Oppau - December 8, 1941.

THE OPERATION OF COPPER SOLUTION ABSORPTION TOWERS OF VARIOUS DESIGNS.

Previous experiments have indicated that the losses in hydrogen and nitrogen in the course of purification by washing with copper salt solution are due not only to solution of these gases in the absorbent but also to the trapping of gas bubbles in the copper salt solution; it was further observed that with the packing presently used in the absorption towers the losses of hydrogen and nitrogen are higher when level of the solution in the tower sump is low. On the basis of experimental results it has been proposed to avoid trapping of gas by keeping the solution level above the gas inlet inside the packed section of the tower. Besides raising the level of the solution the use of a "solution brake" was recommended and it was also proposed to use a low grate instead of arranging the grate higher in the tower and using a "solution brake". Lowering of the level of the solution decreases the sump volume but increases the effective absorption height of the tower. A large part of the absorption towers at Louna has been equipped with "solution brake" and this resulted in a decrease in the hydrogen and nitrogen content of the gas liberated from the solution from 29.6% (1936) to 20.7% (1939). However, during 1940 the amount of entrained gas increased again to 24.4%. A number of absorption towers in Oppau were equipped with grates arranged in the lower part of the tower but no unambiguous results relative to decrease of gas losses could be found in continuous operation. This is due to the fact that a number of factors as, e. g., the CO content of the raw gas, channeling of the washer, careful operation of the washer, specific solution consumption, and so on, affect the operation of the tower.

Analysis of the experimental methods used up to now indicated the following sources of error: The method of sampling using small amounts of the solution leaving the tower does not permit to follow the concentration of trapped gas over a prolonged period (several hours) so that the values obtained are only valid for the time when the sample was taken; previous comparative tests had been carried out on absorption towers of different designs so that no direct comparison could be made.

In order to obtain a clear quantitative picture as to which tower design is most effective in the prevention of the mechanical trapping of gas in the solution leaving the tower sump, tests were run in which the entire amount of solution in the tower is run into a special receiver; the quantity of gas liberated in this way is registered by a gas balance and the CO and CO_2 content of the liberated gas is determined every hour. The hydrogen content was also determined from time to time by means of combustion. Simultaneously with the absorption tower to be tested, a tower of the same design was operated as "control" under normal conditions. The solution from the "control" tower was also released into a special receiver and analyzed.

The feed of gas and copper salt solution to the tower, as well as the composition and the temperature of the fresh solution, were kept as constant as possible; comparative tests, however, had shown that slight differences in these conditions do not affect the amount of gas trapped. The experiments were mainly carried out with absorption towers of 1.6 feet diameter but towers of 2.6 feet diameter were also tested, although, in the latter case control tests could not be carried out because not sufficient receiver capacity was available. The difference in tower design consisted in the type of grate, the height at which the grate was arranged and, in a few cases, in the diameter of the gas inlet pipe.

The experimental conditions are given in the following tabulation and only the solution level in the tower sump was varied.

1. Experiments with Nitrogen.

Throughput: Tower of 1.6 feet diameter, 39 feet high; 215,000 cubic feet of gas per hour, 7,500 gallons of solution per hour.

Tower of 2.6 feet diameter, 36 feet high; 459,000 cubic feet of gas per hour, 15,850 gallons of copper solution/hour.

Specific Solution Requirement:
34.4 gallons per 1000 cubic feet of gas.

Composition of Raw Gas:
6.2% CO; 1.6% CO_2.

Washed Gas:
0.009% CO.

Fresh Solution:
Temperature - 75°F.
Composition:
0.15 pound atoms Cu^{++} per 100 gallons of solution.
1.24 pound atoms Cu^{+} per 100 gallons of solution.
0.011 cubic feet CO per gallon of solution.

Spent Solution:
1.95 cubic feet CO per gallon of solution.
2.2 psig. release pressure.

2. Experiments with Hydrogen.

Throughput: Tower 1.6 feet diameter, 39 feet high; 38,900 cubic feet of gas, 10,400 gallons of solution.

Specific Solution Consumption:
268 gallons per 1000 cubic feet.

Raw Gas Comsumption:
32.0% CO; 0.2% CO_2.

Washed Gas: 0.004% CO.

Fresh Solution:
As above.

Spent Solution:
2.16 cubic feet CO per gallon.
1.9 psig. release pressure.

The figures given are average values; deviations are caused by normal fluctuations in plant operations and are mainly limited to the throughput through the tower and the CO and CO_2 content of the raw gas. These deviations which amounted to ± 10% were, as mentioned before, without effect on the amount of gas trapped by the copper salt solution. The experiments were carried out for a period of 12 hours for each absorber but, in some cases, the test period was extended to 24 hours - 3 days.

The following conclusions can be drawn from the experimental results:

1. In the tower with a low grate whose gas inlet pipe is 4.6 feet long, only very small amounts of gas are trapped at a solution level of 3.9 feet. The figure for the concentration of nitrogen and hydrogen in the spent solution (0.214 cubic feet per gallon) ~~indicates that it is almost entirely due to physical solubility~~ since, under the conditions of the experiment, the solubility is 0.204 cubic feet of hydrogen and nitrogen per gallon of solution. Lowering of the solution level to 2.6 feet increased the gas losses.

2. Considerable quantities of gas are trapped in the high-grate absorption tower with a low gas inlet; at a constant solution level of 2.3 feet, differences of 2 inches in the length of the gas inlet tube have already a noticeable effect.

3. The gas losses in a high-grate absorption tower could be considerably lowered by the installation of a Leuna "solution brake'' when this change is made the tower is equivalent to a low-grate tower.

4. The greatest amount of gas is trapped by a high-grate tower with the gas introduced from the side.

5. It is not to be recommended to keep the solution level above the gas inlet.

The experiments have shown that the tower sump must be packed in order to avoid trapping of gas. As was shown by tests with the "solution brake" in Leuna, a layer of about 2 inches of Raschig rings inside the solution is sufficient to reduce the gas losses. The packing should cover 2 inches from the surface of the solution down. Since the level of the solution is not always maintained constant, it is preferable to have a somewhat greater depth of packed space. Trapping is controlled by the distance from the solution level to the grate or the lower edge of the "solution brake". In the present experiments it was found that under the experimental conditions selected this distance must be at least 2.3 feet for a tower of 1.6 feet diameter. Under these conditions the solution only carries away the physically dissolved gas when leaving the tower. In a high-grate absorption tower without packing in the sump, the copper salt solution must have sufficient time for release of trapped gas and this can be reached by a correspondingly high solution level or a sufficiently long residence time in the sump. Leuna has adopted this measure and requires a solution level of 5.3 feet for the 2.6 feet diameter tower but, as indicated in the experimental results, this solution level is not sufficient to limit gas losses to the gas in physical solution. When a choice is to be made between a low-grate tower and a tower equipped with a "solution brake", the former should be preferred for the following reasons: The installation of a low grate is considerably simpler, the height of the tower can be utilized better and the pressure drop is lower in a low-grate tower since there is no loss in the available cross-section caused by a second grate. The objection that in low-grate towers the gas inlet pipe is subject to copper deposition does not seem to be justified. As observed on the occasion of the inspection of a larger number of towers in 1940, copper deposition on the gas inlet tube takes place primarily when the solution level is kept above the gas inlet, which means that the mouth of the gas inlet pipe is in the solution and also in the case that the diameter of the gas inlet tube exceeds a certain dimension. In that case the gas velocity is not sufficiently high to eject the solution which might have entered the pipe. The gas inlet can be protected against copper deposition by an arrangement shown in Figure 5. Some washers have been equipped with a syphon above the solution outlet pipe in order to avoid gas entering the pipe due to eddy formation but this measure was only successful in the high-grate towers, whereas it was found unnecessary in low-grate towers.

In the design of the low-grate towers the grate was usually arranged at a height of 1.6 feet above the bottom of the tower but there are no theoretical reasons for this choice and, according to the present experimental data, even the installation of a grate is theoretically unnecessary. However, practical considerations speak against such a design in which the packing is placed directly on the bottom of the tower, since, in this case, the packing could settle in front of the solution outlet.

The following practical conclusions for the arrangement of the packing of absorption towers can be drawn from the experimental results:

Height of the Grate:	4 inches above the tower bottom.
Gas Inlet:	At least 2.6 feet above the grate.
Solution Level:	At least 2.3 feet above the grate.
Diameter of the Gas Inlet Pipe:	Equal to that of the raw gas inlet pipe outside the tower.

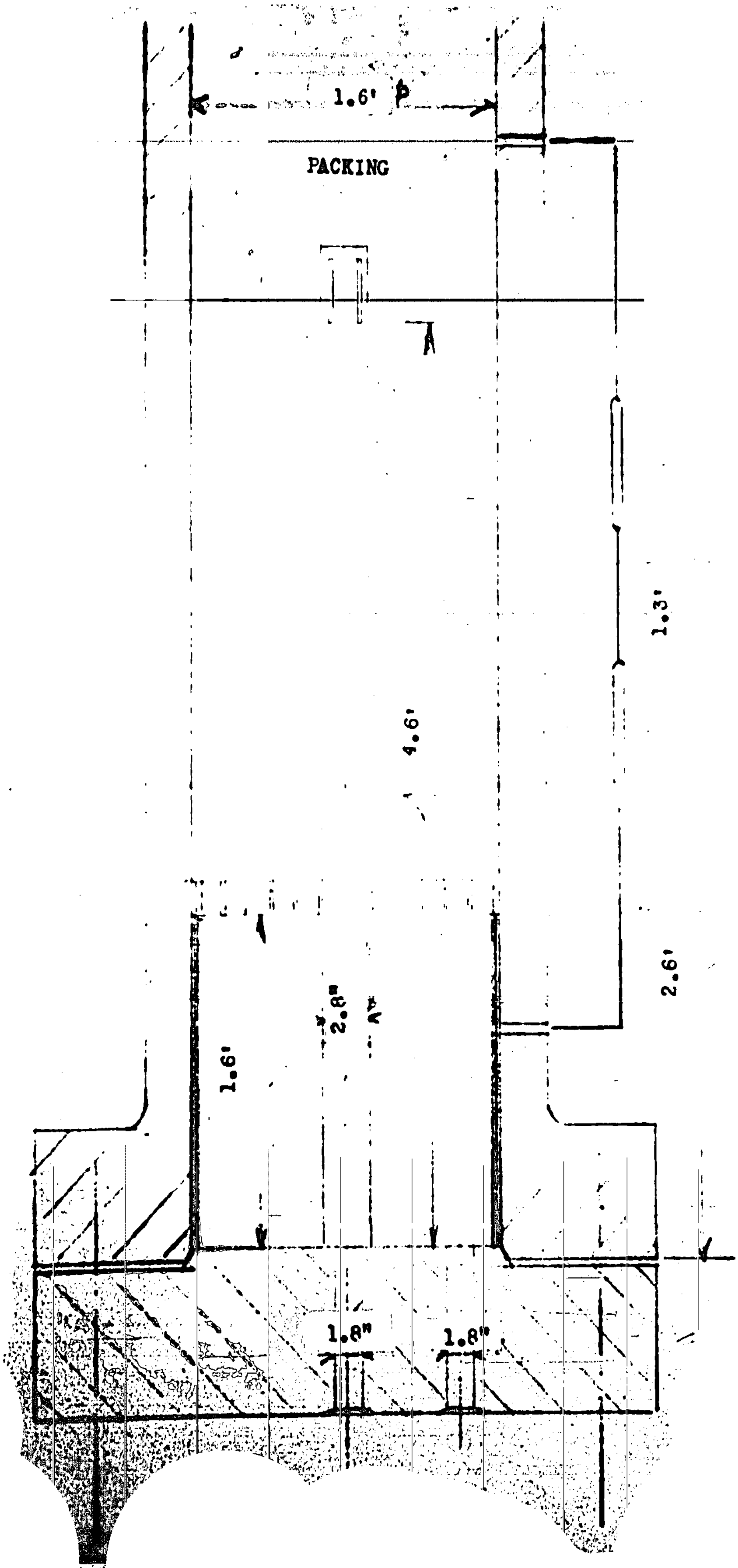
With respect to the composition of the gas liberated from the spent solution, it will be found that on comparison of the two last columns of the attached table the figures in the next-to-the-last column do not always correspond to those in the last column. This is explained by the fact that the figures in the last column were calculated from the next-to-the-last column with consideration of the total quantity of gas and solution depressured. The amount of gas liberated was, in the present experiment, dependent on the CO and CO_2 content of the raw gas and the specific solution consumption. With a change in these conditions, changes in the composition of the liberated gas occurred. This indicates that the effectiveness of the tower can only be estimated approximately from the hydrogen and nitrogen content of the gas obtained on depressuring the solution.

In order to obtain best results it is, of course, necessary to observe the rules of good operating practice. Frequent priming of the tower which is caused by sludge precipitation in the tower causes a considerable increase in the hydrogen and nitrogen concentration of the gas liberated from the absorption solution.

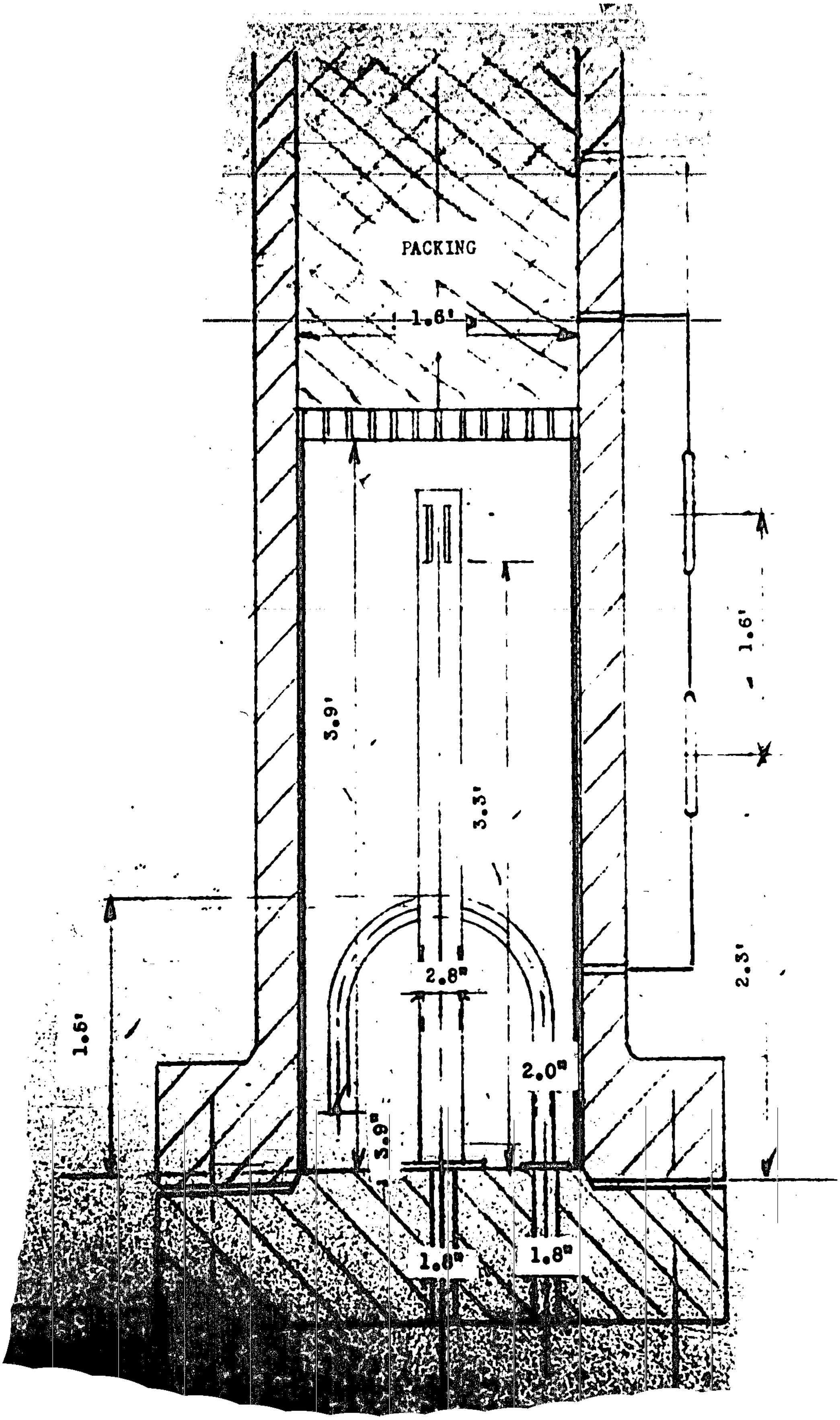
Experimental Results

Type of Grate	Height of Grate ft.	Gas Inlet ft.	Liquid Level ft.	% $H_2 + N_2$ in depressured gas	Vol. of $H_2 + N_2$ per vol. of Solution	Remarks
			N_2 1.6 ft. diameter tower			
Low grate	1.6	4.6	3.9	11.6	1.6	Fig. 5
"	1.6	4.6	2.6	13.9	2.0	"
"	1.6	4.6	2.6	13.3	2.0	"
"	1.6	4.6	3.9	12.1	1.6	Fig. 1
"	1.6	4.6	2.6	13.2	2.1	"
High grate	3.9	3.3	2.3	22.5	4.1	Fig. 2
"	3.9	3.4	2.3	20.8	3.5	"
"Solution brake"	3.9 & 1.6	3.4	2.3	13.5	2.2	Fig. 3
Low grate	1.6	3.4	2.3	16.0	2.3	-
High grate	5.9	5.7	4.6	26.3	5.1	Fig. 4
"Solution brake"	5.9 & 1.6	5.7	4.6	13.6	1.9	-
			N_2 2.6 ft. diameter tower			
Low grate	1.6	3.9	5.1	14.8	1.8	-
High grate	5.5	5.9	5.3	18.5	2.2	-
			H_2 1.6 ft. diameter tower			
Low grate	1.6	4.6	3.9	16.0	1.6	Fig. 1
"	1.6	4.6	2.8	19.9	2.1	"
Medium high grate	2.3	3.6	2.3	19.3	2.4	-

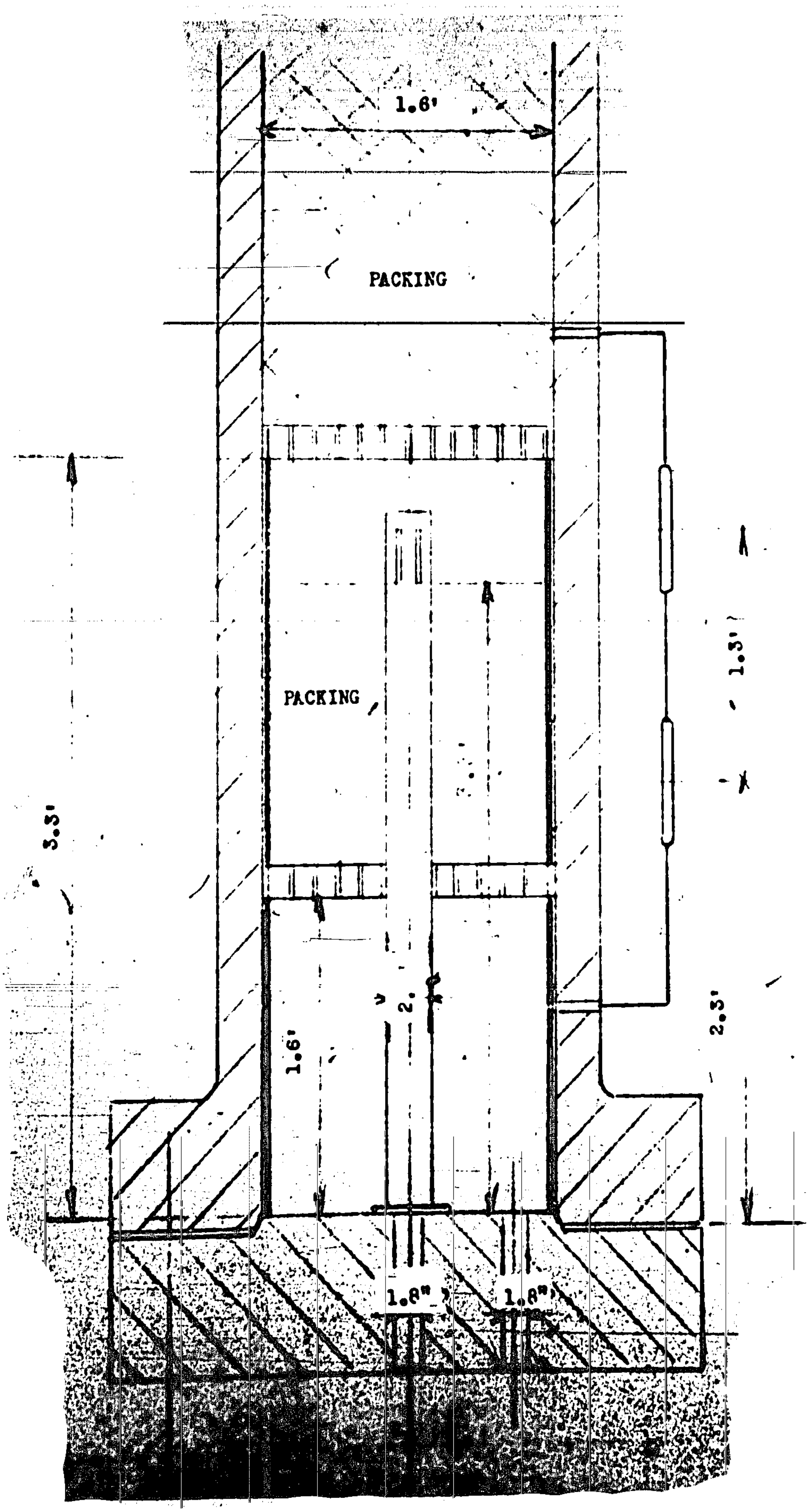
I. TOWER WITH LOW GRATE



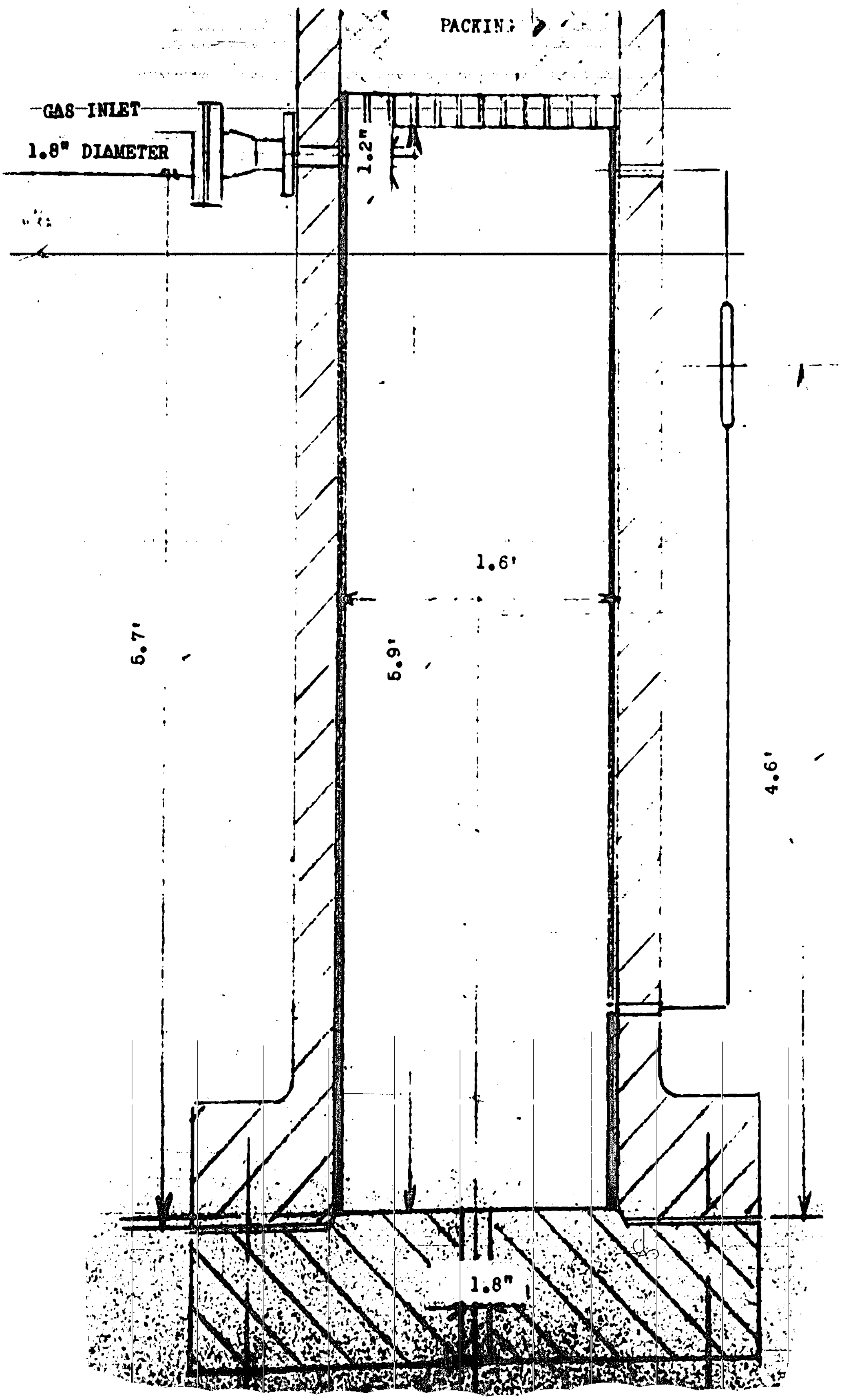
II. TOWER WITH HIGH GRATE



III. TOWER WITH "SOLUTION BRAKE"



IV. TOWER WITH HIGH GRATE AND GAS INLET AT THE SIDE



V. TOWER WITH PROTECTED GAS INLET PIPE