Whenever necessary, the noncondensable moisture was determined by means of a drying agent, and errors due to the absorption of other gases were investigated. Tests showed that anhydrous calcium sulfate removed much of the hydrogen sulfide and carbon oxysulfide, but the amounts present in the gas were so small that the effect on the moisture determination was negligible. Little carbon monoxide and no carbon dioxide or hydrogen were removed from the synthesis gas by this material. The completely dry desiccant does not remove H_2S from completely dry gas. Data are given in table 2.

TABLE 2. - Absorption of gases by anhydrous calcium sulfate

(10 cubic feet of gas passed in each case)

Composition of gas feed					Gain in weight, grains	Constituent removed	Percent removed
H_2, percent	CO, percent	CO_2, percent	H_2S, grains per 100 cu. ft.	COS, grains per 100 cu. ft.			
100	0	0	0	0	0	H_2	0
0	0	100	0	0	0	CO_2	0
90	0	10	0	0	0	H_2 & CO_2	0
90	0	10	0	18.5	1.111	COS	60
100	0	0	0	20	1.497	COS	75
50	50	0	0	0	2.143	CO	0.053
0	0	0	100	0	0 1/	H_2S	0
0	0	0	100	0	27.752/	H_2S	-

1/ Dry calcium sulfate.
2/ Calcium sulfate that had previously been used to remove moisture from air.

Pressures in the gasification units were usually sufficient to force the gas through the sampling system. If not, small blowers were used to exhaust the gas. Ejectors operated by steam, air, or inert gas proved satisfactory for low rates of gas flow. Stairmand gives precise dimensions for an ejector for dust sampling, and Noss[40] incorporates an ejector in the same unit with a filter thimble. Stairmand,[41] in addition, developed a portable unit for determining dust in air.

Highly Purified Gas

The problems involved in testing highly purified gases are entirely different from those encountered when determining dust in crude gases. Since the desired dust concentrations are in the same range as in atmospheric air, similar methods are applicable. However, dust-weight determinations are rarely made on air because of the difficulties mentioned below. Brown and Schrenk give a general review of this subject.[42] Exceptions are the work by Yant and coworkers on air in the Holland Tunnel[43] and the work described by Dalla Valle,[44] both of whom relied on weighing

40/ Noss, P., Messverfahren und Messgerate zur Staubgehaltbestimmung in Stromenden Gasen: Brennstoff-Wärme-Kraft, Vol. 4, No. 7, July 1952, p. 227.
41/ See work cited in footnote 12, p. 34.
42/ Brown, C. E., and Schrenk, H. H., Standard Methods for Measuring Extent of Atmospheric Pollution: Bureau of Mines Inf. Circ. 7210, May 1942, 19 pp.
43/ See work cited in footnote 39.
44/ Dalla Valle, J. M., Micromeritics: Pitman Publishing Corp., New York, N.Y., 1943, p. 360.

the deposited dust. Usually, however, the rate of dust fall from the atmosphere is measured or "dust counts" are made on samples obtained by impingment. These are much simpler but variations in dust content determined by "dust counting" are not significant in synthesis gas work.

Large Area Filter Papers

Determinations of weight concentrations in pure gases are difficult because of the large volumes that must be treated to collect enough material to weigh. Also, particle sizes are frequently small so that fine-grained high resistance filters must in such cases be used. The performance of fine filters was studied by Bangert[45] who showed that their use becomes especially difficult if a very long period is not available for sampling.

The application of radioactive tracer techniques to this problem was considered, but the probability of developing a practical method did not seem great enough to justify initiating a research project.

Thus, it became necessary to develop a filter of large area but small and light enough to be weighed on an analytical balance. As a filtering medium, filter paper fulfilled these conditions because a large area has little weight and the paper can be folded or rolled into a small volume. Furthermore, high-quality papers are readily available in a wide variety of sizes, porosities, strengths, thicknesses, and ash contents. Papers with fine pore spaces are desirable to retain the smallest possible particles, but this increases the pressure drop per unit area. Fortunately, filters remove particles much finer than the pore spaces because dust removal is not entirely a straining action. The larger particles impinge on the fibers, and smaller ones diffuse rapidly to the fibers by Brownian movement and adhere to them. Particles of 0.3 micron diameter are the most difficult to remove.[46] Thus, for best efficiency, the finest papers and lowest possible velocities are desired, which calls for large filtering areas.

The Army Chemical Corps has developed a special type of filter paper of high retentivity with small pore spaces and low pressure drop, but, unfortunately, it is unsatisfactory for this purpose because its great thickness makes it impossible to roll or fold it into a small weighing bottle. Silverman[47] used a type S unimpregnated filter made by Mine Safety Appliance Co.

Filter paper, however, has a disadvantage; it is much more hygroscopic than other filtering mediums, often absorbing 6 percent or more of its weight of moisture. This is unimportant with mineral dusts because ashless paper can be used and ignited. For this work, however, it was equally necessary to develop an accurate and reproducible method for drying and weighing the paper.

45/ Bangert, F., (Problem of Finest Filtration of Aerosols): Staub, vol. 25, 1951: pp. 158-170.

46/ Jucker, F. T., Jr., Rickard, H. B., and O'Konski, C. T., Photoelectric Instruments for Comparing the Concentrations of Very Dilute Aerosols, and Measuring Low Light Intensities: Jour. Am. Chem. Soc., Vol. 69, 1947, pp. 429-38.

47/ Silverman, Leslie, and Ville, F. J., Jr., A High-Volume Air-Sampling and Filter-Weighing Method for Certain Aerosols: Jour. Ind. Hyg. and Toxicol., Vol. 30, 1948, p. 124.

Designs for Large-Area Filter-Paper Holders

The design of a suitable holder for large-area filter papers was also a major problem. The instrument shown in figure 9 (described previously in connection with tests on crude gases) was the first practical large area filter paper holder found suitable for tests on highly purified gases. The most recent design shown in figures 10 and 11, however, was much simpler to construct. Being flat instead of cylindrical, it is less likely to leak, easier to use, and more accurate. The flow of gases through the flat filter papers is shown in figure 10, and a working drawing of a design is given on figure 11. Its important feature is that the paper is narrow enough to be rolled instead of folded into the weighing bottle before and after the test. This decreases the change in weight due to handling and the possibility of leakage caused by creases. If folding is to be eliminated the strips of filter paper can be no more than 6 inches wide, since this is the inside height of the tallest weighing bottle that can be used on an ordinary analytical balance. weighing bottles of such dimensions had been specially made for this purpose.

Several other designs, some very complicated, have been investigated, but the one described proved to be the best method for rapid determination of dust concentration in a highly purified gas stream. This design is very compact for the fast flows it can handle; the wall area in the inlet chamber was reduced to minimum. Further decrease in cross-sectional area would cause significant pressure drops. As shown in figure 11, an orifice meter is incorporated into the same unit. A blower or an ejector is readily fitted to the outlet when necessary. The pressure drop through this filter holder is only 6.3 inches of water with a flow of 6,000 cubic feet per hour, therefore, the entire gas output from small-scale gasifier units may be passed through the filter, which eliminates an important source of error - the sampling step.

Gas Under Superatmospheric Pressures

Little work has been done to date on the removal of dust and moisture from gases under pressure. The laboratory design illustrated in figure 7 has been successfully used with the arrangement shown in figure 12. The same method can probably be used with the large-area design illustrated in figure 13, although this has not yet been investigated. The Research Corp. design[48] can be used up to 100 p.s.i.

The problem is complicated further if condensable water is also present in the gas. Condensation after dust removal would probably be most practical, although a large area filter would have to be used and its temperature held within close limits. The alternative, an adaptation of the method used for low pressures, is also possible.

Thus, the separation of condensable water in the presence of large amounts of dust was accomplished in flasks or jars. Dust was sometimes removed with Soxhlet extraction thimbles. For large amounts of dust, special large-area filters were designed. For dust determination on highly purified gases, another design of large-area filter was developed.

48/ Research Corporation, Bound Brook, N. J., Tech. Bull. 3-E, August 1931.

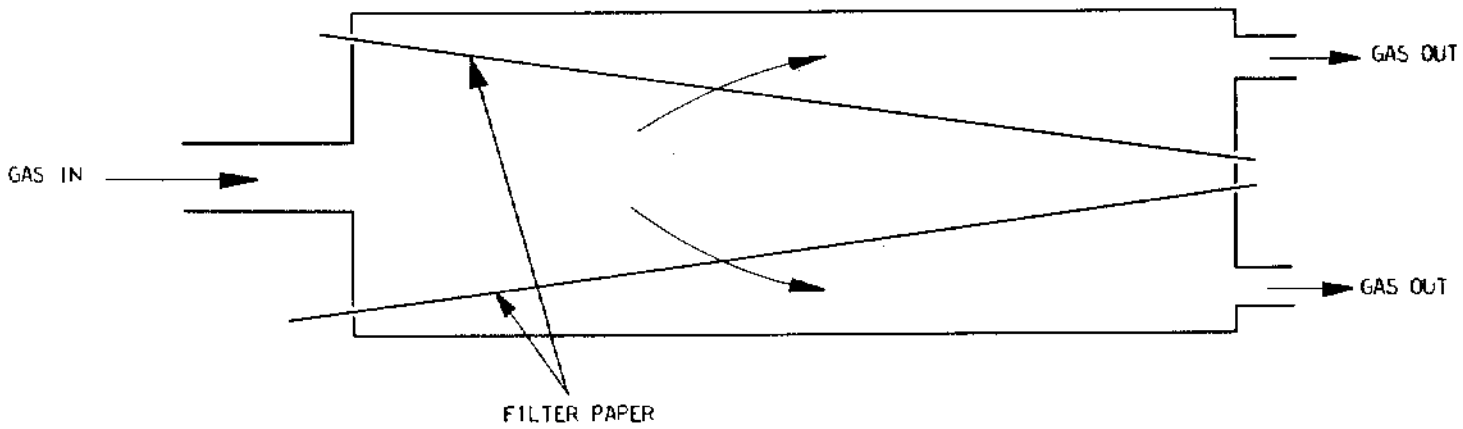

Figure 10. - Metallic holder for large-area filter-paper strips (cross-sectional diagram).

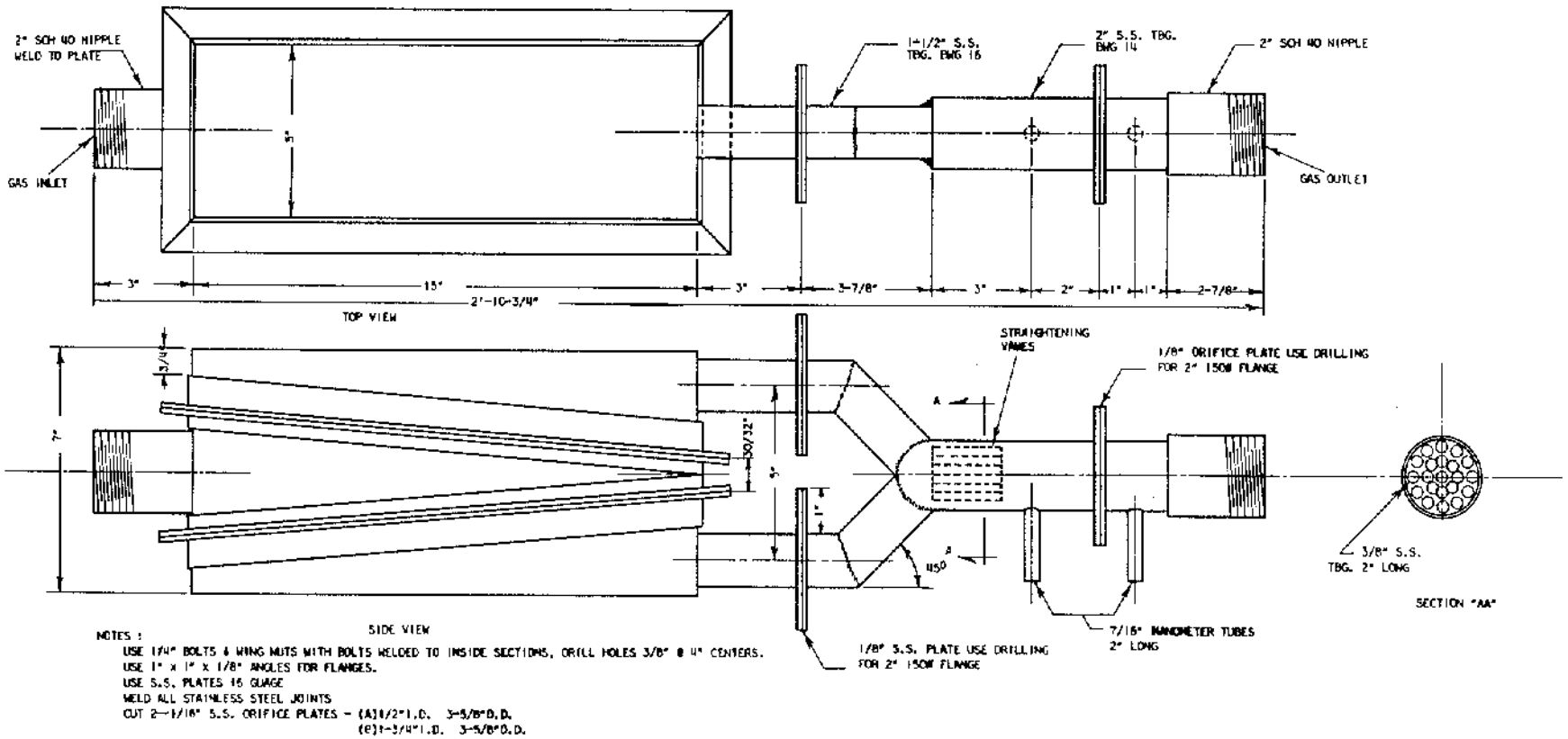

Figure 11. - Design of large-area filter-paper holder.

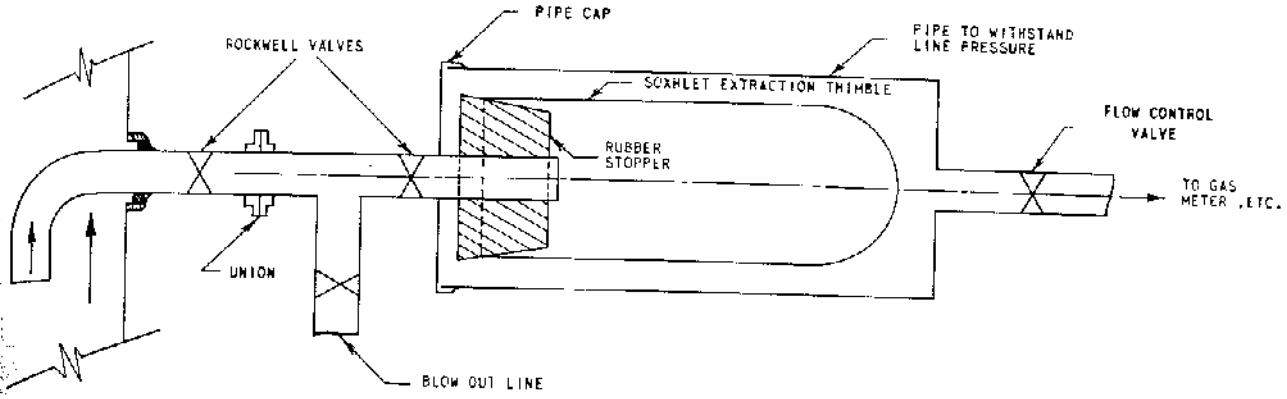

Figure 12. - Simple filter-thimble holder for high pressure.

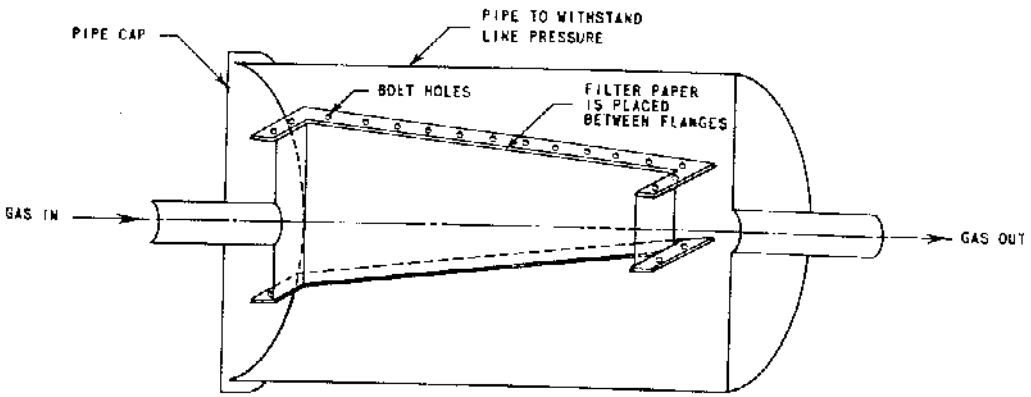

Figure 13. - Large-area filter-paper holder for high pressure.

EXAMINATION OF THE SOLID AND LIQUID IMPURITIES

The solid and liquid impurities separated from the sample gas stream by the methods previously described may be examined for many different research or control purposes to determine their quantity, particle size and size consist, chemical composition, porosity, hardness, and other properties.

The most common examination is measurement of the quantity collected to determine the concentration of these impurities in the gas stream. In industrial processes this is usually expressed as weight of impurities per unit volume of gas. The necessary weights are easily obtained if enough material is collected for accurate weighing and if the containing vessels do not change weight appreciably owing to humidity or other causes. In testing crude gases (fig. 6), moisture as well as dust concentration was determined by weighing each flask as removed from the train, containing dust and condensed moisture, and weighing the same flasks again after drying. The flasks were then washed out, dried, and weighed a third time. The glass connecting tubing was similarly treated when it contained significant amounts of dust or moisture. About 80 percent of the dust and all of the condensed moisture were obtained in these flasks. The extraction thimbles or filter papers used to collect the remaining dust were weighed before and after the tests. From these figures and the amount of gas treated the concentrations of moisture and dust in the gas were calculated. When necessary, the weights were determined with an analytical balance, but this was not generally necessary when working with crude gases.

However, when air or highly purified gases are tested, very small amounts of dust were collected and a more refined weighing technique was necessary. Little information exists in the literature on this problem as it rarely occurs in industrial practice. Determinations on air are usually made for industrial hygiene purposes with dust concentrations expressed as number of particles per unit volume.[49/50/] Weight concentrations would be misleading for this purpose, since particles greater than 10 microns are physiologically harmless.

Even when "weight concentrations" instead of particle counts are determined, precise weighing is often avoided by using less accurate but more rapid and convenient methods, such as measuring the darkening or increase in pressure drop of filter paper through which gas is passed.[51/]

Weighing Procedure

For synthesis-gas work, however, it was necessary to develop an accurate weighing procedure. Although highly erratic, the weight change due to variations in moisture content of the filter medium may amount to 6 percent, or 120 milligrams for a 25- by 80-mm. thimble having 0.05 square foot of usable filter area. With unusually wide variations in humidity the weight change may even be greater.

49/ Forbes, J. J., Davenport, S. J., and Morgis, G. G., Review of Literature on Dusts: Bureau of Mines Bull. 478, 1950, pp. 136-146.

50/ Trostel, L. J., and Frevert, H. W., Collection and Examination of Explosive Dusts: Ind. Eng. Chem., Vol. 5, 1923, p. 232.

51/ Dill, R. S., A Test Method for Air Filters: Am. Soc. Heat. and Vent. Eng., Trans., Vol. 44, 1938, p. 379-386.

See also footnote 39.

Simple drying of the thimbles in a desiccator was first attempted, but constant weights could not be obtained. Drying in an oven was much faster, but the thimbles regained much weight when cooled in the desiccator. This was overcome by leaving the lids on the weighing bottles during cooling in the desiccator and lifting them for an instant before weighing to bring the air inside to atmospheric pressure.

These experiences and results are almost identical with those described by Arbogast.[52] Silverman and others[53] have suggested the use of a gas-mask type filter unit and weighed it at 111° C. in a drying oven by placing the balance directly over the oven. Difficulties due to air convection were overcome, and an accuracy of 0.5 mg. was attained. Watson[54] describes a method for weighing filter papers 5.5 cm. in diameter with a standard error of a single weighing of 0.08 mg. However, with the method developed here, a standard deviation of 5.0 mg. was obtained under practical conditions. As shown in table 3, this error was reduced to 0.6 mg. by using a blank thimble and weighing bottle to correct for changes in weight due to atmospheric humidity, oven temperature, desiccator humidity, etc. In routine work the weighing bottle containing the blank thimble was simply put on the right hand pan of the balance, but in experimental work it was weighed separately and the proper correction applied. No method was found to reduce the weighing error further, but the precision of weighing was estimated by using two blanks and determining the difference in increase or decrease in weight. This was adopted as standard procedure for weighing paper filter thimbles.

TABLE 3. - Precision of method for dust determination

Determination No.	Change in weight on successive weighings				Difference	Difference Squared
	Thimble #1		Thimble #7			
	Mg.	Squared	Mg.	Squared		
1	2.2	4.9	2.0	4.0	0.2	0.04
2	5.4	29.2	4.4	19.4	1.0	1.00
3	1.1	1.2	.7	.5	.4	.16
4	1.3	1.7	1.6	2.6	.3	.09
5	11.8	139.2	11.2	126	.6	.36
6	9.6	92.2	8.5	72.3	1.1	1.21
7	.6	.36	.5	.25	.1	.01
8	1.0	1.0	1.1	1.21	.1	.01
9	3.0	9.0	3.7	13.7	.7	.49
10	2.7	7.3	2.3	5.3	.4	.16
11	3.7	13.7	3.1	9.6	.6	.36
		299.76		254.86	11/5.5	11/3.89
				+299.76	0.50	0.354
				22/554.62		
				25.2		

Standard deviation without correction for blank: $\sqrt{25.2} = 5.02$.

Standard deviations with correction for blank: $\sqrt{0.354} = 0.60$.

52/ Arbogast, A. E., Iron and Steel Engineer, October 1948, pp. 1-8.
53/ Silverman, Leslie, and Fredrick, J. V., Jr., A High-Volume Air-Sampling and Filter-Weighing Method for Certain Aerosols: Jour. Ind. Hyg. and Toxicol., vol. 30, 1924, p. 124.
54/ Watson, H. H., Determination of the Dry Weight of Filter Papers: Engineering, July 25, 1952, p. 102.

Weighing of Large-Area Filters

To weigh large-area (1.5-sq. ft.) filter papers, however, some changes were necessary. Constant weight was impractical - it was not attained even after 38 hours at 150° C. Therefore, blank corrections were employed except when heavy dust loadings made high precision unnecessary. By drying at 150° C. instead of 110° C., the standard deviation after the correction for change in blank weight was reduced to 2.3 mg. The latter is 4 times the error obtained in thimble weighing, but the filtering area is 30 times greater (1.5 instead of 0.05 sq. ft.), so that the weighing error per hour of sampling time is one-seventh as great.

One hour was adopted as the standard drying time in the oven, followed by a 2-hour cooling period in a desiccator used without desiccant to decrease the rate of weight change on the balance pan. Specific details of the calculations used in determining the weight concentration of dust in gas are given in the Western Precipitation Co. Booklet 50.

The above discussion is concerned only with drying and weighing errors. Weight changes due to handling of the filter paper are minimized by using chamois-skin glove fingers. All designs except those shown on figures 8 and 9 make this relatively easy.

Use of Automatic Instruments

In addition to the described test equipment for determining dust impurities, a synthetic liquid-fuel plant requires an automatic alarm and control device in the purified-gas line to prevent equipment damange due to sudden failure of the dust-removing apparatus. An instrument was investigated that determined the dust concentration in a flowing gas stream by comparing the intensity of a light beam that had passed through the gas stream with light from a similar source that had passed through a clean air path of identical length,55/ but the instrument could not be made sensitive enough to detect 0.05 grain of dust per 100 cu. ft. Illumination of the gas stream perpendicularly to the direction of flow may prove to be more satisfactory. Gibbs56/ gives equations derived for the use of the Tyndall effect with particles larger and smaller than the wave length of light.

Moisture Determination

Moisture determination in the presence of dust presented two problems. When occurring in liquid phase admixed with dust, no satisfactory means of determination was developed because of the extreme difficulty in sampling the stream, as previously stated. Determinations were occasionally made by the usual procedure, but merely to estimate the order of magnitude of the moisture concentration. On the other hand, when the moisture was present as vapor, laboratory tests showed the usual method to be very accurate.

Results of tests on the method at three widely different moisture concentrations are given in table 4. In these tests a known amount of moisture was added to the air stream by passing it through a flask of water completely submerged in a water bath. The resulting moisture concentration in the air stream was determined from the loss of weight of the vaporizer or from the vapor pressure of water at the temperature of the water bath. The moisture determination was made by the test previously described - passing the gas through three 300-ml. flasks in series in a bath of ice water and then through a bottle of Drierite (anhydrous $CaSO_4$) drying agent. The gas

55/ Stone, D. E., Kane, L. J., Corrigan, T. E., Wainwright, H. W., and Seibert, C. B., Investigation of a Photoelectric Device for the Determination of Low Concentrations of Dust: Bureau of Mines Rept. of Investigations 4782, 1951, 6 pp.

56/ See work cited in footnote 12, p. 119.

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flow was determined with a wet test meter. This table shows the accuracy of the method and the maximum flows for which the apparatus is satisfactory. It also proves that the moisture leaving the last flask can be determined from the temperature without significant error, thus eliminating the need for the drying agent.

TABLE 4. - Accuracy of test method developed for moisture determination

	Test 1	Test 2	Test 3
Gas rate.....std. cu.ft. per hr.	12	10	4.5
Vaporizer temperature.....°C.	6.6	45.5	79.1
True moisture concentration, percent, wet basis[3]:			
As determined from vaporizer weight loss.....	(1)	(1)	45.8
As determined from vaporizer temp.....	1.01	10.32	[2] 46.7
Moisture concentration as determined by increases in flask weight and drierite weight, percent, wet basis[3].....	1.06	10.32	45.7
Error percent of total gas passed.....	0.05	0.16	0.1
Error percent of true value.....	5.0	1.6	0.2
Temperature in last flask.....°C.	2.5	2.5	5
Estimated maximum possible air flow without overheating flasks..... std. cu.ft./hr.	-	30	10
Error if temp. of last flask is used instead of drierite.. .....percent of total gas passed	0.12	0.02	0.01

1/ Inaccurate because of small amounts evaporated from large vaporizer.
2/ Slightly inaccurate because of small fluctuation of vaporizer temperature.
3/ Ratio of volume of water vapor to the sum of volumes of water vapor plus dry gas.

Particle-Size Determination

In addition to the determination of dust concentration, examination of dust frequently includes the measurement of particle size or particle-size distribution (size consist). This is particularly difficult if a large range of sizes is present. Extensive literature exists on this subject.[57] Analytical screens are convenient and accurate for sizes larger than about 70 microns,[58] and numerous sedimentation and elutriation methods using air or other fluids are commonly used for smaller sizes. Although results are stated in terms of size, these methods actually separate particles into fractions. The range of sizes in each fraction is a function of particle shape, density, size, and other factors. However, regardless of the method used for size separation, particles of different masses and volumes may appear in the same fraction. Even screens ignore the longest dimension of a particle.

57/ Drinker, Philip, and Hatch, Theodore, Industrial Dust: McGraw-Hill Book Co., Inc., New York, 1936, 316 pp.
Symposium on New Methods for Particle Size Determination in the Subsieve Range: Washington Spring Meeting, Mar. 4, 1941.
British Colliery Owners Research Association and the British Coal Utilization Research Association, Report of Discussions. Determination of Particle Size in the Subsieve Range.

58/ American Society for Testing Materials, Standard D392-38, pt. 6, 1949, p. 55; E11-39, pt. 2, p. 1015; pt. 3, p. 1121; pt. 4, p. 1165; pt. 5, p. 1585; pt. 6, p. 1287; D502-39, pt. 5, 1949, p. 521.