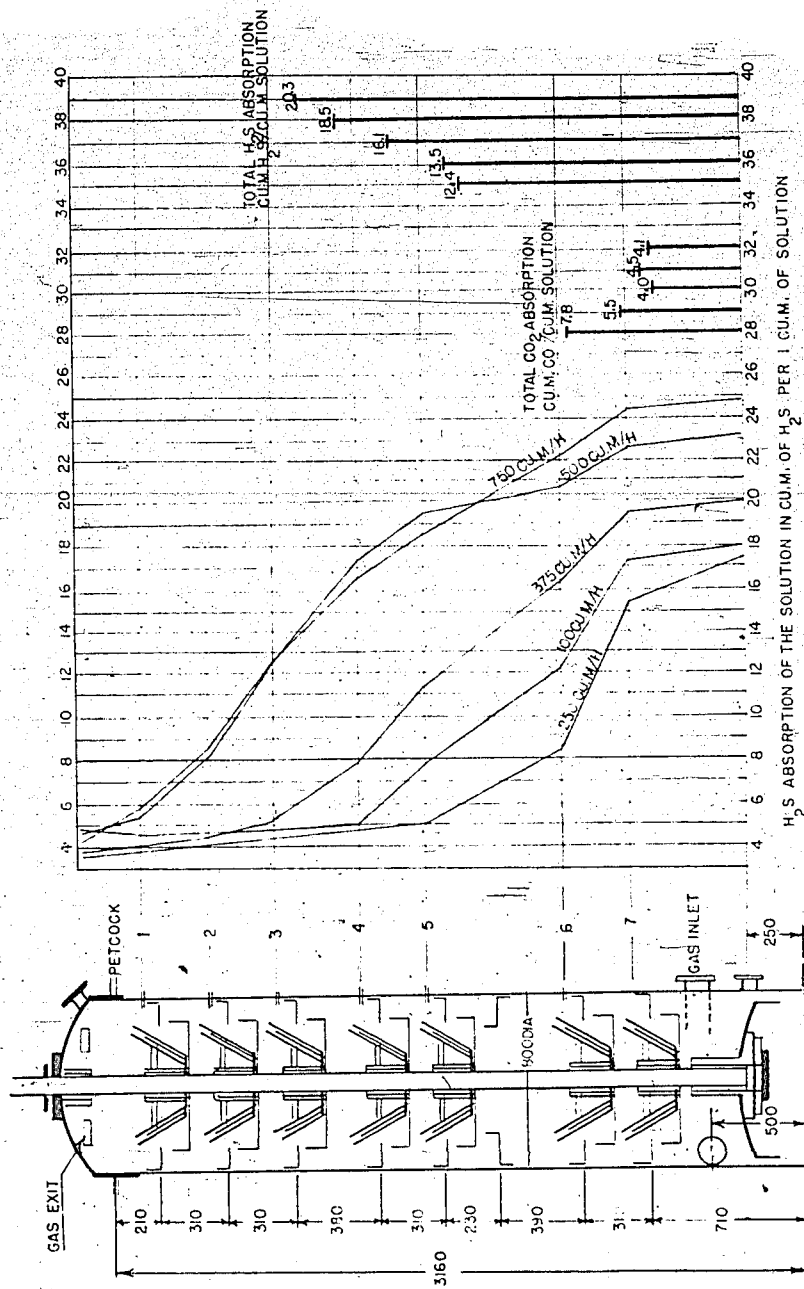
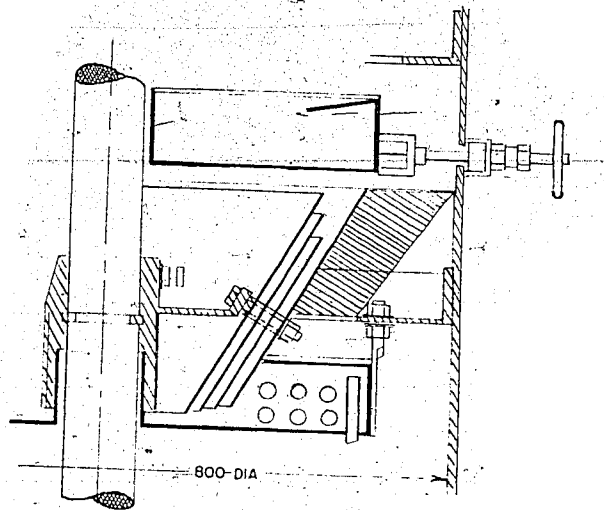


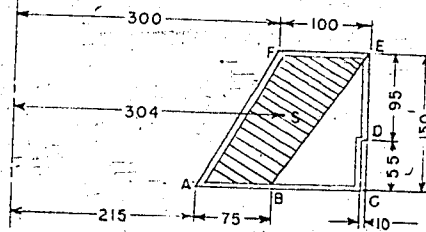
FIGURE 2

SCALE 1:5



SPRAYED SPACE A-C-D-E-F =
0.0425 CU.M. = 42.5 L.

SCRUBBING SPACE A-B-E-F =
0.025 CU.M. = 25 L.



G.C.E. 7/12/47

But there is the big difference that the high H_2S absorption is obtained in combination with a better purification effect (3-5 g/ m^3 H_2S) than was observed during the disintegrator tests (8-10 g/ m^3 H_2S). Furthermore the energy consumption is much less than that of a disintegrator. For the purification of 1,000 m^3 gas 9 kwh were consumed by two disintegrators operated in series, 2-3 kwh by the Feld scrubber operated in series. Furthermore table 1 shows that the favorable conditions with respect to large absorbed gas volumes do not exist if smaller gas volumes than 375 m^3 /hr are treated, or if the contact time is longer than 1.7 sec. Gas and solution are in contact over such a long period of time that larger volumes of CO_2 are absorbed than H_2S . (See test 1 and 2 and figure 1).

With a gas output of 1,000 m^3 per hour a satisfactory purification effect could not be obtained because the scrubber was overloaded. The proper volume of a Feld-scrubber for the Alkazid desulphurization which must be carefully observed is 300 liters scrubbing volume for 1,000 m^3 gas (\pm 35% per hour).

The progress of the H_2S absorption in the various groups is shown in table 2 and figure 1. If the scrubber is operated under favorable conditions, the total number of the groups participate in the absorption of the H_2S . But up to 5 groups did not participate in the H_2S extraction if smaller gas volumes are treated. Those 5 groups which could be found at the entrance and exit of the scrubber absorbed, however, more or less CO_2 , thus decreasing the volume of the solution which is available for the H_2S extraction. Therefore should a Feld-scrubber be operated with smaller gas volumes than it was designed for a more favorable gas absorption value will be obtained by eliminating a corresponding percentage of the groups.

H_2S -extraction and CO_2 -content of the Gas

With an equivalent H_2S extraction the solution absorbs more CO_2 if a Feld-scrubber is applied (20-30% of the extracted H_2S), whereas a disintegrator absorbs not more than 5-10% of the extracted H_2S . Since in the future the proportion $H_2S:CO_2$ from the present 1:1 will change to 1:2, the influence of a higher CO_2 -content of the gas on the H_2S -extraction of a Feld-scrubber was investigated. The experiments were carried out in such a manner that CO_2 was added to the gas before it was fed to the scrubber. Table 3a contains the obtained results. The H_2S extraction decreases but slightly if the proportion of $H_2S:CO_2$ = 1:1. But with a $H_2S:CO_2$ proportion = 1:3, the CO_2 had a deteriorating effect. No satisfactory purification effect could be obtained if 13 m^3 H_2S were absorbed by 1 m^3 solution.

Variations in the Axle Speed

An increase of the revolutions of the shaft from 300 r.p.m. to 400 r.p.m. did not result in a better gas absorption of the solution, but the energy consumption was twice as high as before (table 3b). But the efficiency of the scrubber was as good as before when the revolutions were decreased to 200 r.p.m. The energy consumption was now 1.5 kwh against 3 kwh before. The energy consumption of a Feld scrubber is not more than 2 kwh per 1,000 m^3 gas if hydrogenation-return gas is desulphurized.

Effect of the Refreshing Process

In most cases the solutions employed for the experiments after stripping contained mostly CO₂ and only small amounts of H₂S. But if a Feld-scrubber is employed which absorbs preferentially H₂S such solutions will be obtained after refreshing which contain more H₂S than CO₂. In order to make sure that equally good results are obtained additional experiments were carried out with solutions from the disintegrator cycle which are supposed to contain higher amounts of H₂S. Table 3c shows how equally high absorption was obtained. If the purification effect decreased a little, it must be born in mind that the disintegrator solutions were not so highly refreshed than the extremely well refreshed solutions which originated from the large strippers.

Summary

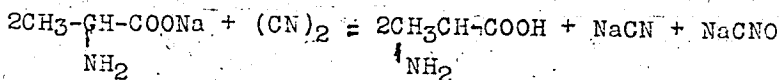
Employing a Feld-scrubber for the H₂S-extraction of the hydrogenation-return-gases it is possible to obtain equally high H₂S-absorption as was experienced during the disintegrator experiments. The Feld scrubber, however, has the advantage over the disintegrator in that the large volumes of H₂S which are absorbed can be obtained with a lower energy consumption combined with a higher effect of the extraction.

Item: 22b

Eliminating of Difficulties Encountered in Using the Alkazid-Process

Both of the Alkazid pilot plants and the commercial plant showed almost the same difficulties. The following phenomena were observed:

1. The efficiency of the Alkazid-solution decreased substantially when it was employed for the purification of coke oven gas.
2. Corrosion trouble was observed at the bottom of the boiler and in the adjacent hot pipes and pumps.
3. The small Leuna pilot plant which was operated with the sodium salt of oxyethyl-glycocoll corroded very badly after it was again put in operation.
4. The deterioration of the efficiency of the Alkazid solution when purifying coke-oven-gas at Heuls can be traced back to the presence of cyanogen (CN)₂. The cyanogen is supposed to react with the Alkazid-solution according to the following equation.



Na-alanine + cyanogen = alanine + Na-cyanide + Na-cyanate. In addition to alanine sodium cyanide and sodium cyanate are formed. By the following heat refreshing of the solution the sodium cyanide is decomposed with formation of sodiumalanine and liberation of hydrocyanic acid (HCN) resulting in a solution which is capable of repeated absorption. The sodium cyanate however cannot be decomposed into the sodium alanine and cyanic acid resulting in a decrease of the absorbing agent. Even if the Leuna- and Oppau-laboratories could not find substantial amounts of the sodium cyanate, the explanation presented above should be the reason for the deterioration of the solution.

Improved Process

The next step was to remove the cyanogen from the gases by a cheap and simple pretreatment of the gases to be purified. Sodium carbonate solution was found to be an effective agent for the desired extraction of the cyanogen. In order to simplify the procedure it was tried to replace the sodium carbonate solution by ammonia liquor which is a by-product of the coal carbonization. A small apparatus equipped with an initial ammonia extraction has been operating for 10 days. According to the determined gas values no deterioration of the Alkazid-solution could be observed. Without a pretreatment the gas value decreased for about 5 vol. per vol. every day, whereas the current test shows not even a decrease of 0.1 vol. per vol. since the beginning of the experiment.

The commercial application of the process does not require any additional costs because the ammonia liquor can be fed to the already existing condensers. It is now possible to apply the Alkazid process for the purification of coke oven gas. In this connection it is worth mentioning that all purification processes which employ alkalies such as the Girdler-Seaboard- and Thylix-process encountered the same difficulties which led to a deterioration of the extracting solution. Because of the importance of the pre-scrubbing process for the applicability of the Alkazid process and others, a patent application has been filed.

October 9, 1933 the Leuna commercial plant after 10 weeks of operation showed heavy corrosion at the bottom of the boiler and inside the hot pipes. The bottom and some pipes were replaced whereafter the plant was again put into operation. Unfortunately a newly installed pipe was completely destroyed by corrosion after 15 hours operation. Corrosion tests proved that the circulating solution was very corrosive against aluminum. The reason was that the solution had been diluted with water resulting in a specific gravity of 1.10 instead of the desired 1.13. A diluted Alkazid-solution is corrosive in itself. In addition a diluted solution is better stripped than a concentrated one resulting in a higher alkalinity. As a result a serious danger arises for the aluminum which has been employed as a construction material for the apparatus. For the moment it seems to be remarkable that the solution showed such a corrosivity after a 5-weeks operation with the diluted solution. But the apparatus was coated with a thick protecting layer which resisted the attack of the diluted solution. At such places however where an excessive mechanical strain occurred, at the bottom of the boiler on which the solution was dripping or at the elbows of the pipes and pumps, the protecting coating was slowly eaten away and could not be renewed by the diluted solution. Consequently such places showed the first corrosions.

According to the results of the corrosion tests such Alkazid solutions which have been more effectively stripped in the pressure-stripper showed a higher tendency to be corrosive. Silumin as well as V₂A are completely resistant against the solutions of higher alkalinity. Aluminum becomes fully resistant to corrosion as soon as it has been heated to 600°C. But due to mechanical deformation such a method cannot be commercially adapted. As a precaution the lower part of the pressure stripper was brick lined and some samples consisting of silumin, aluminum heated and not heated, and V₂A were fixed into the hot discharge line in order to be able to determine the resistance of the various metals. The Alkazid plant was completely dismantled and a following inspection showed no further corrosions.

The corrosivity of Solutions of Sodium-oxy-ethylglycocall

The investigation of the oxy-ethyl-glycocall-solution which was continuously used for 2 months resulted in the fact that the compound had been partly decomposed with the formation of ethanclamine ($\text{NH}_2\text{-CH}_2\text{-CH}_2\text{-CH}$). It had already been experienced that ethanclamine is very corrosive against aluminum. It can therefore not be used as "Alkazid-solution". But even without the observed corrosivity, the oxy-ethyl-glycocall would have been without interest for the H_2S -extraction because its absorptiveness is inferior to that of alanine and it is more expensive than the latter. The problem of the purification of the coke oven gas by means of the Alkazid-process is solved in so far that by pretreating, the destructive cyanogen can be eliminated. Applying a low pressure stripping process the question of the constructing material to be employed is solved. In order to improve the economy of the process the application of a pressure stripper is considered. Experiments with reference to the durability of the material and the solution are under way.

Item 22c

Inspection of the Alkazid-pilot-plant Deuben and Discussion of the Boshlen Project

The pilot plant Deuben was shown to the representative of the government and of the A.S.W. By means of gas analysis it was demonstrated that the S-content of the gas was lowered from 30 to 1.2g per cu. m. No figures were given with reference to gas output, volume of the recirculated solution, and steam consumption.

Upon questioning it was explained that at first S- and later on DiK-solution was used during the 2 months operation. It was further explained that the nature of the destructive constituents of the gas are still unknown, that they increase the acidity of the solution and that a pretreatment of the gases to be extracted is provided for. With reference to the economy of the process it was mentioned that the manufacturing costs of the sulfur would be higher than they used to be.

The following figures for the energy consumption etc. were submitted:

Gas volume to be treated: 30,000 m³/h (735.5 mm Hg, 15°C)
The gas contains 40 g H₂S/m³ and 20-25% CO₂
H₂S content of the finished gas: 1g/m³

1. 20 tons (metric) per hour low pressure steam.
 2. 70 kwh per hour
 3. 140 m³/h fresh water
 4. 630 m³/h recirculating cooled water
 5. Consumption of the Alkazid solution RM26 .-per hour
 6. Operating staff: 4 workers, 1 fitter
 7. Sulfur production 1 metric ton per hour.
- The rest of the report is without technical interest.

Development of the Alkazid Plant 282 in 1937

In the early days of 1937 the total volume of the hydrogenation gas was jointly desulphurized in the available apparatus (comp. picture 1). During 1937 the plant was greatly enlarged, the hydrogenation gases were separated and the Winkler-O-gases were desulphurized by means of bubble-cap-scrubbers. Picture 2 represents the situation at the end of the year.

1. The hydrogenation-return-gases were divided into 3 streams:
 - a. Hydrogenation-rich-gas for Linde liquefaction and acetylene factor! Removal of H₂S to 0.5 g/m³ and CO₂ to 0.03%.
 - b. Hydrogenation-rich-gas for compressed power gas H₂S extraction to 4-5 g/m³.
 - c. Hydrogenation-lean-gas for fuel gas
2. Winkler-O-Watergas

Sulfur extraction by means of bubble-cap-scrubbers to approx. 1g/m³ H₂S.

In addition to many small-scale-tests with Winkler-)gas, coke-gas, Pattenhausengas, the following full-scale experiments were carried out in commercial plants:

1. Concentration of the H₂S liberated from the Winkler-waste-water.
2. Extended purification of Winkler-O-gas.

The program for 1938 comprises the following items:

1. Rich-gas-extraction-plant working under 2-3 atm.
2. Enlargement of the Winkler-O-gas pre-desulfurization for 70,000 m³/hr.
3. Construction of a commercial plant for the final purification of Winkler-O-gas.
4. Replacement of the packed towers by bubble-cap-scrubbers for the desulphurization of hydrogenation-lean-gas.

Picture 3 shows the situation to be expected at the end of 1938. Additional experiments will be carried out with the air to utilize the vapors which leave the stripper and to heat the stripper by means of gas.

Item 22e

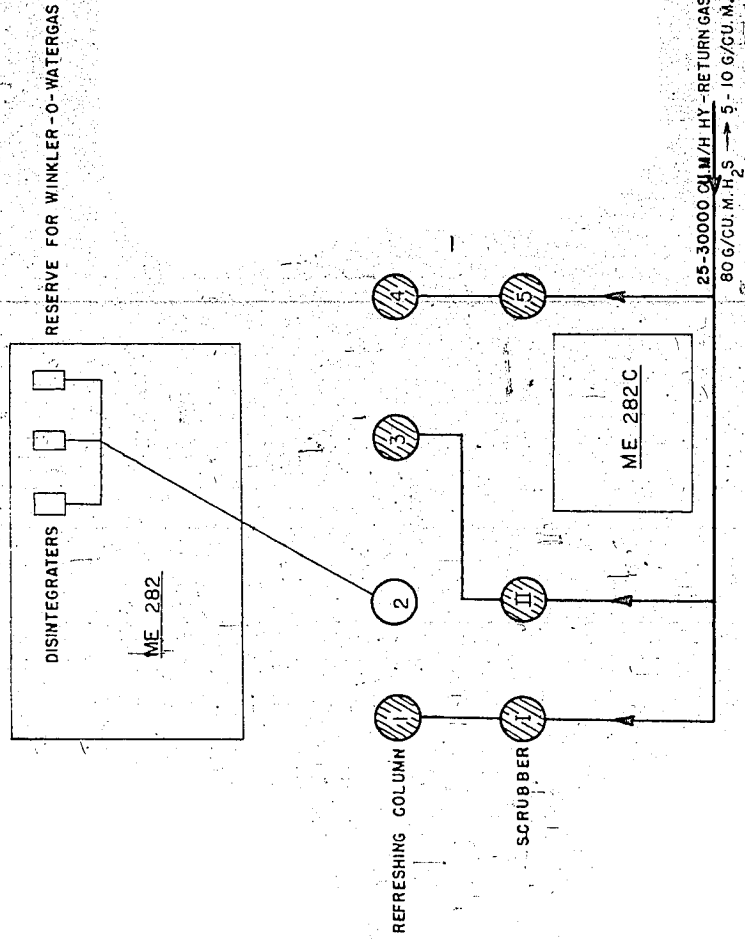
Ref: Priority of the stripping system

A) Capacity of the Alkazid-plant

The capacity of the Alkazid plant prior to May 12 was:

	Normal operation	Peak load
Hydrogenation-rich-gas	20,000 m ³ /hr with 14% H ₂ S and 24% CO ₂	22,000 m ³ /hr with 16.5 H ₂ S and 24% CO ₂
Hydrogenation-lean-gas	32,000 m ³ /hr with 32 g/m ³ S	37,500 m ³ /hr with 35 g/m ³ S
Winkler-O-water-gas First stage	60,000 m ³ /hr with 13 g.	70,000 m ³ with 15 g.
Winkler-O-water-gas Final stage	40,000 m ³ /hr roughly desulphurized	
Winkler-O-waste-water gasification (for concentrating purposed)	1,300 m ³ /hr containing 4% H ₂ S	2,500 m ³ /hr containing 4% H ₂ S

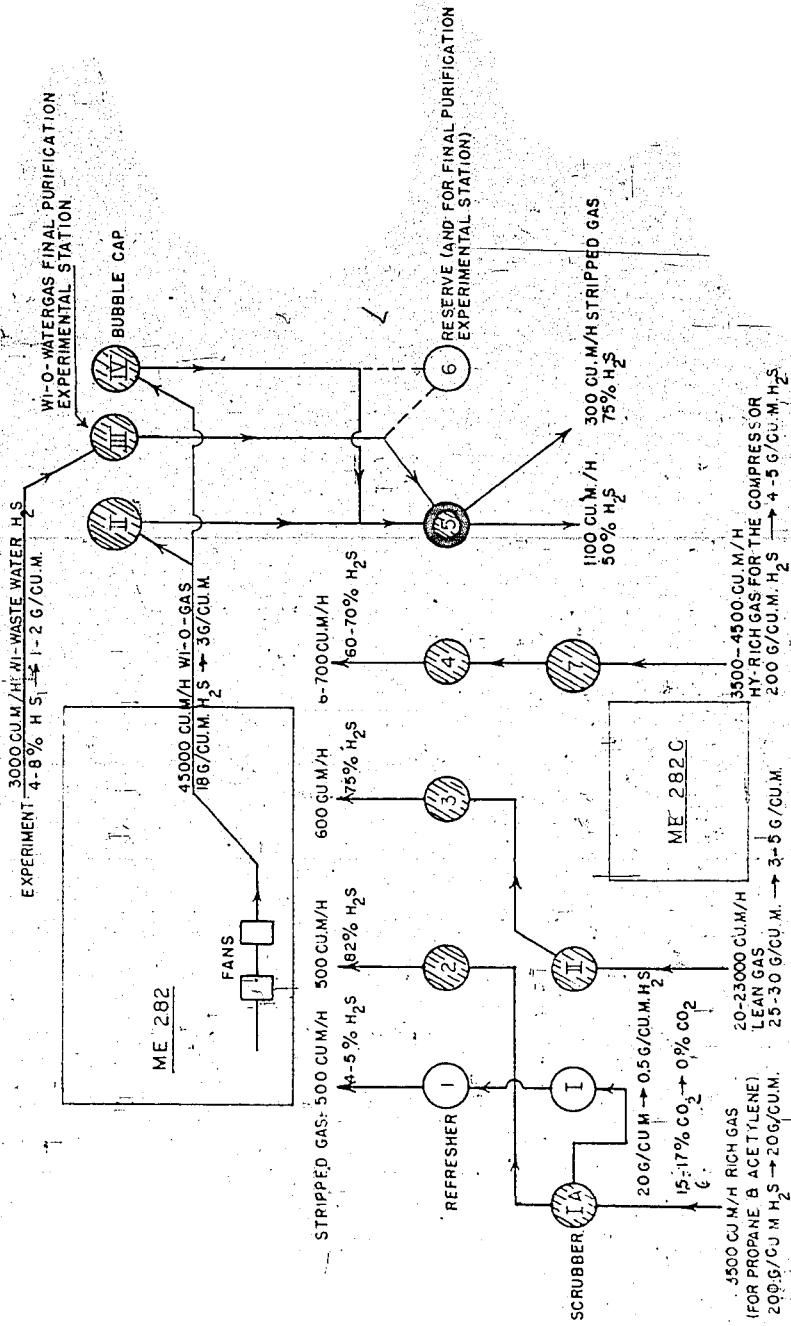
PICTURE 1
ALKAZID PLANT ME 282X SITUATION IN THE EARLY DAYS OF 1937



PICTURE 2

ALKAZID PLANT ME 282.X

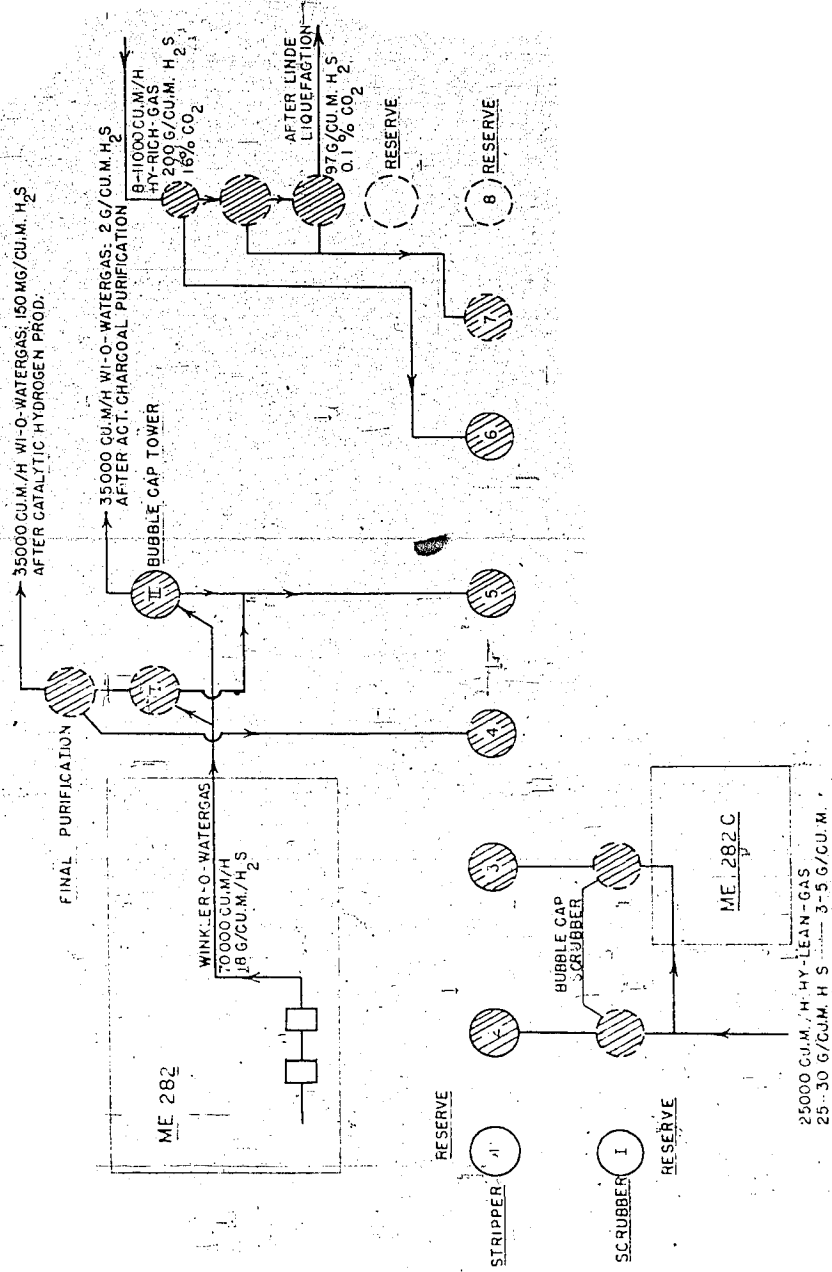
SITUATION IN THE MIDST OF DECEMBER 1937



PICTURE 3

ALKAZID PLANT ME 282 X

SITUATION AT THE END OF 1938



	Normal Operation	Peak Load
Coke-oven-gas-H ₂ S after pre-purification to claus-kiln	800 m ³ /hr. containing 30% H ₂ S	1,000 m ³ /hr contain- ing 35% H ₂ S
Phenol-rich-H ₂ S after pre-purification to claus-kiln	700 m ³ with 30% H ₂ S	1,000 m ³ with 35% H ₂ S
Phenol-lean-H ₂ S to concentration system	4,000 m ³ /hr containing 4% H ₂ S	

In addition the stripped gases which are obtained by the CO₂-removal from hydrogenation-rich-gas and by the final purification of the Winkler-O-gas and which contain comparatively small amounts of H₂S must be concentrated so that they are fit for the claus process.

Due to severe destructions of the final purification of the Winkler-O-gas desulphurization system it was necessary to transfer the final-desulphurization of the Winkler-O-gas to the active char coal purification for a considerable period of time with the result that the normal rotating procedure of the reactors could not be maintained. In addition due to the repairs which were caused by the air raid the dust content of the gases to be purified was extremely high resulting in a considerable decrease in the efficiency of the active-char-coal-plant. Formerly the final purification of the Winkler-O-gas was partly carried out by the active charcoal plant. (comp. fig. 6) With the compressors running at peak load the installations 40 and 235 are almost overburdened so that they cannot perform the final purifications of the Winkler-O-gas to be desulphurized. An improvement of the present situation is possible only if the capacity of the Alkacid plant will be increased by the construction of the stripper 10. A decrease of the Winkler-O-gas-final desulphurization means a simultaneous reduction of the hydrogenation capacity. The gas volume which can be desulphurized by the Alkacid plant depends largely on the sulfur content of the gas, the latter depending on the type of fuel to be gasified. The cruce gas which has been obtained by the gasification of Deuben char contains 8-11 g S/m³ whereas Esphenhaier char furnishes a gas with 15-16 g S/m³ and TBK 17 one with up to 22 g S/m³. During the necessary repair of one of the two strippers which are connected with the Winkler-O-water gas desulphurization system, the entire final purification system must be shut down because the other existing strippers cannot be employed (See below). Such a shut down means a reduction of 1,500 revelations.

With the construction and operation of the new stripping system 10 the capacity of the Winkler-O-gas desulphurization is increased to 70,000 cu. m. per hour. The above mentioned improvements do not only prevent such reductions during repair periods but even a necessary shut down of the active char coal plant can be compensated. Present situation and situation to be expected:

Gasified type of coal	Present Situation Pro-purification m ³ /hr	Situation with operation final purif. of stripper 10 m ³ /hr.
Deuben char	70,000	70,000
Esphenhaier	70,000	70,000
TBK	30,000	70,000
Repair of the stripper	70,000	0
		45,000

FIGURE 1

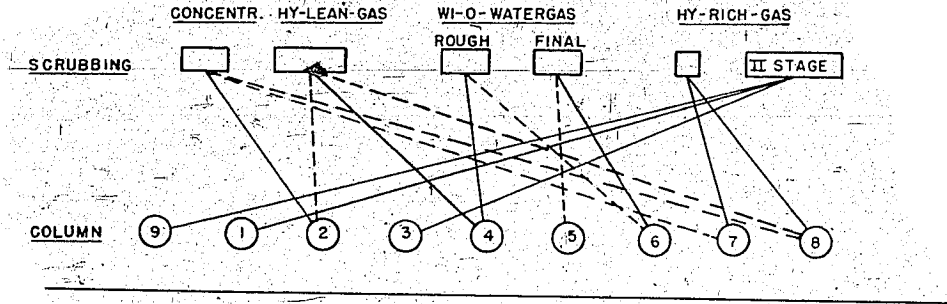
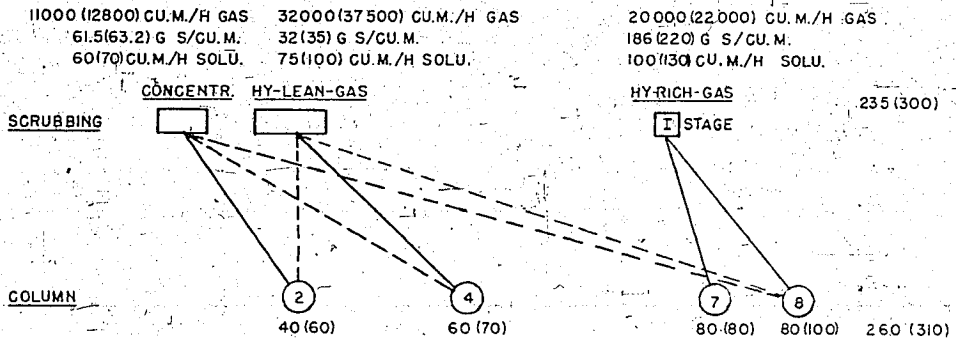
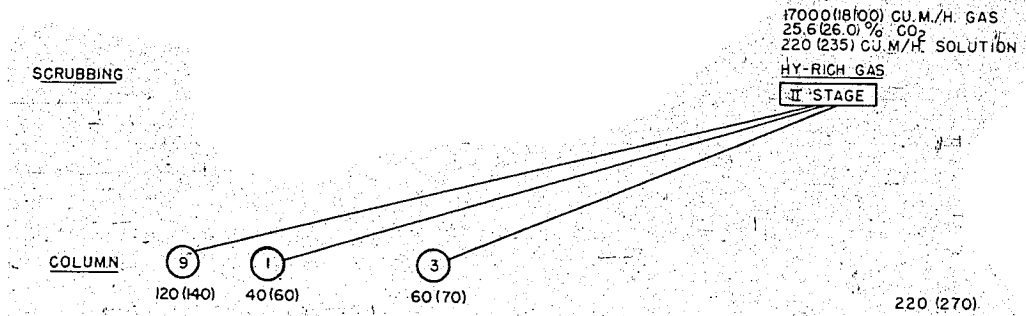


FIGURE 2



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FIGURE 4



WINKLER-O-WATERGAS
60000 (70000)

FIGURE 5

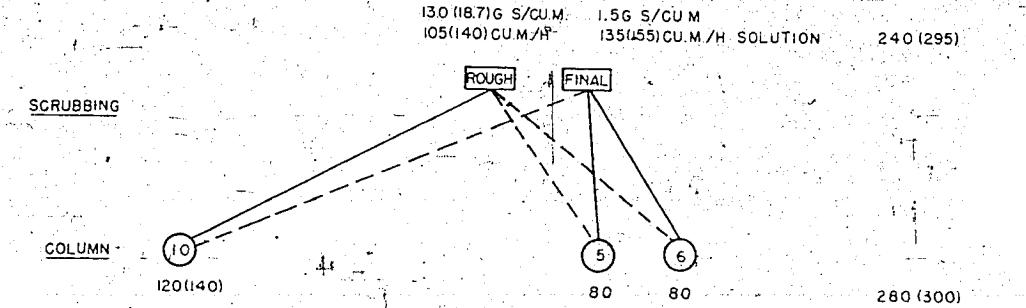
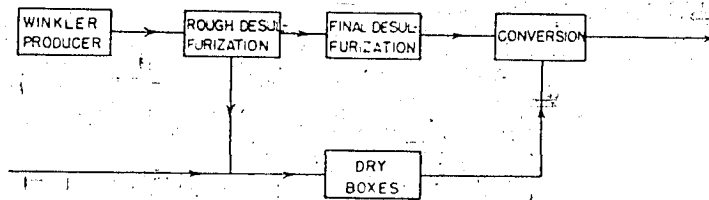


FIGURE 6



B. Distribution of the strippers

The strippers can be connected with different scrubbing systems for reasons of their respective capacity and for reasons of the historical development of the plant. Three groups must be distinguished, the Alkacid solutions of which must be kept separated, in order to maintain a high extracting efficiency.

The following groups must be distinguished:

1. Rich gas-desulphurization, lean gas desulphurization concentration (blue cycle)
 2. Winkler-O-gas desulphurization (green cycle)
 3. CO₂-extraction from rich gas (red cycle)
- (Compare the figures 3,4, and 5)

Above the scrubbing systems the normal capacities (peak loads) with respect to volume and sulfur content are recorded. In addition the necessary volume of the recirculating solution is marked. Below the strippers their normal capacity and peak load is specified. On the right side the sums of the required and available volumes of the solutions are compared. It is obvious that in the groups 1 and 3 the required volumes are covered by the respective capacities and that by proper operation of the plant repairs can be made without affecting the output. Group 2 however shows very clearly that the capacity of the columns is much too small in order to be able to furnish the necessary quantity of refreshed solution, in addition the final purification must be completely shut down should it become necessary to repair a column. With column 10 constructed and operated a satisfactory correspondence between the required and furnished volumes is secured as shown by picture #5.

Item 22f and g

Sulfur Scrubber Combined with the Claus-kiln Luetzkendorf

We were informed by the Leuna-people that experiments are being carried out to replace the electric precipitator which ordinarily extracts the elementary sulfur from the gases leaving the claus kiln by a scrubber which is operated by recirculating liquid sulfur. Since the Doehlen electric precipitator is overburdened, the Luetzkendorf method is of great interest.

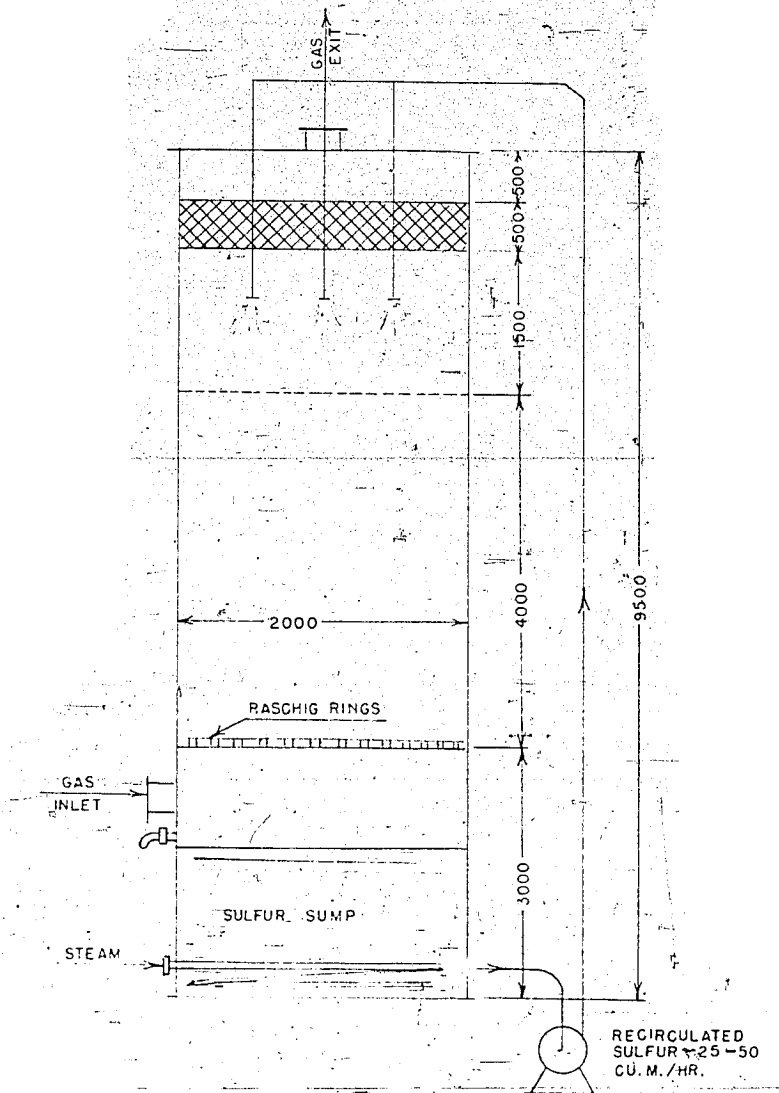
Description of the experimental system:

The scrubber is 9,500 mm high and 2,000 mm wide and packed with a 400 mm thick layer of Raschig rings. The scrubber is brick-work lined. A pump (capacity 25-30 cu. m.) recirculates liquid sulfur at a temperature of 125-140°C. The distribution of the liquid sulfur is carried out by means of a ring shaped line with baffle sheets. The scrubber is equipped with a burner which however was never operated. The output of the claus kiln is 1,200 cu. m. per hour gas to be converted. The H₂S concentration of the gas is 60%. The sum of the scrubber is 3,000 mm high.

Accordingly to obtained information the system operated very satisfactorily. The sulfur is almost completely removed from the gas.

Analyses:
 Scrubber entrance 60 g/m³S
 " exit 4-6 g/m³S

Under normal conditions the heat loss of the scrubber is so high, that temperatures of 125-140°C. can be maintained. Experiments were made to cool the sulfur by means of coils, but without success. It was proposed to inject water into the recycling sulfur for cooling purposes. Experiments have not yet been made.



SULFUR SCRUBBING
ITEM 22.g

The question whether the scrubber should be lined with brick work was discussed. It was mentioned that it is not necessary and that corrosion is not to be expected. It is necessary to insulate the feed pipe which connects the pump with the inlet of the scrubber. The stuffing gland of the pump must be heated but it is not necessary to heat the casing of the pump. The head of the pump must be 20 m water column. With respect to the capacity of the motor it must be born in mind that the spec. gravity of the liquid sulfur is as high as 1.98.

Item 22g

Sketch of the sulfur scrubber described in 22f. T

Item 22h

Proposal for Delivery of an Alkazid-plant

We submit to you a detailed offer which contains weight and prices of an Alkazid plant according to German conditions.

Output of the plant	5900 m ³ /hr	(15°C., 735 mm Hg.)
H ₂ S content of gas	5%	
H ₂ S content of the purified gas	0.35 g/m ³	
Actual pressure of the gas	7 atm.	
Actual temp. of the gas	32-38°C.	

Gas analysis:

1. H₂S-- 5%
2. CH₄-- 4%
3. C₂H₆-- 22%
4. C₃H₆-- 14%
5. C₃H₈-- 50%
6. C₄H₁₀-- 5%
7. CO₂-- traces

Steam pressure	5.6 atm.
Properties of the steam	Saturated
Cooling water	27°C.
Pressure of the water	0.7 atm.

Description of the plant:

The gas enters a bubble cap scrubber (item 1) under a pressure of 7 atm. The scrubber is equipped with very efficient humbles which secure an intimate contact of the gas with the Alkazid solution. The purified gas leaves the scrubber and passes through a cyclone which separated the drops which are carried away from the gas.

The extracting solution is fed to the top of the washer and leaves at its bottom enriched with H₂S. The pressure of the solution is released by means of a let down valve (item 13), passes through a heat exchanger (item 8) and enters the stripper (item 5). During its passage through the heat exchanger the cold saturated solution is preheated to a suitable temperature by the stripped solution which leaves the stripper. The heat exchanger is of a special design which due to its construction requires but a small heat transmitting area. It is made of "Silumin".

The preheated warm solution is stripped from the H_2S partly during its passage through the stripper (item 3) (atmospheric pressure) by means of live steam partly during recycling through the tube system of an evaporator which is indirectly heated by steam (item 4).

The stripper is packed with ceramic rings. It contains devices which secure a uniform distribution of the solution over the entire cross sectional area of the stripper. It is made of steel and brick lined. The evaporator is made of "Silumin" (item 4). By a catchpot (item 5) it is protected against any damage caused by particles of the ceramic rings which are carried away by the solution.

The hot stripped solution is pressed through the already mentioned heat exchanger (item 8) and cooled by means of a "Silumin" pump (item 7) which is protected by a catchpot also. The final cooling is performed in a cooling-coil-system (item 9) by means of cold water. The cooled solution is recirculated to the scrubber. The stripped H_2S together with water vapor leaves at the top of the stripper.

The vapors pass through a cyclone (item 10) which separates particles of the solution from the gases. The vapor mixture is led to a tube and shell condenser made of wrought iron (item 11). The water vapors are condensed and the H_2S is cooled. Should no utilization exist for the H_2S the condenser may be left out. Item 12 represents two storage tanks, one for fresh solutions and the other for already used liquids. Because we are not familiar with the properties of your cooling water we provided all apparatus (items 9 and 11) as well as the water carrying pipes to be made of steel.

ESTIMATE

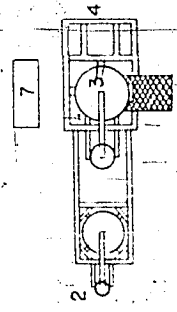
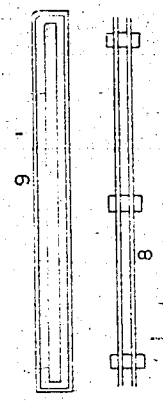
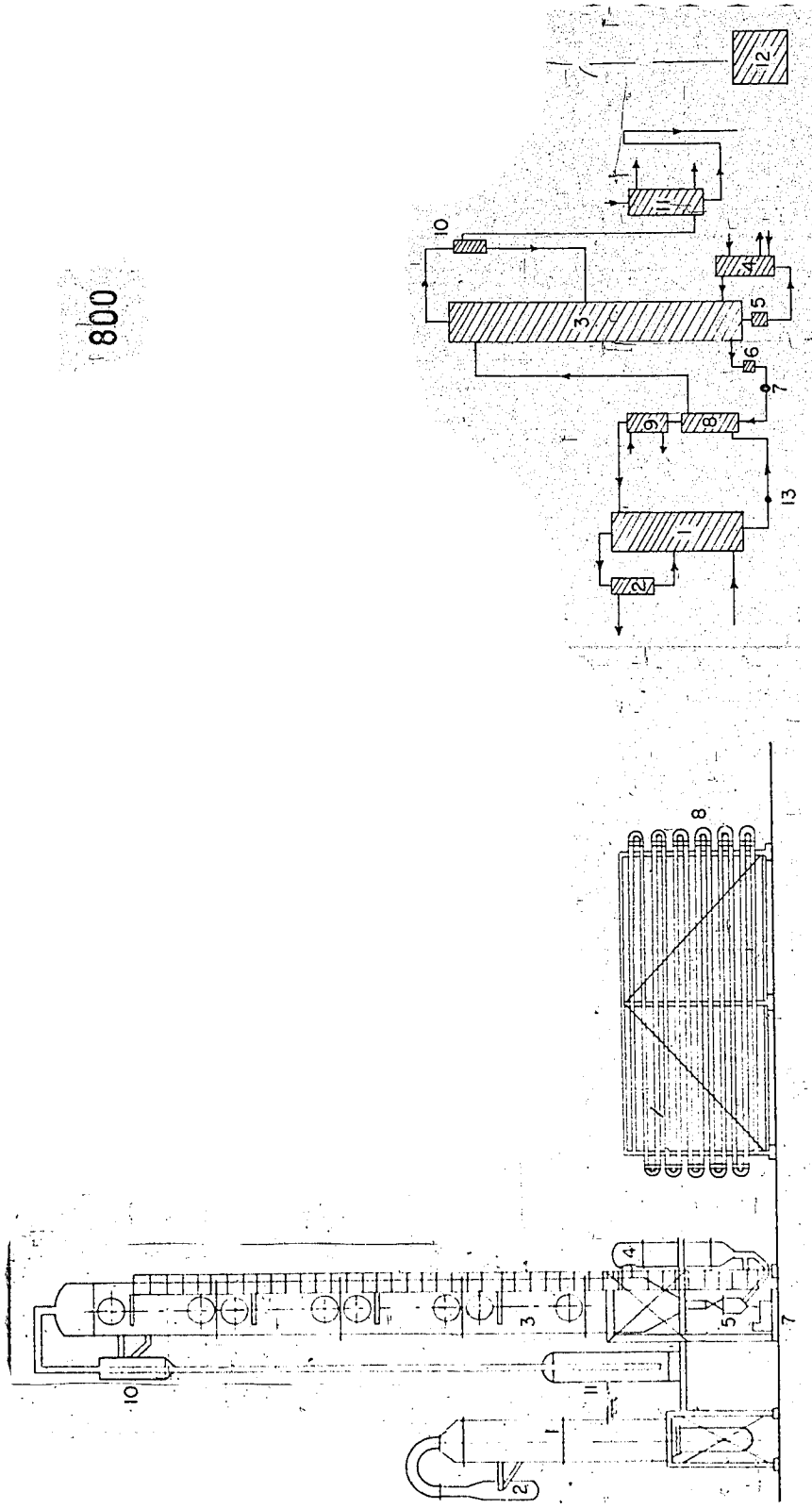
The plant comprises the following items:

- | | | |
|--------|--|------------|
| Item 1 | 1 scrubber, inside diameter 900 mm, height 5,200 mm. electrically welded, containing 12 bubble cap trays and a sump welded to the bottom of the scrubber, inside diameter 500 mm, height 1,500 mm, walls and drum-ends of boiler plate thickness of the walls 7 mm, inside devices of ingot-iron. Operating pressure: 7 atm, weight: 280 kg. | RM 12,400. |
| Item 2 | 1 cyclone electrically welded, walls and drum ends of boiler plate, thickness of the wall 6 mm, inside devices of ingot-iron. Operating pressure 7 atm; weight 280 kg. | RM 280. |
| Item 3 | 1 stripper, inside diameter 1,200 mm, height 14,000 mm. electrically welded, consisting of a conical lower part, 4 cylindrical sections and an upper part with a vaulted cover, with 4 grates, 4 distribution trays. Each section has man-holes for packing and unpacking the ceramic rings. Walls and cover of boiler plate, thickness 7 mm. The conical lower part and the 4 cylindrical sections are lined with 35mm thick tapered bricks and fastened with a special kind of putty. The grates distributing trays, as well as all other inside devices, are of pure aluminum or aluminum alloys. Operating pressure 0.5 atm. total weight 6,850 kg. Weight of the aluminum 350 kg. | RM 9,200 |
| | 10 cu. m. ceramic rings 25 x 25 x 3mm | RM 1,200 |
| | 50 m ² lining with 35 mm thick bricks | RM 2,500 |

Item 4	1 evaporator, 10 m ² heating surface suitable to be operated with indirect steam 5.6 atm saturated steam, direct steam 0.5 atm saturated steam, shell, tubes and tube sheets of "Silumin", steam injector for direct steam of V ₃ A. Weight 400 kg.	RM 1,550 RM 17,130
Item 5	1 catchpot 0.05 m ³ situated between stripper and evaporator of pure aluminum, thickness of the wall 6 mm. weight 75 kg.	RM 290.-
Item 6	1 catchpot, 0.03 m ³ situated between stripper and pump of pure aluminum. Weight 60 kg.	RM 230.-
Item 7	1 centrifugal pump of "Silumin", capacity 12 m ³ /hr. solution, head 120 m water column. Motor with switch	RM 1,500. RM 700.-
Item 8	1 heat exchanger, 70 m ² heating surface consisting of 24 heating elements in series, of silumin.	RM 8,800
Item 9	1 cooling coil, 40 m ² cooling surface of ingot-steel	RM 3,000
Item 10	1 cyclone, 0.3 m ³ of pure aluminum. Weight 60 kg.	RM 240.
Item 11	1 condenser, 40 m ² cooling surface shell and tube sheets of boiler plate tubing of ingot steel. Weight 2,500 kg.	RM 2,500
Item 12	2 storage tanks, 5 m ³ capacity each inside diameter 1,800mm height 2,000 mm of ingot steel. Weight 2,000 kg. Supports, platforms for scrubber, stripper and condenser Weight 8,000 kg.	RM 1,600 RM 4,000
Item 13	Pipe-system gas pipes of ingot iron solution-pipes of pure aluminum cooling-water-pipes of ingot-steel	RM 6,000
Item 14	Gate valves, valves and liquor level indicators	RM 14,000
Item 15	Insulation of the stripper, heat exchanger and the pipes which carry the hot solution Mounting	RM 7,000 RM 9,000
Total sum		RM 75,990

No costs for the necessary drawings to be furnished are included. No spare parts are included, we recommend to install a second pump item 7. The offer does not contain all pipes which carry and discharge gas, electric current, H₂S and steam to and from the plant. No provisions are made for foundations, preparations of the area, houses roofing, measuring instruments and the electrical installation.

800



G. C. E. 7/17/47

ALKAZID PLANT EQUIPMENT

Approximately 5 m³ DiK-solution are required to start the plant. Price in U.S.A. RM 2.4- per liter or 36.30 per 100 lbs. including freights and customs.

Guarantees

The gas to be treated must be free from HCN, O₂, SO₂ and other non-volatile acids. The energy consumption is as follows:

Steam consumption	1.2 tons per hour
Cooling water	30 m ³ per hour
Electric current	12 kw

The volume of the solution which is carried away by the gas does not exceed 2.4 cu. m. per year.

The H₂S-content of the treated gas is 0.35 g/m³ H₂S.

Item 22i

Correspondence and Report Concerning the Development of Small Hydrogen Plants by General Electric Co. and I.G. Farben

Ref: Hydrogen catalyst 633

With regard to the Williams patent application which covers the conversion of hydrocarbons by means of catalyst 410, Standard carried out experiments with the recently developed hydrogen catalyst 633. The attached memorandum shows the results of the experiments.

No experiments have been made at Baton Rouge for the conversion of propane and butane with the catalyst 633, but they will be performed in the near future.

Since Standard is of the opinion that the claims of the Williams patent are not justified, the catalyst 410 shall be employed at the Bayway- and Baton Rouge- plants. But we agreed not to sell the catalyst 410 for other purposes (hydrogenation of fats) and hope that catalyst 633 can be used.

Conferences held with the General Electric Co. concerning Hydrogen

The General Electric Co. has developed an apparatus by means of which butane can be cracked in the presence of water vapor whereby electrical heating is applied.

The apparatus is supposed to furnish but small volumes of hydrogen, 2,000 cu. ft. H₂/hr. and is intended to replace the compressed hydrogen which is employed for the production of stainless steels, aluminum alloys, etc.

Butane mixed with the theoretically necessary steam volume, is converted to a gas of the following composition applying temperatures of 1,100°C:

70.5%	H ₂
28.2%	CO
0.5%	CO ₂
0.3%	CH ₄
0.5%	N ₂

The gas is further converted using iron oxide as a catalyst and adding 2-4 vol. water vapor per vol. gas by passing through 2 furnaces (1st furnace 430°C. followed by a H₂S extraction by means of sodium hydroxide, 2nd furnace 300°C.) into a gas of the following composition

79%	H ₂
20%	CO ₂
0.2-0.3%	CO

The CO₂ has not yet been extracted but the Girdler Co. furnished proposals and drawings for a CO₂-extraction.

Since Standard is interested in furnishing butane, it has watched the G.E. experiments and furnished suitable catalysts. In a conference held between G.E., Standard and us, we proposed to apply the recently developed Alkazid solution M for CO₂ extraction instead of Girbotol or Dapol respectively because Alkazid shows some advantages and does not require a special apparatus for its recovery as is necessary if Dapol is utilized. Furthermore we proposed to apply a CO-converting catalyst of I.G. Farbon as for instance the re-activated catalyst according to U.S. patent 1,115,776. Instead of 2 reactors with a sodium hydroxide extraction arranged between them, we proposed the application of only 1 furnace which is equipped with 2 catalyst beds, whereby the temperature of the second catalyst bed can be kept lower by the introduction of steam should the conversion of the above mentioned gas to 0.2% CO by only one catalyst bed require too much steam or be impossible. The low CO-content of the gas is desired with respect to the C-content of the steels. Whether the conversion to 1-1.5% CO followed by a hydrogenation of the CO to CH₄ is permissible from the standpoint of the consumer is not yet known.

Should the I.G. catalyst be applied, probably the H₂S extraction is not necessary because the butane contains but 1 grain/100 cu. ft. S. The future development is supposed to be as follows:

G.E. constructs the apparatus ready for use and sells them to Standard. The price is supposed to be \$5,000 which includes a reasonable profit.

Standard rents the apparatus, maintains them in a good working condition and supervises them in a reasonable interval of time. Due to the electrical heating system and a far reaching mechanical operation of the plant, and the application of automatically controlled valves the apparatus is almost foolproof. By a market analysis Standard came to the conclusion that approx. 30 customers which now use compressed hydrogen are willing to apply the new process. According to information obtained from Standard the price of the compressed hydrogen ranges between \$6.50 and \$22.50 per 1,000 cu.ft. Lower production costs may be expected if larger volumes of hydrogen are consumed. Producing 1,000,000 cu. ft. of hydrogen per day and applying the Messerschmidt-process (iron-steam) the price is supposed to be \$0.90-\$1.30 per 1,000 cu.ft., whereas the electrolytic hydrogen costs at least \$1.50 per 1,000 cu.ft.

The production costs of the hydrogen applying the butane-conversion-apparatus of G.E. are estimated to be \$1.- per 1,000 cu.ft. amortization, repairs, royalties not included. It is intended to sell the hydrogen for \$4.- per 1,000 cu.ft. to the consumer. Necessary amortization is supposed to amount to \$1,000 per year per apparatus and \$500.- per year are consumed for maintenance and supervision.

With an average daily production of 10,000 cu.ft. per apparatus (5 hours operating time) the following calculation can be set up:

1. Production costs	\$1.- per 1,000 cu.ft.	=	\$10.- per day
2. Rent (a) amortization	\$1,000.- per year		
(b) maintenance	\$500.- per year		
	\$1,500.- per year	=	\$5.- per day
			\$15.- per day
Revenue	\$4.- per 1,000 cu.ft.		\$40.- per day
			<hr/>
Profit			\$25.- per day

The profit must be distributed between Standard and I.G. whereby Standard must bear the financial risk, the advertising and the sale. We intend to propose that I.G. obtains 33 1/3 % whereas Standard was mentioning 20%.

Under the above mentioned conditions I.G. would receive per year:

	33 1/3 %	20 %
per apparatus $\frac{300 \times 25}{3} =$	$\$2,500.-$ per year	$\frac{300 \times 25}{5} =$
		$\$1,500$ per year
per 30 apparatus	$\$75,000.-$ per year	$\$45,000.-$ per year

Even with a 20% profit I.G. would receive a considerable revenue without any risk.

We informed Standard that I.G. is willing to place at disposal the CO-catalyst and the Alkazid-process. We ask you to endorse our proposal.

We ask you to ship as soon as possible to U.S.A. such a volume of concentrated Alkazid which allows to produce 100 liters 30% solutions. In addition we ask you to provide the following technical information:

1) CO-conversion

gas volume to be converted 2,000 cu.ft. per hour 100%
composition of the gas to be converted:

70.5%	H ₂
28.2%	CO
0.5%	CO ₂
0.3%	CH ₄
0.5%	N ₂

~~Information concerning the operating conditions (vol. of steam, temp., quantity of the catalyst) so that the discharged gas does not contain more than 0.2% CO, as well as a proposal concerning the design and size of the reactor, durability and price of the catalyst.~~

2) Alkazid-scrubbing

Gas volume: 2,000 cu.ft. per hour hydrogen 100%
Composition of the gas: 79% H₂
20% CO₂
remainder CO, CH₄, N₂

The discharged gas is supposed to contain not more than 0.1% CO₂. Proposal with reference to the apparatus (design, size, material of construction, iron and aluminum, operating conditions, steam and power consumption, losses, flow sheet). We proposed a scrubber with a 10 ft. depth raschig ring packing (3" rings). The inside diameter of the scrubber is 14". It is desirable that the scrubber is not higher than 12 ft.

Office Memorandum on Investigation of New Batch of Catalyst 633 for Production of Hydrogen from Natural Gas

Introduction

Previous test work at Baton Rouge on this catalyst had shown excellent catalytic activity but poor mechanical strength. Originally, this catalyst had arrived from the I.G. in the form of 10 mm pills, which, because of pressure drop consideration, were ground and re-pilled to a 20 mm size. The subsequent failure of these pills in the

test-work brought up the question whether the re-pilling operation itself was responsible. To investigate this possibility, a new batch of catalyst 20 mm in size was ordered from I.G. This catalyst was then tested out in the 20-foot experimental tube at Baton Rouge.

Summary

The 20 mm pilled I.G. catalyst 633 was tested out for activity on natural gas containing 90% CH₄, 5% C₂H₆ and 5% N₂; the excess steam used was 100% (two volumes steam per volume gas). The activity at various throughputs and temperatures is summarized below, and compared with former results on Catalyst G-410:

Space Velocity	Temperature °F.	% Residual CH ₄	
		on Cat. 633	On Cat. G-410
615	1400	0.4	1.5
615	1300	1.1	3.5
1080	1500	0.5	1.5

The new I.G. catalyst is definitely more active on natural gas than G-410. It seems to take an operating temperature about 100°F. lower than for G-410 for the same activity.

The strength of this batch of catalyst is immeasurably better than that of the previous batch, but is still not satisfactory. Over 21 days' operation the catalyst showed the following losses:

Catalyst originally installed, lbs.	323
Catalyst removed at end of run, lbs.	300
% loss in weight	7.1
Dust removed, lbs.	48
Good catalyst recovered, lbs.	252
% dust (on catalyst removed)	16
Shrinkage, inches	8
% shrinkage per day (21 days)	0.15

The appearance of the so-called "good" pills recovered was not very satisfactory. They seemed subject to easy dusting and had lost considerable strength.

Based on these results it seems very desirable to continue the work at Baton Rouge on the addition of a binding agent during the manufacturing process.

Item 22 j

Alkacid-desulphurization-plant Luetzkendorf

Correspondence concerning the operation of the Luetzkendorf-Alkacid-plant under the supervision of I.G. (Leuna)-engineers.

Apparently the Alkacid-plant was not in compliance with the giver guarantees and Leuna was asked to operate the plant by their experts.

Luetzkendorf must fulfill the following conditions:

- 1) Technically pure steam at a constant pressure of 2 atm with not more than 5 mg. residue per liter of condensate.
 - 2) Suitable cooling water
 - 3) Filter cloths
 - 4) Material for repair and improvement
 - 5) Personnel required for repair and operation
- Leunais responsible for the H_2S -extraction, losses of the solution, corrosion, etc.

Item k

Alkazid-plant Luetzkendorf

Requirements for cooling water:

According to obtained information the following volume of water is available.

- 1) Water from the cooling tower with a maximum temperature of $32^{\circ}C$. A temperature increase of 10° is admissible.
- 2) Water from the Saale river with a maximum temp. of $16^{\circ}C$.
- 3) Processed water (Permatit) with a maximum temp. of $16^{\circ}C$. can be heated as high as possible because no boiler scale is to be expected.

Water consumption depending on the output of the plant:

- 1) 20,000 $m^3/hr.$
- 2) 35,000 " "
- 3) 50,000 " "

H_2S -extraction from 23 $g/m^3 H_2S$ to 150 $mg/m^3 H_2S$

Water consumption in $m^3/hr.$	1	2	3
Water from the cooling tower	175	310	445
Processed water	70	120	175

With processed water not available and replacement by water from the cooling tower for the final cooling of the solution and cooling of the vapors:

Water consumption in $m^3/hr.$	1	2	3
Water from the cooling tower	430	760	1,100
Water from the Saale-river	90	155	220

Additional water volumes are required for cooling the gas which is fed to the plant. Maximum inlet temp. of the gas $25^{\circ}C$.

Suppose the gas must be cooled from $80^{\circ}C$. to $25^{\circ}C$. (dew point $40^{\circ}C$ the following water volumes are required if the water is heated from $18^{\circ}C$. to $35^{\circ}C$.)

- 1) 48 $m^3/hr.$ water from the Saale river
- 2) 84 " " " " " " " "
- 3) 120 " " " " " " " "

Item 22L

Necessary Repairs and Alterations of the Alkazid-plant Lutzkendorf

1) Bubble-cap-scrubber

The trays shall be equipped with detachable caps and 2 down flow pipes which have a diameter of 300 mm because Wintershall doubts that the formation of dust deposits can be entirely prevented.

2) Heat exchanger

The heat exchangers are repaired and altered in such a manner that they can be easily drained.

3) Condensers

The condensers shall be made exchangeable.

4) Stripper

The trap of stripper 3 shall be shortened to 5 m.

Claus plant

5) The injector shall be made of stainless steel

6) Some gaspipes shall be altered

7) The multiclone must be repaired.

8) The sulfur drain must be simplified

The injection of the live steam is to be altered according to former proposals. Additional requested repairs and alterations are without significance.

Item 22 m

Description of the Alkazid Desulphurization Plant near Installation

-Me-887

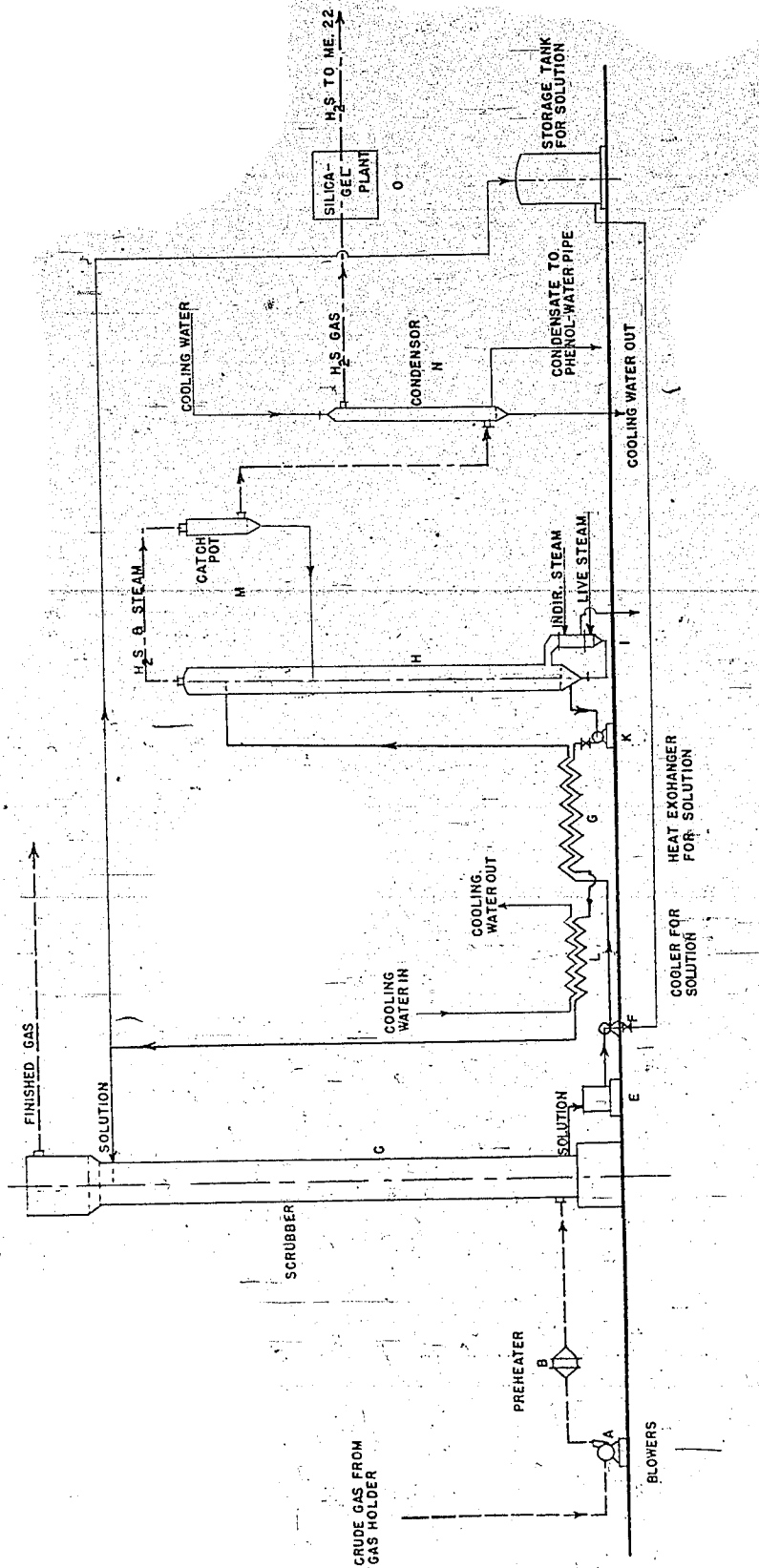
The gas to be purified is passed by a blower "a" through a heater "b", which controls the temperature into the scrubber "c", which is packed with Raschig rings. The gas is scrubbed by means of Alkazid solution which extracts the H_2S from the gas. The extracted gas leaves the scrubber fit for further use. The enriched solution is drawn off from the receiver "c" by means of pump "f" and passed through the heat exchanger "g" where it is preheated to $85^{\circ}C$. by the returning solution to the stripper "h". The solution trickles over the Raschig ring packing whereby it is heated and stripped by introduced live steam. By recycling through a reboiler "i" it is brought to a boil by live steam and indirect steam in order to remove the last traces of the extracted H_2S . The refreshed solution is pumped by a pump "k" through the above mentioned heat exchanger "g" and a cooler "l" back to the scrubber "c".

The vapor mixture consisting of H_2S and water vapor leaves the top of the stripper at a temperature of $85^{\circ}C$. and flows through the catchpot "m" to the condenser "n" where the water vapors are condensed. The H_2S gas is dried by a silica-gel-plant "o" and now is ready for use.

Item 22 n

Memorandum on Low-Pressure-Scrubbing of CO_2 from Raw Hydrogen by means of Girbotol and Diaminopropanol SolutionsIntroduction

This memorandum presents results of test work on an experimental CO_2 scrubbing system to determine the maximum capacity of a plant size



——— CRUDE AND FINISHED GAS
 - - - SOLUTION
 - - - H₂S GAS

ALKAZID - H₂S EXTRACTION PLANT - ITEM 22 m

G.C.E. 7/15/47

system using packed towers, and to compare the relative advantages of girbotol and diaminopropanol as scrubbing media.

Summary

With the use of Raschig ring packing in place of the bubble plate in the present 6 ft. diameter plant CO₂ scrubbers, the maximum capacity of each will be as follows, using girbotol as the scrubbing medium:

Size of rings	CF Scrubbed H ₂ /day	50% T.E.A. circulated GPM	Residual CO ₂ -%
3/4 inch	4,000,000	156	0.0
2 inches	8,000,000	343	0.8
3 " +)	9,300,000	400	0.0

The results when using D.A.P. in the experimental system are as follows, translated in terms of the large scale scrubber:

Size of rings	CF Scrubbed H ₂ /day	30% D.A.P. circulated GPM	Residual CO ₂ -%
2 inches	8,000,000	169	0.0
2 " "	13,000,000	300	0.0

The capacities noted above for D.A.P. solution are not maximum. Higher rates have not been obtained due to shortage of experimental stripping capacity.

When using TEA at 50% concentration, the present size stripper (6 ft. I.D.) will satisfactorily handle the amount of solution required when scrubbing about 9,000,000 CF H₂/day. When using D.A.P. at 30% concentration, the same stripper will handle the solution corresponding to 9,000,000 CF H₂/day.

+) Estimated

The relative steam consumption for T.E.A. and D.A.P. for stripping and preheating as obtained in the experimental system are as follows:

T.E.A.	50 lbs. steam / MCF raw gas to scrubber	"	"	"	"
D.A.P.	60 " " " " " "	"	"	"	"

The higher D.A.P. steam consumption is due to the greater heat of CO₂ absorption, 900 B.t.u. vs. 600 B.t.u. per pound of CO₂ absorbed.

Discussion

1. General

A flow sheet of the experimental system is shown in Fig. 1. The scrubber and actifier were designed to contain the same packed height as the large scale scrubber and actifier with bubble plates removed, so that the extrapolation from small to large scale units could readily be made on the basis of cross-sectional areas alone. The small system contains in addition a flash chamber inserted the preheater and actifier to remove part of the CO₂ before the actifier. The flashed gas, together with the gas from the top of the actifier is condensed, and the condensate returned to the system at the flash chamber.

2. Operation with Triethanolamine (T.E.A.)

As first constructed, the scrubbing tower was packed with 3/4 in. Raschig rings to a height of 35 ft., the tower being 12 in. in diameter. The actifier (same diameter) was packed with 2 in. Raschig rings to a height of 26 ft. below the feed inlet, and 6 ft. above this point.

The first scrubbing operations were made with a 50% T.E.A. solution in water. Raw gas containing 18% CO₂ (s.g.o. 365) was passed through the tower in increasing amounts, using the minimum quantity of liquor to completely remove the CO₂. At a throughput of 6000 CF/H. raw gas and a liquor rate of 370 GPH (35% T.E.A.) the tower flooded and liquor appeared in the gas trap. However, the CO₂ was completely removed. This throughput, translated into terms of one of the large 6 ft. I.D. towers is equivalent to 49000,000 CF scrubbed H₂/day.

A short run was next made circulating pure water through the system to determine whether the carry-over was due to liquid viscosity or entrainment. At 13,000 CF. raw gas and as high as 700 GPH water no carry-over was experienced. It was then concluded that the T.E.A. solution viscosity was too great for the 3/4 inch rings, and that 2 inch rings would probably be large enough to handle the equivalent throughput of 9,000,000 CF H₂/day on the large scrubber for which the small system was designed.

After changing over to 2 in. rings in the scrubber, the runs were continued, increasing the gas as rapidly as possible to reach the maximum tower capacity. In table I are given the results of these runs. The maximum gas rate obtained was 12,000 CF/Hr., equivalent to 8,000,000 CF H₂/day on one large unit. It was not possible to circulate enough solution (44% T.E.A.) to completely remove the CO₂ (because of tower flooding at increased circulation rates) but the final gas contained only 0.8% CO₂.

The effect of scrubbing temperature is shown by a comparison of runs 5 and 5A. They were made under similar conditions except for scrubbing temperature.

Raw Gas CF/Hr.	Liquor GPH	% T.E.A. Conc.	Scrubber Inlet	Temp. exit	% Exit CO ₂
11,000	595	46.4	149	157	0.0
11,200	595	47.0	165	172	1.2

Exit temperatures exceeding 165°F. cause the appearance of CO₂ in the exit gas unless, of course, more scrubbing liquor is used to compensate for the decreased absorption per gallon.

Flash drum operation was irregular throughout the test work. The temperature at which flashing took place varied from 192 to 207°F., but it was difficult to determine the extent to which the flashing was affected. From the inspections made, roughly half the dissolved CO₂ was released in the chamber. Operation of this apparatus would be much better if the chamber were larger in cross section to allow more disengaging area. The present apparatus is 12 in. in diameter and does not allow the gas sufficient time or area to disengage.

The actifier operating at about 225°F., kettle stripped the liquor down to approximately 0.5 CF CO₂/gallon. The top temperature was held at 180-190°F. This system operated satisfactorily, but at high ratings there was a tendency for it to boil over unless the preheat temperature was kept high. With a high kettle steam requirement at high ratings, the capacity of the packing is exceeded and the tower boils over. By putting more load on the preheater and flash chamber this tendency was overcome.

The total steam consumption in the preheater and actifier (when running a 200°F. preheat) was 50 lbs. per thousand CF raw gas. In actual plant practice, where use is made of heat exchange this figure amounts to 40 lbs./MCF gas.

3. Operation with Diaminopropanol

This substance absorbs CO₂ mol. for mol. whereas T.E.A. absorbs only half as much. With its high heat of absorption (900 B.t.u./#CO₂) it was considered inadvisable to use a higher solution concentration than 30% since the heat effect in the scrubber would be too great.

The first runs (Table II) were made at an equivalent rate of 8,000,000 CF H₂/day. In all of these the CO₂ was completely removed, but the liquor rate required varied considerably due to the large variation in stripping efficiency. The following tabulation shows the effect of residual CO₂ in the stripped liquor upon the liquor rate, all for the same gas rate, equivalent to 8,000,000 CF H₂/day.

Run No.	Liquor GPH	D.A.P. Conc.	CF CO ₂ /Gal. Stripped liquor	Steam Used #/MCF Raw gas	Remarks
2	340	31.1	4.36	-	Residual CO ₂
3	425	29.4	4.03	59	appeared.
3	425	24.7	2.90	74.5	
3A	340	24.9	2.90	102	

The high steam consumption in run #3A was incurred in trying to lower the CO₂ content below 2.9 CF/gal.

Further runs were made at equivalent gas rates of 12 to 16 mill. CF H₂/day (Table III). At these rates the scrubber operated satisfactorily with no entrainment nor flooding difficulties. The exit CO₂ at equivalent rates as high as 13,000,000 CF H₂/day was 0.0%, and amounted at times to as much as 0.6% depending on the stripping efficiency.

At these rates it became apparent that the actifier was operating very close to the flood point so that slight variations in kettle steam resulted in flooding. This is best shown by two runs noted below.

Raw Gas CFH	D.A.P. GPH	Preheat Temp.	Kettle Temp.	Steam CFH	Actifier % D.A.P. in Condensate
18,650	525	202	225	15,960	0.16%
19,850	525	190	232	17,000	1.19%

By increasing the kettle steam only 1000 CF/H, the D.A.P. content in the condensate from the vent gases rose from 0.16% to 1.19%, indicating boiling over.

At 16,000,000 CF H₂ ratings the exit gas contained about 5% CO₂ due to the fact that the liquor rate was not increased because of lack of stripping capacity. The runs indicated (Tables III and IV) however that the scrubbing tower was physically capable of handling the materials successfully without flooding.

Subsequent investigations (Table IV) were made on the actifier noting the effect of varying preheat temperatures. These are summarized on the following page.

Equip. Gas rates MM CF H ₂ /day	Equip. Liquor GPM	Preheat Temp.	% of Absorbed CO ₂ Flashed
15.3	315	220	27
14.0	330	222	73
13.1	315	215	42
14.6	330	215	33.5
16.2	315	210	25.6

These results indicate about 25% flashing of the absorbed CO₂ at 210°F, 35% at 215 and over 50% at 220°F. The data at 220°F. are not in agreement, but it is felt that the average of the two results is not far wrong. It will be noted, however, from Table IV that the steam consumption was greatly increased by the use of flashing.

In the runs made from November 20 to 23, the stripping steam was decreased to note the effect of increased CO₂ in the liquor upon scrubbing efficiency. Although the liquor analysis were inconsistent the results were nevertheless interesting.

Liquor Rate GPH	% Conc.	Gas Rate CFH	Actifior steam CFH	Stripped Liquor of CF CO ₂ /Gal.	% CO ₂ in Scrubbed Gas
525	28.1	20,000	16,068	5.27	0.0
525	26.6	20,000	13,936	2.73	1.0
600	29.1	20,000	14,000	5.96	0.0
600	33.0	19,800	12,000	6.80	1.4
525	20.0	20,000	14,000	2.28	1.0

At 525 GPH, lowering the kettle steam from 16,000 to 14,000 CF/H resulted in a rise of exit CO₂ from 0.0 to 1.0%. The stripped liquor analysis, however, showed a drop in CO₂ from 5.27 (28.1% solution) to 2.73 (26.6% solution). Raising the circulation rate to 600 GPH and keeping the kettle steam constant brought the exit CO₂ back to 0.0%, with a stripped liquor content of 6.8 CF CO₂/gal. These data indicate that fairly high contents of CO₂ in the circulating liquor may be used while completely scrubbing the raw gas of CO₂.

Runs were made at 173-180°F. scrubber outlet temperature (estimated for plant scale conditions using D.A.P.) to determine whether such a high temperature would require much of an increase in circulating rate to completely scrub the gas. At 173°F. scrubber outlet, 525 GPH circulations and 19400 CF/H raw Gas, the residual CO₂ varied from 2 to 4%. Increasing the circulation to 600 GPH (and at a scrubber outlet of 180°F.) the exit CO₂ was 1.4%. Since the operation of the plant scrub with 30% solution would result in temperatures above 180°F. when using theoretical liquor rates with a resulting increase in exit CO₂, it will be necessary to use greater circulation to reduce the temperature rise to about 165°F.

4. Extrapolation to Plant Scale Equipment

For operation at the Baton Rouge Hydro Plant up to 12,500,000 CF per day of pure hydrogen, the gas could be scrubbed free of CO₂ in the present plant system (two scrubbers and two actifiers) with TEA with no spare apparatus. It is realized that the experimental data were obtained in packed towers whereas the plant towers are equipped with bubble plates, but even these will be at capacity when using TEA.

TABLE IV
EXPERIMENTAL CO₂ SCRUBBING UNIT
SCRUBBING MEDIUM D. A. F.

Date	Time	Equivalent rates on Plant Scrubber		Rates on Experimental System		Temperatures		Steam, CF/H		*CF CO ₂ /gallon		% DAP Recovered					
		Pure H ₂ CP/L	D.A.F. % G.M.P.	Raw Gas CF/H	D.A.F. % G.P.H.	Scrubber Inlet	Scrubber Outlet	Pre-heat Kettle Top	Pre-heat Kettle	Exit CO ₂ %	Scrubber Inlet	Flash Drum	Stripped Liquor	Final Drum	Condensate		
11/15/34	9:30 PM	15,300,000	315	26.8	525	98	154	206	235	16,700	17,300	2.0	9.72	7.56	3.27	24.4	0.320
11/17/34	2:30 PM	16,100,000	315	27.4	525	98	162	204	230	16,800	15,200	3.2	10.00	7.56	4.48	28.6	0.626
11/17/34	12:30 AM	16,200,000	315	27.4	525	102	169	204	234	17,325	11,800	3.2	8.6	7.16	3.88	24.1	0.264
11/18/34	9:30 AM	15,800,000	330	26.2	550	94	155	200	235	16,270	13,402	1.8	9.22	7.68	4.47	3.17	0.209
11/19/34	5:30 AM	14,600,000	330	27.5	550	93	158	204	235	17,430	13,650	0.4	11.47	6.90	5.27	0.255	0.192
11/20/34	10:00 AM	14,000,000	330	30.3	550	105	160	222	210	17,430	10,394	1.4	11.47	6.90	5.27	0.255	0.192
11/20/34	4:45 PM	13,300,000	315	28.1	525	105	162	198	205	16,068	6,798	0.0	11.47	6.90	5.27	0.255	0.192
11/20/34	9:00 PM	13,300,000	315	26.6	525	102	152	200	204	15,386	8,034	1.0	11.47	6.90	5.27	0.255	0.192
11/20/34	6:00 AM	13,500,000	360	23.1	600	108	162	200	206	15,000	8,034	0.0	11.47	6.90	5.27	0.255	0.192
11/20/34	9:30 PM	13,200,000	360	33.0	600	108	158	201	203	15,090	8,736	1.4	11.47	6.90	5.27	0.255	0.192
11/23/34	6:00 AM	13,300,000	315	20.0	525	101	142	201	199	14,000	7,300	1.0	11.47	6.90	5.27	0.255	0.192
11/24/34	10:15 AM	18.2	20,000	525	18.2	105	145	201	195	14,215	7,910	1.2	11.47	6.90	5.27	0.255	0.192
11/24/34	8:30 PM	12,900,000	315	25.8	525	132	173	201	200	14,000	7,000	2.4	11.47	6.90	5.27	0.255	0.192
11/24/34	11:30 PM	12,900,000	360	23.8	600	155	180	205	205	14,000	7,000	1.4	11.47	6.90	5.27	0.255	0.192

TABLE III
EXPERIMENTAL CO₂ SCRUBBING UNIT, SCRUBBING MEDIUM D. A. P.

Date	Time	Equivalent Rate on Plant-Scrubber		Rates on Experimental scrubber		Temperatures				Steam CP/H				99 CO ₂ / Gal.				% D. A. P.			
		Pure H ₂ CP/D	GPM	% Conc.	Raw Gas CP/H	D. A. P. % Conc.	Scrubber		Actifier		Kettle	Pre-heat		Ao-tifier	% Exit CO ₂	Scrubber		Flash		Stripp. Liquor	Flash Liquor
							Inlet	Outlet	Pre-heat	Top		Exit	Exit			Flash	Liquor				
11/7/34	6:00 AM	12,650,000	315	29.5	19,000	525	107	148	207	207	225	8,944	---	---	0.5	9.9	9.0	5.78	26.4	5.78	26.4
11/7/34	12:00	12,200,000	315	21.2	18,400	525	100	151	201	203	231	7,696	---	---	0.4	7.2	8.7	4.00	27.7	4.00	27.7
11/8/34	2:00 AM	13,300,000	315	32.0	20,000	525	106	151	200	200	227	7,280	15,200	0.6	10.1	9.96	5.04	24.8	5.04	24.8	
11/9/34	6:00 PM	13,000,000	314	30.3	19,550	522	104	155	201	203	226	7,000	15,850	0.3	---	---	---	---	---	---	---
11/10/34	6:00 PM	12,850,000	315	30.3	19,300	525	101	155	202	200	226	7,240	15,960	0.1	---	---	---	---	---	---	---
11/11/34	6:00 PM	12,440,000	315	31.3	18,650	525	102	151	202	202	225	7,100	15,960	0.0	---	---	---	---	---	---	---
11/12/34	5:00 PM	13,200,000	315	---	19,850	525	103	146	199	197	232	6,600	17,000	0.0	---	---	---	---	---	---	---
11/13/34	9:30 PM	13,200,000	315	26.7	19,800	525	106	137	210	207	226	10,192	16,100	0.0	8.18	6.85	4.89	24.5	4.89	24.5	
11/13/34	9:00 PM	16,000,000	315	27.0	24,000	525	109	132	209	205	226	10,192	16,100	3.0	10.4	---	---	---	---	---	---

TABLE II

EXPERIMENTAL CO₂ STORAGE EFFICIENCY USING DIAMETHYLENE AS SCOURING MEDIUM
 TOWER PACKED WITH 2 INCH RASCHIG RINGS

Run No	Fuel Scrubbed lb-22/24	Equilibrium Pressure on Flue Gas	A. F. %	Status on Expor. Scrubber Raw Gas	Temp. in tower		CF CO ₂ /gal. liquor		% D.M.P. in con- dense	Steam CF/H	Exit CF/H
					Coiler inlet	Scrubber outlet	After Scrubber	After Stripper			
1	8,000,000	204	23.2	12,000	36.1	24.9	57	5.0	2.30	2.4	0.0
2	8,000,000	204	31.1	12,000	34.0	21.1	83	1.6	4.86	2.4	0.2
3	5,000,000	255	28.5	12,000	12.3	20.5	68	1.5	4.03	15,000	0.0
4	3,500,000	255	24.7	12,000	22.5	21.7	101	1.1	2.90	19,000	0.0
5	8,000,000	204	24.9	12,000	33.0	21.9	85	1.9	2.91	25,000	0.0

Table 1
EXPERIMENTAL CO₂ SCRUBBER OPERATION ON RAW HYDROGEN GAS USING TRIETHANOLAMINE AS SCRUBBING MEDIUM

Date	Run No.	Scrubbed H ₂ -CF/D	GPM	T.E.A. %		Experim. Scrubber Temperatures		Cooler		Pro-Heater		CF CO ₂ p. Gal. in Liquor		Steam CF/Hr.	Exit % CO ₂ from Scrubber	Remarks
				Conc.	Raw gas	CP/H	CPH	%	Outlet	Outlet	Outlet	Flash	After Stripped			
2/26/34	A	4,000,000	240	45.1	6,000	400	45.1	144	148	199	221	0.535	11,000	0.0	0.0	Scrubber filled with 3/4" Raschig rings.
2/27/34	B	4,000,000	222	35.0	6,000	370	35.0	142	152	202	227	-	-	0.0	0.0	Liquor came over.
2/28/34	1	4,000,000	240	19.7	6,000	400	19.7	154	156	204	226		9,200	3.1	3.1	Filled scrubber with 2" rings
2/28/34	1A	4,000,000	230	40.1	6,000	400	40.1	155	160	204	225		9,200	0.0	0.0	Concentr. probably too low.
2/28/34	2	5,180,000	265	42.0	7,800	440	42.0	142	152	204	216	0.446	9,200	4.1	4.1	
2/28/34	2A	5,180,000	295	37.4	7,800	491	37.4	147	153	207	221	1.47	10,000	0.0	0.0	
2/29/34	3	6,060,000	300	41.5	9,100	500	41.5	140	142	202	222	2.50	12,700	0.0	0.0	
2/29/34	4	6,260,000	300	43.4	9,400	500	43.4	152	159	201	235	0.72	10,400	2.6	2.6	
2/10/34	4A	6,260,000	336	47.7	9,400	560	47.7	147	150	192	226	0.53	13,500	0.0	0.0	
2/11/34	5	7,300,000	357	43.4	11,000	595	46.4	149	157	196	225	1.38	13,200	0.0	0.0	
2/11/34	5A	7,450,000	357	47.0	11,200	595	47.0	165	172	200	229	0.369	13,200	1.2	1.2	Tower flooded at higher liquor rates.
2/12/34	6	8,000,000	330	44.0	12,000	660	44.0	153	157	202	227		15,000	0.8	0.8	

With D.A.P. solution, all the gas can readily be handled with one scrubber, leaving one as a spare. For actifying this solution (about 400 GPM) both the present actifiers will be necessary, requiring the addition of another one as a spare, of 8.42 to 9 ft. diameter. In addition, the use of a flash drum is indicated, since the actifiers will be operating fairly close to the hang-up point (for packing or entrainment point (for bubble plates) and hence a flash chamber where some gas can separate and decrease the tendency of the liquid to form light droplets will be advantageous.

Item 22 o
Drawing of an Alkazid-scrubbing plant M-691-2

Item 22 p
Flow sheet of the Alkazid plant Me-936-a