which is very likely at slow flows unless all particles are very small. This method can also be used on ducts too small for the usual procedures. It is unnecessary to know the pressure or temperature in the duct, but a correction must be applied for any difference in conditions of flow measurement in the main gas stream and the sampling stream.

Detailed directions for dust sampling in the range of concentrations and particle sizes occurring in coal-fired furnace stacks are given in the ASME Power Test Code 21 (1941) and other publications.22/

Thus, it is essential that the gas velocity in the sampling tube be the same as the velocity in the duct. This is most often done by making the static pressures equal. Samples should be taken at several points across the cross section of the duct. It is simpler, but much less reliable, to sample at a single point (the point of mean velocity). In this case, the proper sampling velocity can be calculated from the flow in the duct and relative cross sectional areas. Sometimes this method is necessary in very small ducts.

Sampling in Pilot Plants

In addition to the dust-sampling experiments made in the laboratory on small controlled gas streams, hundreds of dust determinations have been made during the last 5 years at various points on a small gasification unit, which gasifies 50 pounds of coal per hour (fig. 4) and also on a large 500 pound hourly capacity pilot plant (fig. 5). Complete details of construction, operation, and results on the smaller unit were given by Sebastian23/ and on the large unit by Strimbeck and Schmidt24/25/26/ The Annual Reports of the Secretary of the Interior for 1950-52 give more recent information. Also, tests are currently being made on three newer pilot plants, including a high-pressure, coal-gasification unit.

The previous discussion refers to actual sampling of the gas stream. Removal of the solid and liquid impurities from the sample gas stream and their examination will be discussed under separate headings.

The difficulties characteristic of pilot-plant dust sampling were encountered at nearly all points. Ducts were necessarily too small for traverses, velocities too low to use the static pressure method for obtaining equality of velocity, and pipe lengths usually too short for good sampling locations.

22/ Western Precipitation Corporation: Bull. WP-50, 1951.

23/ Sebastian, J. J. S., Edburn, P. W., Bonar, F., Bonifield, L. W., and Schmidt, L. D., Laboratory-Scale Work on Synthesis-Gas Production: Bureau of Mines Rept. of Investigations 4742, 1951, 41 pp.

24/ Strimbeck, G. R., Holden, J. H., Rockenbach, L. P., Cordiner, J. B., Jr., and Schmidt, L. D., Pilot-Plant Gasification of Pulverized Coal with Oxygen and Highly Superheated Steam: Bureau of Mines Rept. of Investigations 4733, 1950, 41 pp.

25/ Schmidt, L. D., McGee, J. P., and Sloane, M. C., A Pilot Plant for Gasifying Powdered Coal Entrained in Oxygen and Steam: Chem. Eng. Prog., vol. 44, No. 10, October 1948, pp. 737-744.

26/ Strimbeck, G. R., Holden, J. H., Rockenbach, L. P., Cordiner, J. B., Jr., and Schmidt, L. D., Pilot-Plant Gasification of Pulverized Coal with Oxygen and Highly Superheated Steam, Am. Gas Assoc. Tech. Paper PC-50-11, May 1950, 64 pp.

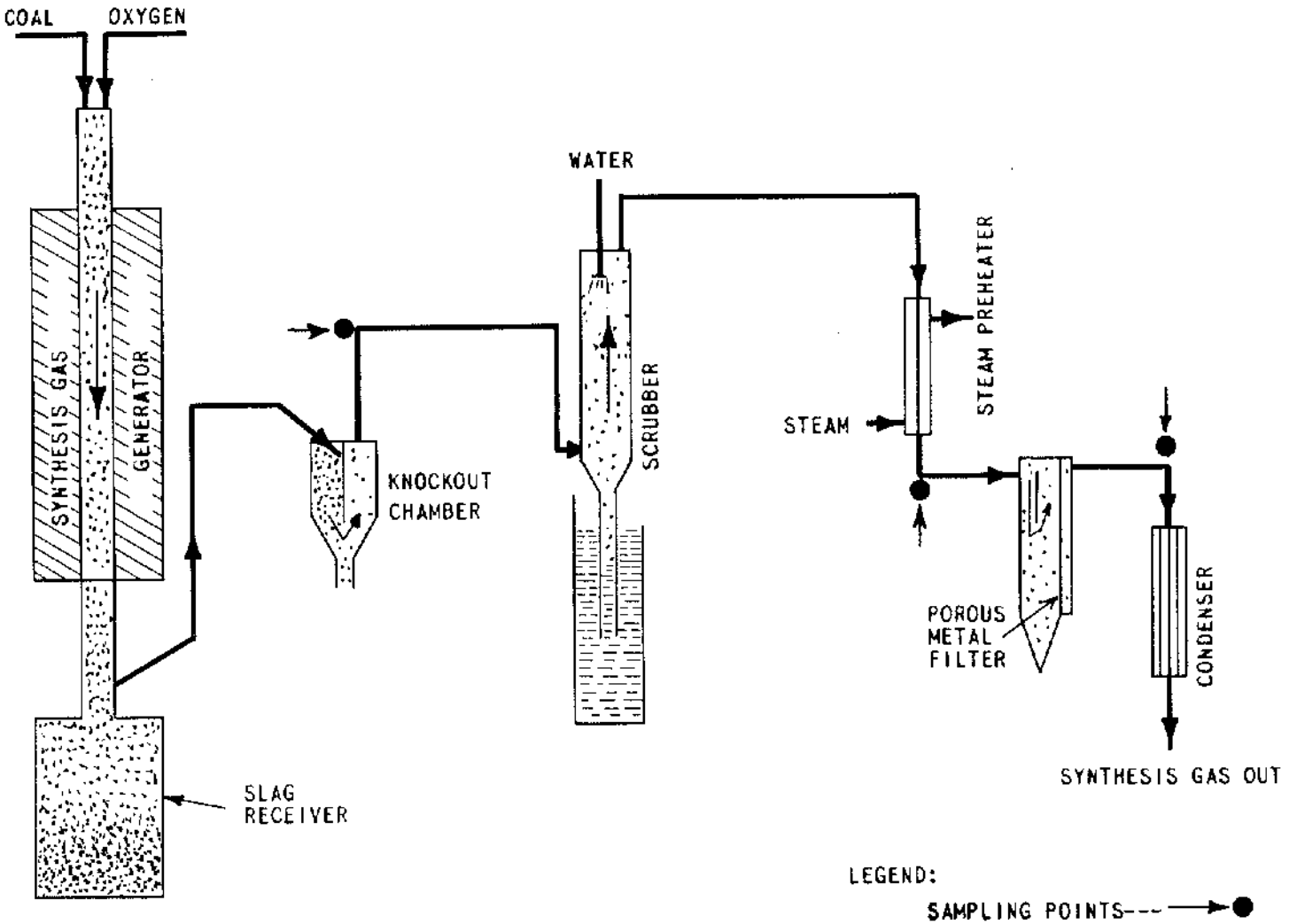

Figure 4. - 50-pound-per-hour coal-gasification unit showing dust-sampling points.

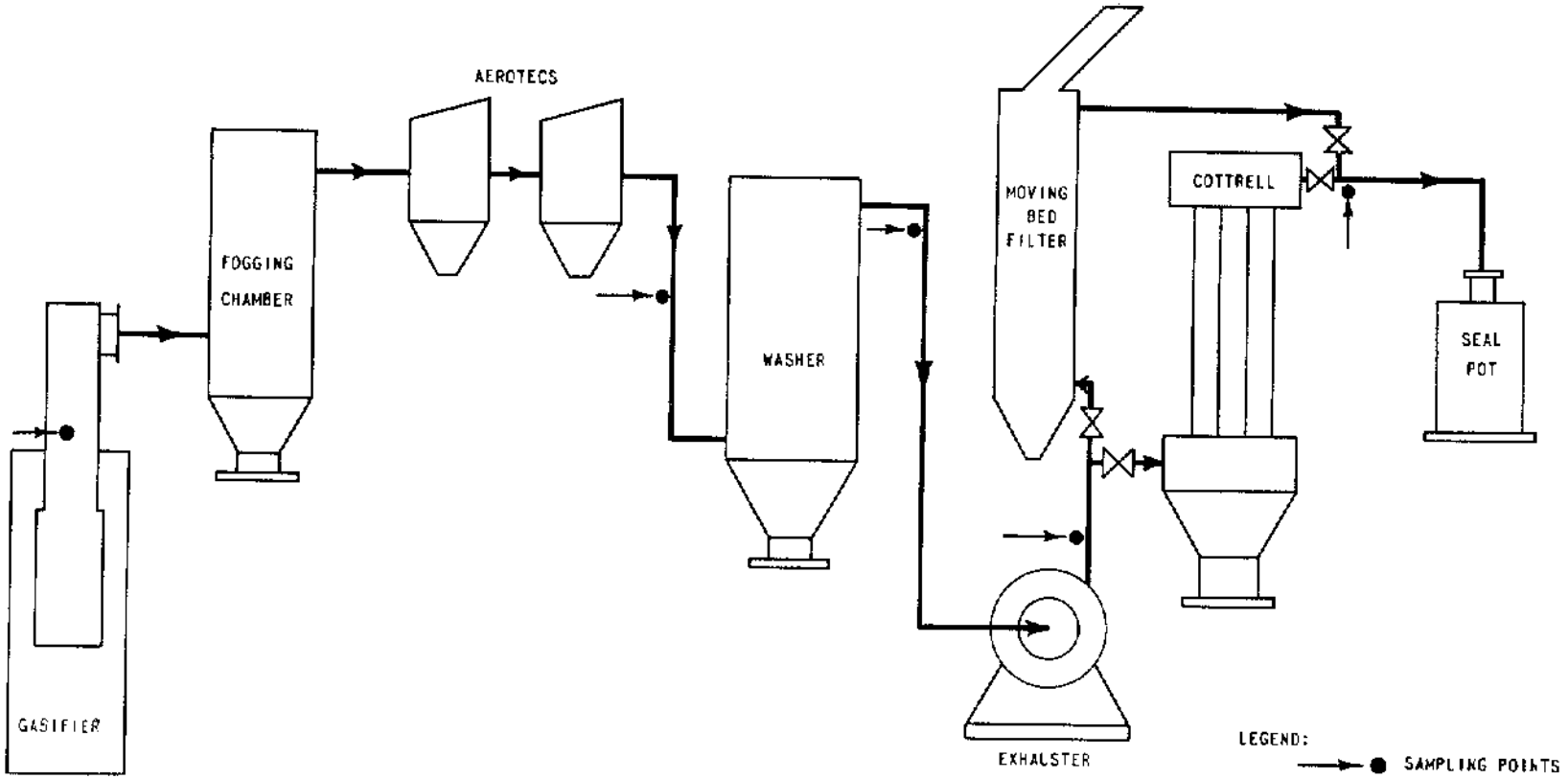

Figure 5. - 500-pound-per-hour coal-gasification unit showing dust-sampling points.

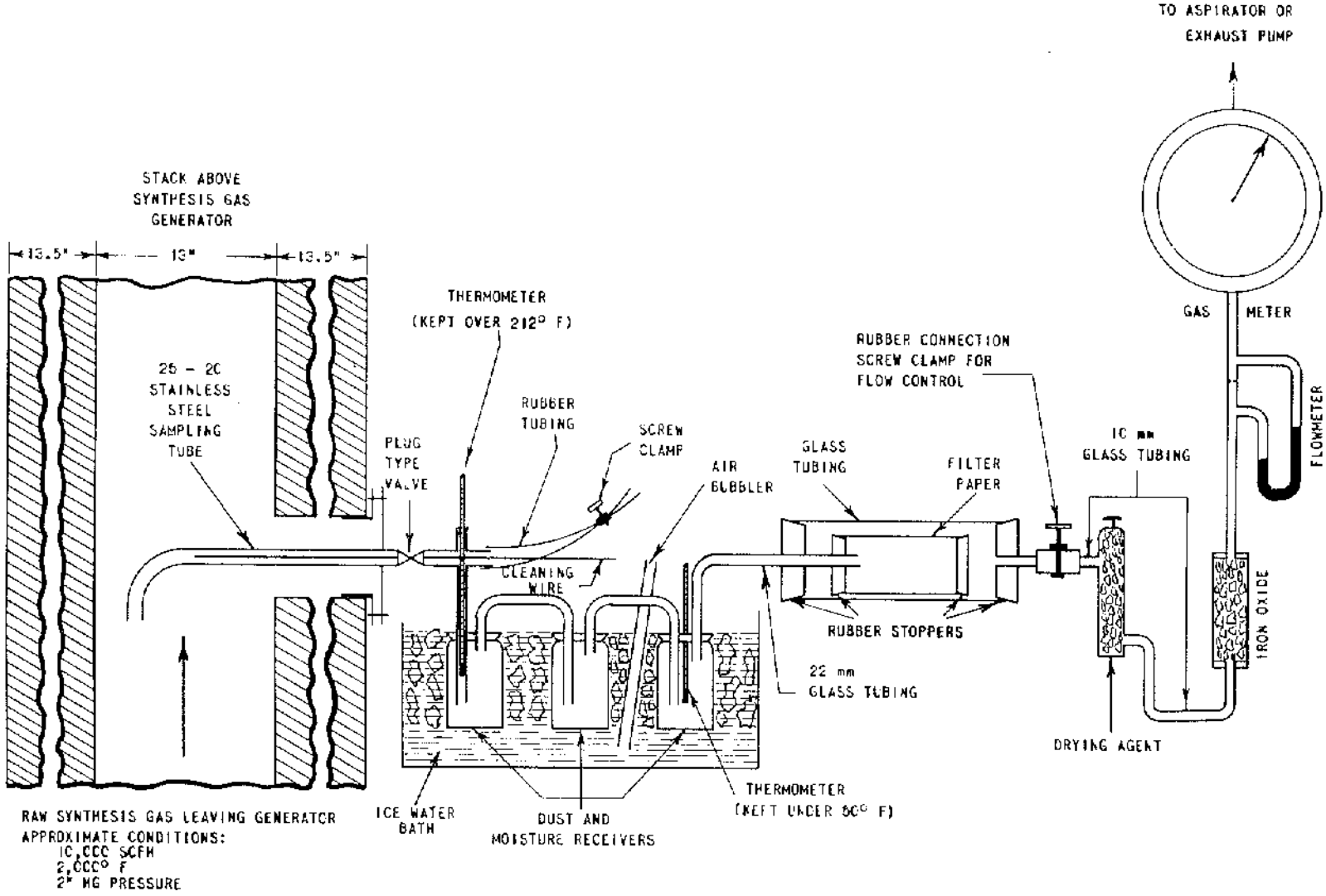

Figure 6. - Apparatus for determining dust and moisture in crude, hot synthesis gas containing condensible moisture.

An additional difficulty - plugging of the sampling tube - was frequently encountered when partly purified gases containing liquid water mixed with the solid impurities were sampled. The cleaning wire shown in figure 6 made it possible to obtain samples under these conditions, but the results were inaccurate. Use of this wire was a wise precaution even when no plugging was noticed, because it was felt that a partial closure of the tip of the sampling tube might occur with no indication of its existence.

The plug-type shut-off valve in the sampling line did not clog. The valve was used fully open, and flow control was obtained with a screw clamp on the clean gas-line. On the smaller plant the moisture problem was satisfactorily solved by heating the main gas stream until definitely unsaturated with respect to water vapor, as shown in figure 4.

High Temperature Sampling

The most useful results on gasification units were obtained from samples of the hot, raw gas leaving the top of the generator before any material was added or removed. Fortunately, sampling at points of high temperature presented less difficulty owing to liquid water in the sampling stream. Since the gas entered the sampler at temperatures over 1,500° F., all water in the duct was present as vapor. The temperature at the outlet of the sampling tube depended on the rate of gas flow through it. If too low, liquid water would condense in the tube; and if too high, the cooling flasks would overheat and liquid water would enter the filter. However, the rate of flow of gas in the sample stream could not be varied for this purpose. Since it was necessary to use the flow that gives equal velocities inside and outside the tip of the sampling tube, the temperature of the gas leaving the tube had to be controlled by altering the diameter of the tip. This additional factor had to be considered in determining the proper sampling-tube diameter. With the equipment shown in figure 6, it was found that a flow of about 15 std. cu. ft. per hr. allowed the gas stream to leave the sampling tube at an appropriate temperature without overheating of the flasks and consequent water deposition in the filter.

It is important to note that, under these conditions, moisture sampling was merely gas sampling and was not affected by the errors previously mentioned, which apply only to the sampling of materials existing as solids or liquids in the gas stream. Therefore, the determination of moisture was far more accurate than the dust determination made simultaneously.

Owing to the slow gas flow and high dust loading, occasional clogging occurred, even though no water condensed in the sampling tube at the top of the generator; however, use of the cleaning wire usually permitted immediate resumption of the test. Occasionally it was necessary to clean the sampling tube by blowing back with compressed gas. Some of the plugging might have been prevented by reducing the diameter of the sampling tube immediately above the inlet to increase the velocity, but this would not have prevented clogging at the tube tip where the velocity could not be changed since duct velocity had to be used. Higher duct velocities in large plants would probably eliminate this difficulty. No such clogging occurred during any of the sampling experiments in laboratory investigations.

Sampling tubes of 25-20 stainless steel proved satisfactory for most of the high-temperature tests in which the gas entered the sampler at a temperature of about 1,600° F. but failed in one series of tests at approximately 1,900° F. A fused-silica tube was substituted. These difficulties were encountered only when crude or slightly purified synthesis gas was sampled. Samples of highly purified gases were obtained without difficulty.

Thus sampling, and especially selection of sampling point, is the most important factor in dust determination. This is frequently very difficult, especially in small plants. Equal velocity in the sampling tube and the duct is essential.

SEPARATION OF SOLID AND LIQUID IMPURITIES FROM THE SAMPLE GAS STREAM

Many methods commonly used to remove dust from gas streams for analytical purposes were studied. Most promising were thermal precipitators, sedimentation cells, cyclones, electrostatic precipitators, impingers, and filters. Thermal precipitators require extremely slow flows but have been used in some cases for dust determination.[27] Sedimentation cells are generally used as absolute laboratory methods, but the principle was found satisfactory for removing the bulk of the dust and all condensable moisture from crude synthesis gas. Condensation of water on the dust particles increased the effectiveness of the method. The method of sedimentation was also used for determining the degree of dust agglomeration in the gas stream. Details are given under "Particle-Size Measurement."

Small cyclones about 2 inches in diameter were found valuable for gross dust removal from highly contaminated gases to relieve the load from the following filter. They were not effective for particles smaller than 5 microns and seldom could handle mixtures of solid and liquid impurities.

Electrostatic precipitators are very efficient, but small sizes for analytical purposes[28] were considered hazardous for highly combustible gases.

Impingers of various types are used extensively in industrial hygiene work, especially in the field, because of their speed and convenience for particle counts. In synthesis-gas-production research, however, it was felt that weight concentration is the more significant measure of gas purity. Although in the work described impingers were used for tar and gum determinations most solid and liquid separations were made by filtration.

A large number of earlier devices for dust removal have been described by Knowles.[29] The relative merits of different methods have been analyzed and evaluated by Drinker[30] and Stairmand.[31] A recent summary and bibliography on the industrial hygiene aspects of the subject is given by Forbes[32] and also in the Air Pollution Abatement Manual.[33]

The removal of solid and liquid impurities from crude and highly purified synthesis gas presented two entirely different sets of problems and will be discussed in the following paragraphs.

27/ Brendl, J. and Grive, T. W., Jour. Sci. Inst., Vol. 28, 1951, p. 21.
28/ Drinker, P., An Inexpensive Precipitator Unit: Jour. Ind. Hyg., Vol. 14, 1932, p. 364.
29/ Knowles, R. R., Dust Determinations in Air and Gases: Trans. Am. Soc. Heat. and Vent. Eng., Vol. 25, 1919, pp. 67-98.
30/ Drinker, Philip, and Hatch, Theodore, Industrial Dust: McGraw-Hill Book Co., N.Y., 1936, p. 130.
31/ See work cited in footnote 16, pp. 21-30, and Appendix III.
32/ Forbes, J. J., Review of Literature on Dusts: Bureau of Mines Bull. 478, 1950, 333 pp.
33/ 1952 Manufacturing Chemists Association, 15th and H. Sts., N.W., Washington 5, D.C.

Crude Gas

The desired method for removing impurities should, if possible, be accurate enough for material-balance calculations and give samples suitable for any desired examination of the dust for theoretical or control purposes. Methods such as are described by Altieri[34] are not applicable to synthesis gas, which contains fewer impurities of the types common in industrial gases.

As the raw synthesis gas contained 20 to 50 percent moisture, it was necessary to remove the dust at a temperature above the dew point or to remove solid and liquid impurities simultaneously. The latter method was used on product streams from gasifiers operating at atmospheric pressure, but the former may prove to be more satisfactory for testing streams from the new 450-p.s.i.g. pressure gasifier. Glass filter cloth may be necessary to remove impurities in both cases, as proposed by Western Precipitation Co.[35] The equipment as finally developed is shown in figure 6.

The gas enters a sampling tube of such diameter that moisture condenses initially in the first receiver. The temperature in the inlet tube to the first flask is held above 212 F. to prevent moisture condensation up to this point. It was found that the shutoff valve shown must be of plug type, with an opening of the same size as the tubing to prevent clogging. It must be used fully open and the flow controlled with the screw clamp placed after the filter. The entrance point of the cleaning wire was made gastight by passing the wire through a hole pinched in the side of a piece of laboratory rubber tubing, as shown.

Originally 300-ml. Erlenmeyer flasks and 10-mm. connecting glass tubing were used to collect the dust and condensed moisture, but frequent clogging occurred. Later 22-mm. connecting tubing was used with satisfactory results. This required wide-mouth but lightweight bottles or flasks for receivers. One-pint fruit jars were also used occasionally. They are sturdier (an important factor when working in the plant) but heavier than desirable for weighing on an analytical balance. These moisture and dust receivers are kept in a bath of ice water surrounded by insulation. Three flasks are enough to prevent liquid water from entering the subsequent filter at a gas flow of 15 cubic feet per hour or less, even with 40 percent moisture in the gas. Water weakens the filter paper in the filter tube and often decreases the capacity of the filter by causing high resistance with a thin layer of dust.

Filters

To remove the remaining dust, paper Soxhlet extraction thimbles were used in the glass holder tube shown in figure 7 or in the metallic holder tube advocated by Research Corp.[36] The latter is very heavy but can be used under pressures as high as 100 p.s.i.g. Less hygroscopic filter mediums, especially alundum, porcelain, and porous stainless steel plates, were also used occasionally. When used on crude gases, these filter mediums had the advantage that prior removal of moisture was not required and moist dust could be weighed as it actually existed in the gas stream. Stairmand[37] found stellite filters to be 100 percent efficient, even for extremely fine dusts, and used them to determine the efficiency of other filtering materials.

34/ Altieri, V. J., Gas Analysis and Testing of Gaseous Materials: 1st. ed., Am. Gas Assoc., 420 Lexington Ave., N.Y., N.Y., 1945, pp. 431-452.
35/ See footnote 22.
36/ Research Corp., Bound Brook, N.J.; Tech. Bull. 3-E.
37/ See work cited in footnote 16, p. 31.

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All devices described above have rather low capacities. In some cases the filtering capacity could be increased somewhat by loosening the layer of dust by physical means, but generally a larger filtering area was required. For this reason a larger holder device using filter paper (shown in fig. 8) was developed. Its use is especially advisable if only a few determinations are planned, because it can be fabricated from standard laboratory equipment. Another advantage of glass-holder devices for filtration is that the direction of flow through them may be reversed, and the dust deposited on the outer surface of the filter medium can be seen and examined. Thus, valuable information on the character of dusts tested for the first time may be obtained, although reversal of the flow makes the weighing more difficult and less accurate.

Later, a metallic holder of different design was used. The filter paper was bound to the holder with wire and then fastened lengthwise with drafting tape. This was satisfactory for determinations made on crude gas of high dust content. For determinations on highly purified gases, a metallic holder with a special binding device (fig. 9) was required to decrease the error in weighing the filter paper.

Large-area filters are also required when duct velocities approach 100 feet per second, because the minimum practical diameter for the sampling tube (1/4 inch) takes 122 cubic feet of gas per hour, which is more than a Soxhlet thimble can safely handle.

A short investigation was made of the relative merits of different filter papers. Perot studied the characteristics required in a paper for separating fine particles from a gas.38/ It was necessary to make tests to determine the approximate flow resistance of different filtering materials since no such information was found in the literature.

The results of this study are shown in table 1. Due to the small pore spaces, the pressure drop is approximately proportional to the first power of the flow instead of the second power as in larger orifices. Filter paper was tested between two glass tubes using soft rubber gaskets, while thimbles were placed on a rubber stopper through which the air was fed. A wide range of flows was used in each, but all were calculated to a common basis for this table. In all cases, velocity head and friction were shown to be negligible by blank tests with no filtering materials in the system. It is evident from this table that filter papers are available in a very wide variety of densities, which can meet the retentivity requirements of any particular application. The table also shows that highly retentive papers have a high pressure drop at a velocity of 1 foot per second, so that much lower velocities should be used. It must be remembered that these tests were made on new filters. As dust collects on the paper the resistance increases greatly. Yant and his co-workers39/ used Whatman No. 42, a highly retentive paper, in their determinations of dust in air in the Holland tunnel. A pad of three papers was used, and no evidence of leakage of dust through the first paper was found.

38/ Perot, J. J., Study of Certain Factors Affecting the Filtration of Smoke by Fibrous Materials: Paper Trade Jour., Vol. 124, No. 25, 1947, p. 54.

39/ Yant, W. P., Levy, Edward, Sayers, R. R., Brown, C. E., Traubert, C. E., Frevert, H. W., and Marshall, K. L., Carbon Monoxide and Particulate Matter in Air of Holland Tunnel and Metropolitan New York: Bureau of Mines Rept. of Investigations 3585, 1941, p. 22.

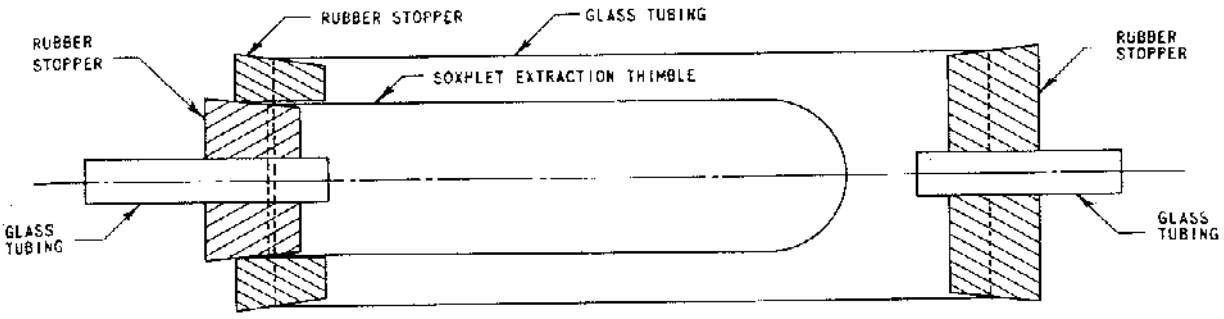

Figure 7. - Small filter-thimble holder.

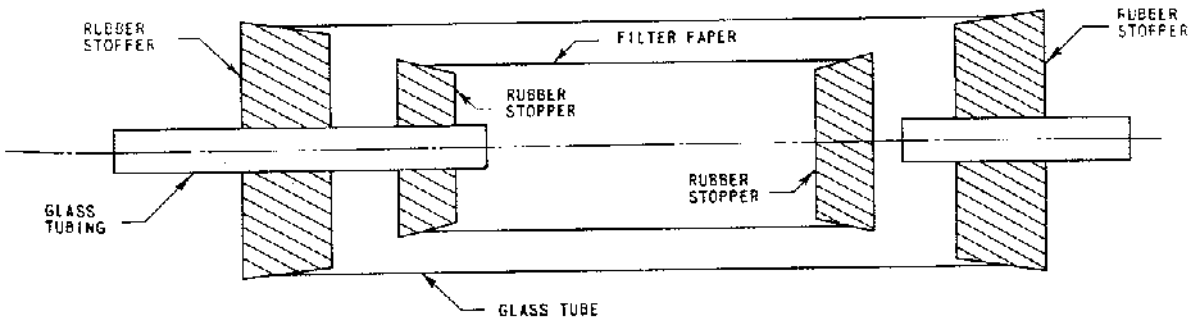

Figure 8. - Large, glass, filter-paper holder.

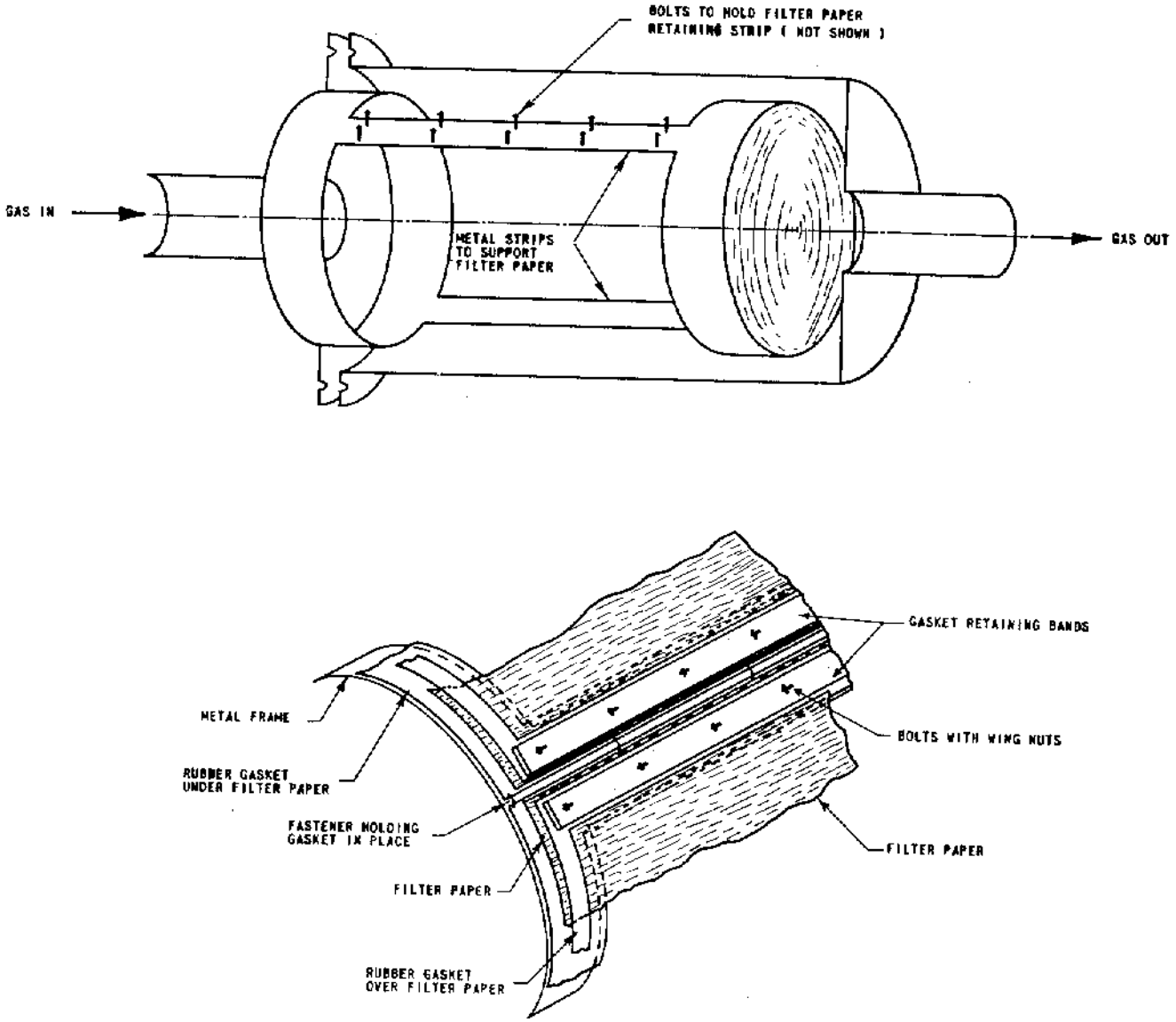

Figure 9. - Large, cylindrical, metallic, filter-paper holder.

TABLE 1. - Resistance to air flow of various filtering materials

Material	Pressure drop in inches of water at air velocity of 1 foot per second
Filter papers	
S & S	
Sharkskin	8
402	42
404	3.2
410	1.4
497	17
576	190
589-1 H	1.5
589 white ribbon	12
589 blue ribbon	57
589 green ribbon	8.2
589 black ribbon	2.9
595	5.3
596	15.2
597	11.3
598	11.8
602	58
602 extra dense	78
604	3.9
Whatman	
1	30
2	34
4	5.8
5	200
30	15
31	7.5
32	220
40	32
41	5.0
42	84
Fisher	
9-795	4.9
Reeve angel	
201	31
Orlon	
FA-1104[1/]	.8
Glass	
G-206[1/]	.7
Nylon	
NS-1209[1/]	.5
L-4086[1/]	6
Micro metallic stainless steel[2/]	
Grade D	1.4
Grade H	84
Thimbles	
Paper	
Whatman	10
S & S	24
Alundum	
5163 RA 350	630
5163 RA 360	580

1/ National Filter Media Co.

2/ Calculated from values in catalog of Micro Metallic Corp. Intermediate grades are available. Material 1/8 inches thick.