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MEMORANDUM

ON

LOW PRESSURE SCRUBBING OF CO_2 FROM RAW HYDROGEN BY MEANS OF GIRBOTOL AND DIAMINOPROPANOL SOLUTIONS.

Introduction

This memorandum presents results of test work on an experimental CO_2 scrubbing system to determine the maximum capacity of a plant size 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 plates in the present 6 ft. diameter plant CO_2 scrubbers, the maximum capacity of each will be as follows, using girbotol (TEA) 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 "	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 G.P.M.	Residual CO_2 - %
2 inch	8,000,000	169	0,0
2 "	13,000,000	300	0,0

The capacities noted above for D.A.P. solution are not maxima. 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 H_2/day. When using D.A.P. at 30% concentration, the same stripper will handle the solution correspondin ~~when~~ to ~~8,000,000~~ CF H_2/day.

*) Estimated.

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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 CO_2 absorption, 900 B.t.u. vs. 600 B.t.u. per pound of CO_2 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 CO_2 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% CO_2 (s.g.0.365) was passed trough the tower in ~~xx~~-increasing amounts, using the minimum quantity of liquor to completely remove the CO_2. 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 CO_2 was completely removed. This troughput, translated into terms of one of the large 6 ft.I.D.towers is equivalent to 4 000 000 CF scrubbed H_2/day.

A short run was next made circulating pure water trough the system to determine whether the carry-over was due to liquid viscosity or entrainment. At 18 000 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 H_2/D 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 H_2/D on one large unit. It was not possible to circulate enough solution (44% TEA) to completely remove the CO_2 (because of tower flooding at increased circulation rates) but the final gas contained only 0,8% CO_2.

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

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Raw Gas CF/H	Liquor GPH	% T.E.A. Conc.	Scrubber Inlet	Temp. Exit	% Exit CO_2
11 000	593	46,4	149	157	0,0
11 200	595	47,0	165	172	1,2

Exit temperatures exceeding 165°F. cause the appearance of CO_2 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 CO_2 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 # CO_2/gallon solution. 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 CO_2 mole for mole, whereas TEA absorbs only half as much. With its high heat of absorption (900 B.t.u./# CO_2 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 H_2/day. In all of these the CO_2 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 CO_2 in the stripped liquor upon the liquor rate, all for the same gas rate, equivalent to 8 000 000 CF H_2/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 appeared.
3	425	29,4	4,05	59	
3	425	24,7	2,90	74,5	
3A	340	26,9	2,90	103	

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The high steam consumption in run #3A was incurred in trying to lowerthe CO_2 content below 2,9 CF/gal.

Further runs were made at equivalent gas rates of 12 to 16 mill. CF H_2/day (Table III). At these rates the scrubber operated satisfactorily with no entrainment nor flooding difficulties. The exit CO_2 at equivalent rates as high as 13 000 000 CF H_2/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.	Actifier Steam CFH	% D.A.P. in Condensate.
18 650	525	202	225	15 960	0.167
19 850	525	199	232	17 000	19,8.

By increasing the kettle steam only 1000 CF/H. the D.A.P. content in the condensate from the vent gases rose from 0.167% to 19,8% indicating boiling over.

At 16 000 000 CF H_2 ratings the exit gas contained about 3% CO_2 d 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 below:

Equiv. Gas Rates MM CF H_2/ D.	Equiv. Liquor GPH	Preheat Temp.	% of Absorbed CO_2 Flashed.
15,3	515	220	27
14,0	330	222	73
16,1	515	215	42
14,6	350	215	33,5
16,2	315	216	25,6

These results indicate about ~~25%~~ flashing of the absorbed CO_2 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 CO_2 in the liquor upon scrubbing efficiency. Although the liquor analyses were inconsistent the results were nevertheless interesting.

Liquor Rate GPH	% Conc.	Gas Rate CFH	Actifier Steam CFH	Stripped Liquor 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	15 936	2,73	1,0
600	29,1	20 000	14 000	5,96	0,0
600	33,0	19 800	12 000	6,3	1,4
525	20,6	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 CO_2 from 0,0 to 1,0 %. The stripped liquor analysis, however, showed a drop in CO_2 from 5,27 (28,1% sol'n) to 2,73 (26,6% Sol'n). Raising the circulation rate to 600 GPH and keeping the kettle steam constant brought the exit CO_2 back to 0,0%, with a stripped liquor content of 6,8 CF CO_2/gal. These data indicate that fairly high contents of CO_2 in the circulating liquor may be used while completely scrubbing the raw gas of CO_2.

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 175 °F. scrubber outlet, 525 GPH circulation and 19400 CF/H raw Gas, the residual CO_2 varied from 2 to 4%. Increasing the circulation to 600 GPH (and at a scrubber outlet of 180°F) the exit CO_2 was 1,4%. Since operation of the plant scrubber with 30% solution would result in temperatures above 180°F. when using theoretical liquor rates with a resulting increase in exit CO_2 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 [illegible] CF per day of pure hydrogen, the gas could be scrubbed free of CO_2 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.

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 8-1/2 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.

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TABLE I

EXPERIMENTAL CO_2 SCRUBBER OPERATION ON RAW HYDROGEN GAS USING TRIETHANOLAMINE AS SCRUBBING MEDIUM.

		Equivalent Rates on Plant Scrubber:			Experim. Scrubber			Temperatures			Actifier	CF CO_2 p. Gal. in Liquor		Steam	Exit % CO_2	
			T. E. A.		Raw Gas	T. E. A.		Cooler	Scrubber	Pre-heater		After	Stripped			
Date	Run. No.	Scrubbed H_2 - CF/D.	GPM.	% Conc.	CF/H	GPH	% Conc.	Outlet	Outlet	Outlet	Kettle	Flash	Liquor	CF/Hr.	from Scrubber	Remarks:
8/26/34	A	4 000 000	240	45,1	6 000	400	45,1	144	148	199	221		0,585	11 000	0,0	Scrubber filled with 3/4" Raschig rings.
8/27/34	B	4 000 000	222	35,0	6 000	370	35,0	142	153	202	227			-	0,0	Liquor came over.
9/5/34	1	4 000 000	240	19,7	6 000	400	19,7	154	156	204	226			9 200	3,1	Filled scrubber with 2" Raschig rings.
9/6/34	1A	4 000 000	240	40,1	6 000	400	40,1	155	160	204	225			9 200	0,0	Concentr. probably too low.
9/7/34	2	5 180 000	265	42,0	7 800	440	42,0	142	152	204	216		0,446	9 200	4,1	
9/8/34	2A	5 180 000	295	37,4	7 800	491	37,4	147	158	207	224	1,47	1,23	10 000	0,0	
9/9/34	3	6 060 000	300	41,5	9 100	500	41,5	140	142	202	222	2,50	0,72	12 700	0,0	
9/10/34	4	6 260 000	300	43,4	9 400	500	43,4	152	159	201	235	1,18	0,63	10 400	2,6	
9/10/34	4A	6 260 000	336	47,7	9 400	560	47,7	147	150	192	226			13 500	0,0	
9/11/34	5	7 [illegible] 000	357	[illegible]	11 000	595	46,4	149	157	196	225	1,38	0,389	13 200	0,0	
9/11/34	5A	7 450 000	357	47,0	11 200	595	47,0	165	172	200	229			13 200	1,2	
9/12/34	6	8 000 000	390	44,0	12 000	650	44,0	153	157	202	227			15 000	0,3	Tower flooded at higher liquor rates.

TABLE XI

EXPERIMENTAL CO_2 SCRUBBER OPERATION ON RAW HYDROGEN GAS USING DIAMINOPROPANOL AS SCRUBBING MEDIUM

TOWER PACKED WITH ½ INCH RASCHIG RINGS.

Date	Run Nr	Equivalent Rates on Plant Scrubber: Scrubbed H_2 - CF/D.	D. A. P. GPM	D. A. P. % Conc.	Rates on Exper. Scrubber: Raw Gas CF/H	D. A. P. GPH	D. A. P. % Conc.	Temperatures: Cooler Outlet	Scrubber Outlet	Pre-heater Outlet	Acti fier Kettle	CF CO_2/Gal. Liquor: After Scrubber	After Flash	Stripped Liquor	%DAP in Con-densate	Total Steam CF/H	Exit CO_2 %
9/14/34	1	8 000 000	219	23,0	12 000	364	23,0	86	150	201	224			2,30		–	0,0
9/15/34	2	8 000 000	204	31,1	12 000	340	31,1	83	155	202	220	10,62	8,10	4,36	2,4	–	0,2
9/16/34	3	8 000 000	255	29,4	12 000	425	29,4	5	151	200	220			4,05		15 000	0,0
9/18/34	3	8 000 000	255	24,7	12 000	425	24,7	91	151	203	221			2,90		19 000	0,0
9/18/34	3A	8 000 000	204	24,9	12 000	340	24,9	85	149	209	229		7,05	2,91	1,54	23 000	0,0

TABLE III

EXPERIMENTAL CO_2 SCRUBBING UNIT, SCRUBBING MEDIUM D.A.P.

		Equivalent Rate on Plant Scrubber			Rates on Experimental Scrubber			Temperatures					Steam CF/H		%	CF CO_2 / Gallon :			% D.A.P.	
					Raw Gas	D.A.P.		Scrubber		Actifier										
Date	Time	Pure H_2 CF/D	GPM	% Conc.	CF/H	GPH	% Conc.	Inlet	Outlet	Pre-heat	Top	Kettle	Pre-heat	Actifier	Exit CO_2	Scrubber Exit	Flash. Liquor	Stripp. Liquor	Flash. Liquor	Condensate
11-7-34	6:00 AM	12 650 000	315	29,5	19 000	525	29,5	107 41.7°	148	207	207	225	8,944	--	0,5	9,9	9,0	5,78	26,4	0,056
11-7-34	12:00	12 200 000	315	21,2	18 400	525	21,2	100 37.8°	151	201	203	231	7,696	--	0,4	7,2	8,7	4,00	27,7	9,8
11-8-34	2:00 AM	13 300 000	315	32,0	20 000	525	32,0	106 41.1°	151	200	200	227	7,280	15 200	0,6	10,1	9,96	5,04	24,8	
11-9-34	6:00 PM	13 000 000	31[illegible]	30,3	19 550	52[illegible]	30,3	104 40°	155	201	203	226	7,000	15 650	0,3			4,74		0,144
11-10-34	6:00 PM	12 850 000	315	30,3	19 500	525	30,3	101 38.4°	155 68.3	202	200 93.4	226	7,240	15 960	0,1			5,04		0,204
11-11-34	6:00 PM	12 440 000	315	31,3	18 650	525	31,9	102 38.9°	151 66.2	202 94.4	202 94.4	225 107.3	7,100	15 960	0,0			5,77		0,167
11-12-34	5:00 PM	13 200 000	315	--	19 850	525	--	103 39.5°	148 63.4	199 92.8	197 92.	232 111°	6,600	17 000	0,0					19,8
11-13-34	9:30 AM	13 200 000	315	26,7	19 800	525	26,7	106 41.1°	137 58.4	210 99	207 97	226	10,29[illegible]	15 100	0,0	9,18	6,85	4,89	24,5	0,0376
11-14-34	9:00 PM	16 000 000	315	27,0	24 000	525	27,0	109 42.8°	132 55.6°	209 98.4	205 96	226 107.8	10,182	16 100	3,0	10,4		4,17		0,132

TABLE IV.

EXPERIMENTAL CO_2 SCRUBBING UNIT

SCRUBBING MEDIUM D.A.P.

Date	Time	Equivalent rates on Plant Scrubber: Pure H2 CF / D.	D.A.P. G.M.P.	% Conc.	Rates on Experimental System: Raw Gas CF / H.	D.A.P. G.P.H.	% Conc.	Temperatures: Scrubber Inlet	Scrubber Outlet	Actifier Preheat	Actifier Top	Actifier Kettle	Steam, CF/H. Preheat	Steam, CF/H. Kettle	% Exit CO_2	CF CO_2/Gallon: Scrubber Outlet	Flash Drum	Stripped Liquor	% DAP Concentration: Flash Drum	Condensate
11-16-34	9:30 PM	15,300 000	315	26,8	23 000	525	26,8	98	154	220	206	235	16 700	17 300	2,0	9,72	7,56	5,27	24,4	0,329
11-17-34	2:30 PM	16,100 000	315	27,4	24 200	525	27,4	98	162	215	228	230	16 300	15 200	3,2	10,0	7,56	4,48	23,6	0,626
11-17-34	12:30 AM	16,200 000	315	25,5	24 300	525	25,5	102	160	210	204	234	17 325	11 300	3,2	8,8	7,16	5,88	24,1	0,264
11.18.34	9:30 PM	15,300 000	330	26,2	23 000	550	26,2	94	155	215	207	235	18 270	13 402	1,3					3,17
11-19-34	5:30 AM	14,600 000	330	27,5	22 000	550	27,5	93	158	215	204	235	17 430	13 620	0,4	9,22	7,68	3,47		0,209
11-20-34	10:00 AM	14,000 000	330	30,3	21 100	550	30,3	105	160	222	210	230	17 450	10 334	1,4	1147	6,9	5,16		0,857
11-20-34	4:45 PM	13,300 000	315	28,1	20 000	525	28,1	106	162	198	20[illegible]	226	16 068	6 798	0,0			3,27		0,255
11-20-34	9:00 PM	13,300 000	315	26,6	20 000	525	26,6	102	152	200	20[illegible]	223	13 936	8 034	1,0			4,73		0,192
11-20-34	6:00 AM	13,300 000	360	29,1	20 000	600	29,1	108	162	200	206	225	14 000	8 034	0,0			5,86		0,094
11-[illegible]-34	9:30 PM	13,200 000	360	33,0	19 800	600	33,0	108	158	201	208	219	12 000	8 736	1,4			3,8		
11-23-34	6:00 AM	13,300 000	315	20,0	20 000	525	20,0	101	142	201	199	240	14 000	7 300	1,0			3,28		3,04
11-24-34	10:15 AM	13,300 000	315	18,2	20 000	525	18,2	103	145	201	193	241	14 215	7 910	1,2			1,91		14,5
11-24-34	8:30 PM	12,900 000	315	25,8	19 400	525	25,8	132	173	201	200	222	14 000	7 000	2,4			4,23		1,93
11-24-34	11:30 PM	12,900 000	360	23,8	19 400	600	23,8	155	180	205	20[illegible]	223	14 000	7 000	1,4			4,13		0,057

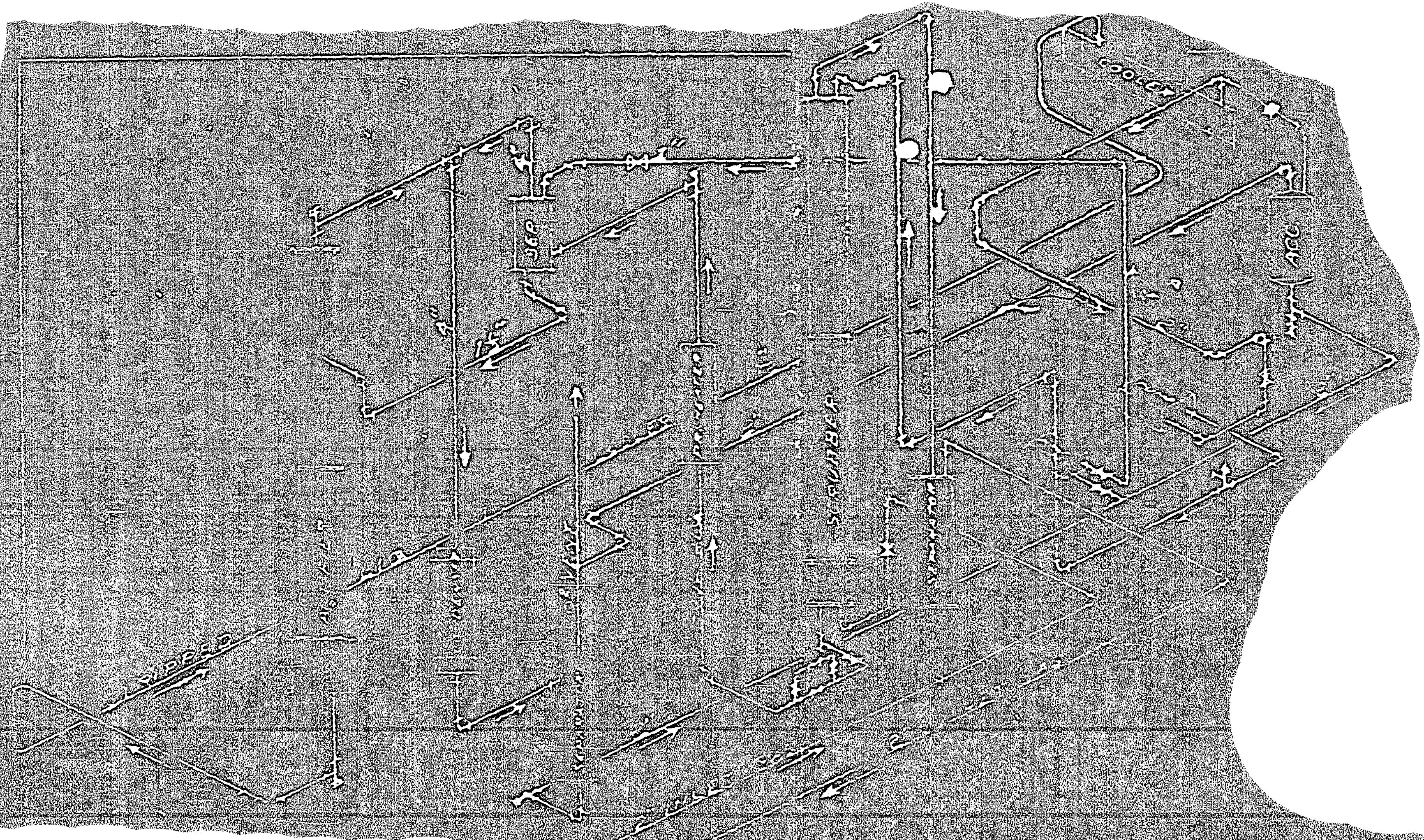

FIGURE 1
EXPERIMENTAL CO_2 SCRUBBER