SUMMARY AND CONCLUSIONS

Applications are described of various methods for determining the weight concentration and physical properties of solid and liquid impurities of gases in connection with problems encountered in synthesis-gas production. Of special interest among these methods may be listed:

1. A method developed for determining the weight concentration of solid and liquid impurities in hot, crude synthesis gas containing a high concentration of moisture.

2. A method developed for the same purpose but for highly purified synthesis gas or atmospheric air.

3. A method for obtaining a uniform dispersion of dry dust on a microscope slide for particle-size determination or other purposes.

The greatest difficulties and largest errors in the determination of solid and liquid impurities in industrial gas streams were usually encountered in the sampling step. This was especially true with short, small-diameter ducts, slow flows, large particles, and solid and liquid impurities occurring together. Most of these errors were due to the fact that, generally, few locations are suitable for dust-sampling points in the crude gas, particularly in small-scale plants. It is, therefore, advisable to consider this factor when plants are designed.

With highly purified gases, however, the sampling step was found to be relatively simple, but removal of the solid and liquid impurities from the sample stream, and accurate determination of the amount removed, were more difficult.

A graphical summary of methods for determining dust and moisture in gases is given in figure 1.

INTRODUCTION

This work is part of the Federal Bureau of Mines Synthetic Liquid Fuels Program, the object of which is to develop methods of producing gasoline, diesel fuel, and other oil products from nonpetroleum sources.

To obtain oil from coal by Fischer-Tropsch or related processes, it is first necessary to produce a mixture of carbon monoxide and hydrogen known as synthesis gas. The cost of gasifying coal and purifying the resultant synthesis gas constitutes a substantial part of the cost of synthetic liquid fuels (60 to 70 percent if the Fischer-Tropsch process in used);[4/] therefore, it is essential that this intermediate product be produced at the lowest possible cost.

4/ Bureau of Mines, Annual Report of the Secretary of the Interior on Synthetic Liquid Fuels, 1950: Pt. I - Oil from Coal: Rept. of Investigations 4770, 1950, p. vi.

The use of coal instead of the more expensive coke as raw material makes possible a substantial reduction in cost but gives a highly contaminated synthesis gas, which presents unusual problems in the determination and removal of sulfur compounds, dust, and other impurities. These difficulties are further increased by the extreme gas purity required by the synthesis catalyst and the fact that the gas must be dust-free when handled in compressors, fixed beds, and other equipment in the system. Much work has been done on the determining and removing various sulfur compounds from the gas.5/6/7/8/

The maximum permissible dust concentration has been set at 0.05 grain per 100 cubic feet of gas,9/ based on European results.

The establishment of satisfactory methods for dust removal at low cost may prove to be one of the most difficult phases of the entire synthetic liquid fuels program.10/ The Locomotive Development Committee found it to be the most difficult problem in building a coal-burning gas turbine.

Investigations have been underway for several years to evolve the best possible methods for determining and removing various impurities from synthesis gas. This paper describes investigations carried out to develop practical, reliable, and accurate methods of determining solid and liquid impurities under any condition that may occur in synthesis gas production.

ACKNOWLEDGMENTS

As the work here summarized covers several projects and long-range development, adequate acknowledgment to all who have contributed in some way to this work is difficult.

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At the Bureau of Mines Demonstration Plant in Louisiana, Mo., H. R. Batchelder and H. A. Ingols were especially helpful. Special thanks are due to J. E. Anderson, T. B. Kirby, B. C. Parks, and others at the Central Experimental Station in Pittsburgh, Pa.

5/ Sands, A. E., Schmidt, L. D., Recovery of Sulfur From Synthesis Gas: Ind. Eng. Chem., Vol. 42, 1950, pp. 2277-2287.

6/ Sands, A. E., Grafius, M. A., Wainwright, H. W., and Wilson, M. W., The Determination of Low Concentrations of Hydrogen Sulfide in Gas by the Methylene Blue Method: Bureau of Mines Rept. of Investigations 4547, 1949, 19 pp.

7/ Sands, A. E., Wainwright, H. W., and Egleson, G. C., Organic Sulfur in Synthesis Gas; Occurrence, Determination, and Removal: Bureau of Mines Rept. of Investigations 4699, 1950, 51 pp.

8/ Wainwright, H. W., and Lambert, G. I., A Colorimetric Method for the Determination of Thiophene in Synthesis Gas: Bureau of Mines Rept. of Investigations 4753, 1950, 11 pp.

9/ Sands, A. E., Wainwright, H. W., and Schmidt, L. D., Purification of Synthesis Gas Produced from Pulverized Coal: Ind. Eng. Chem., Vol. 40, 1948, p. 608.

10/ See work cited in footnote 9, p. 608-609.

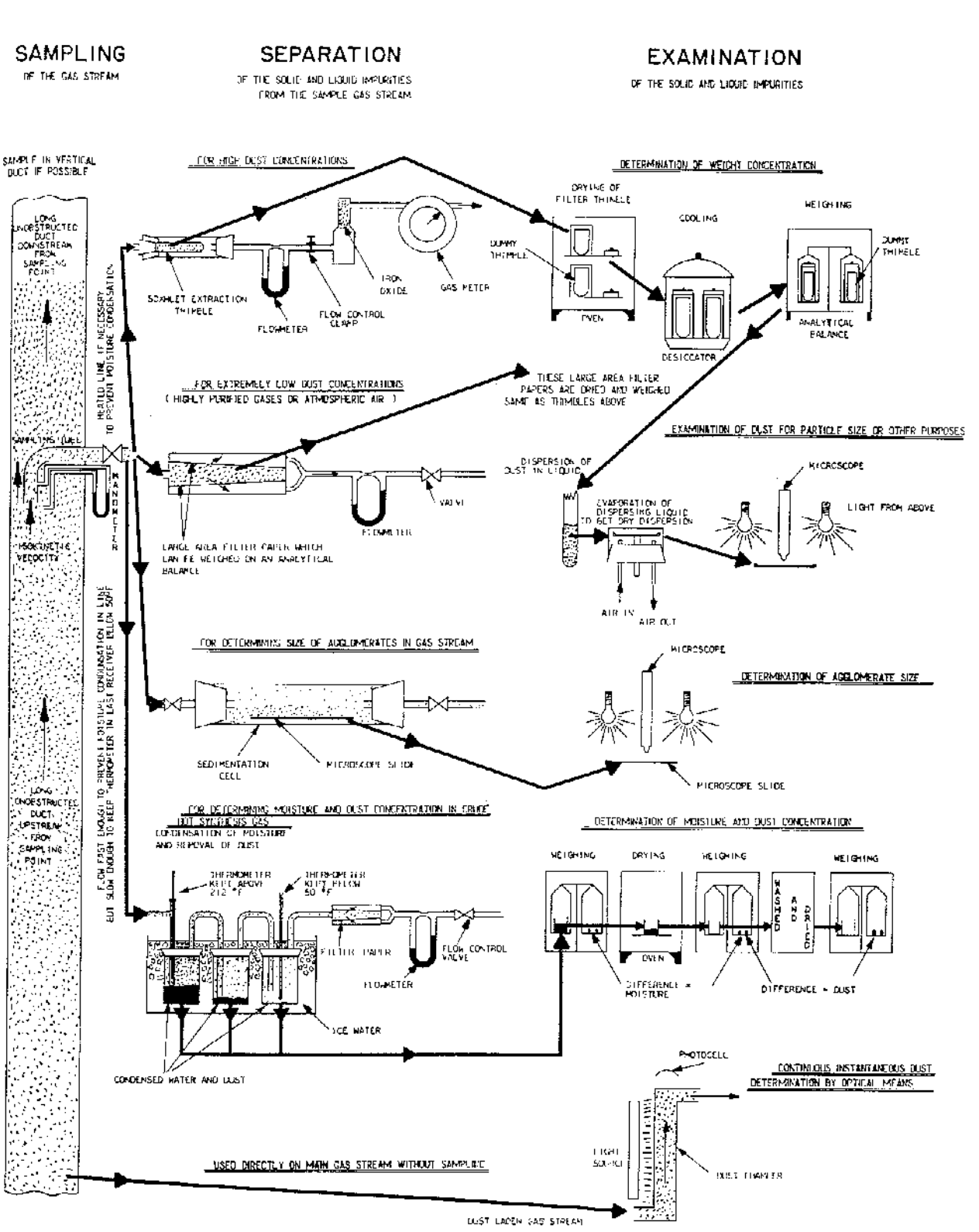

Figure 1. - Summary of methods suitable for determining dust and moisture in synthesis gas.

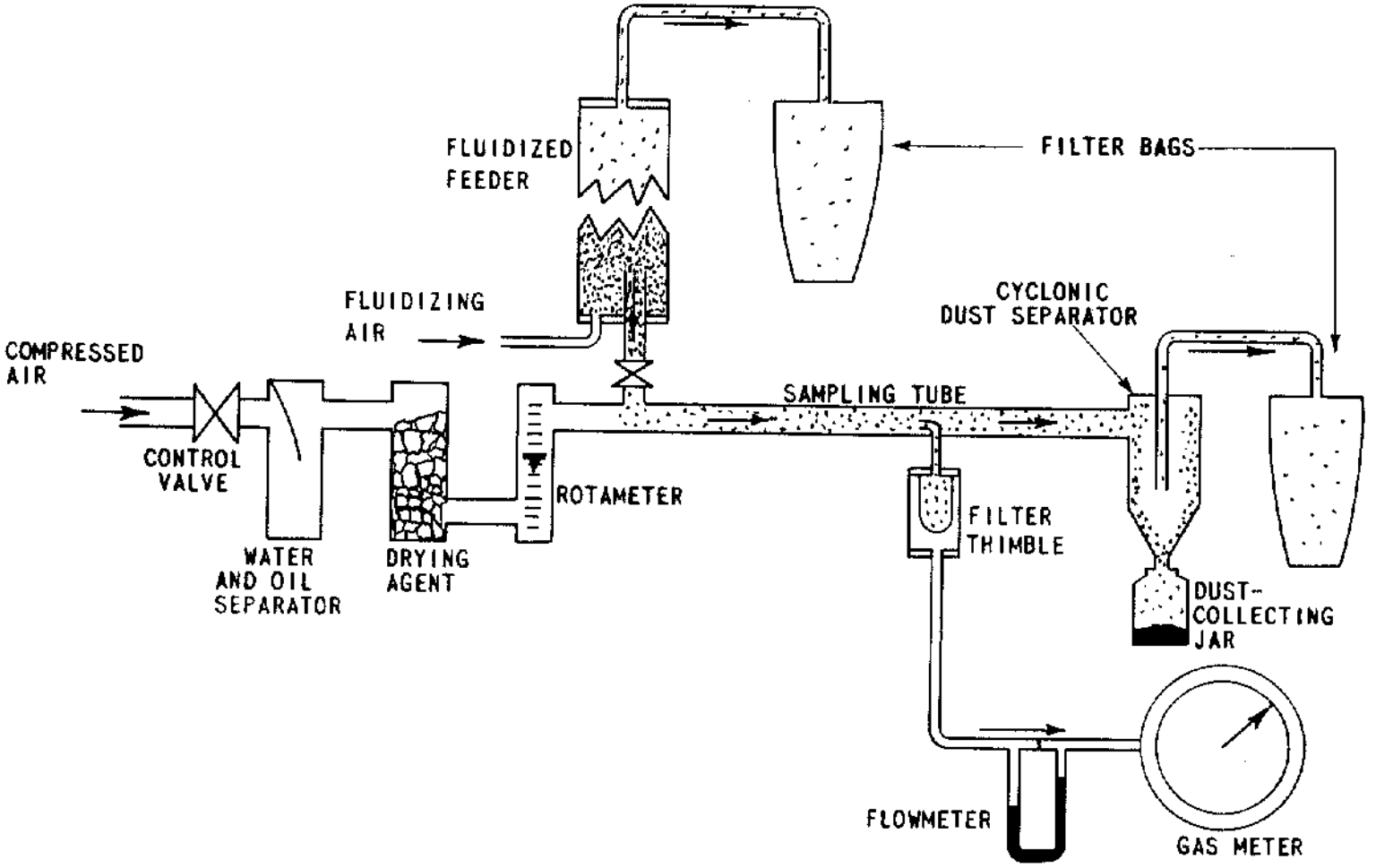

Figure 2. - Apparatus for testing dust-sampling methods.

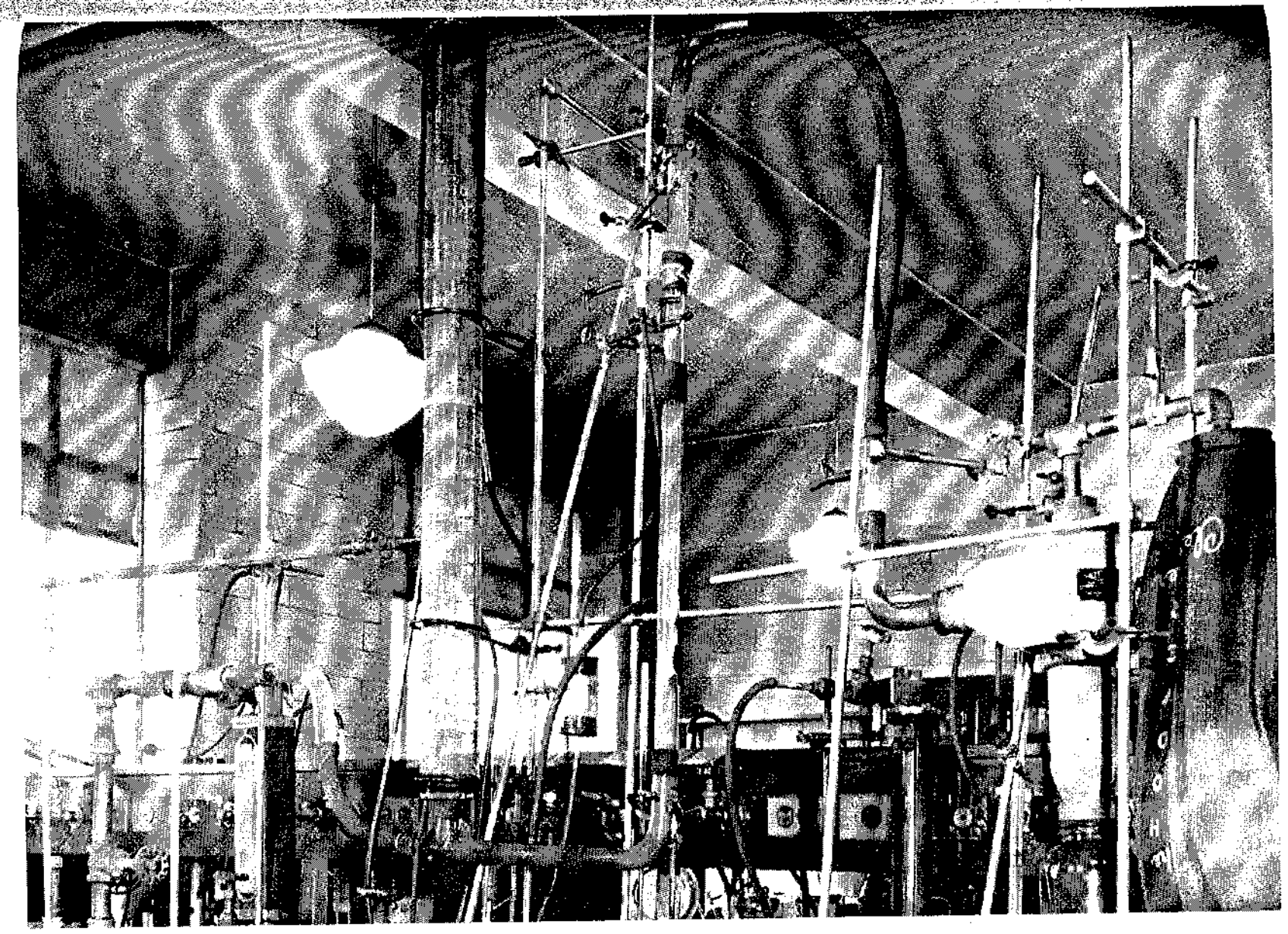

Figure 3. - Photograph of apparatus for testing dust-sampling methods.

SAMPLING THE GAS STREAM

The subject of dust determination was studied in two ways - with specially designed laboratory apparatus and by means of practical tests made on pilot-plant gas streams.

Laboratory-Scale Equipment for Testing Sampling Methods

Figure 2 shows schematically the apparatus as used for tests on a horizontal duct, and figure 3 is a photograph of the same equipment arranged for tests on a vertical duct. The apparatus consisted of an arrangement for imparting any desired dust concentration to a stream of air. After being purified and dried the air stream was measured with a rotameter and dust added (as shown) from a vessel in which coal, coke, or other material was fluidized.

The dust-laden air stream was then sampled under various conditions, and dust determinations were made using the filter thimble, flowmeter, and wet test meter. The results were compared with the true dust concentration calculated from the flow rate of the main gas stream and weight of dust recovered in the cyclonic dust separator and filter bag.

Considerable difficulty was experienced in developing a suitable method for feeding the dust into the gas stream. Originally a hopper with a screw clamp for flow control was used, but the dust flow was erratic. It was found that fluidizing the dust in the feed pipe below the hopper improved results considerably, but later the dust in the hopper was fluidized as shown. This produced an apparently uniform flow in the feed tube; but complete uniformity in the air stream itself was never attained, although many methods were tried.

Procedure

In all, 47 runs were made using cracking catalyst and coal as well as dusts of different types and sizes, which had been separated from pilot-plant gases. The effects of numerous variables were studied, including position of sampling tube, distance of sampling tube from the duct wall and from upstream and downstream obstructions in horizontal and vertical ducts. Sampling errors varied from 1 percent to 90 percent, depending on the experimental conditions. The data obtained and conclusions drawn are summarized in the following sections.

Selection of Sampling Point

The results showed that errors are very large unless proper distribution of gas velocity and uniform concentration of dust across the duct exist at the sampling point. In practice, this is done most often by choosing a section for sampling, which is preceded by considerable straight pipe without restrictions, bends, or any other obstruction that might cause irregular flow and consequent stratification of dust. Recommendations of various authors range from 5 to 38 diameters of straight pipe length ahead of the sampling point. Experimental results, however, showed that general recommendations of this type cannot be made. The type of obstruction, the particle size of dust, and other variables have very great effect. Although usually neglected in the literature, downstream as well as upstream obstructions, were found to have significant effect on the results. Another objection to the custom of relying on a specified number of diameters of straight pipe length is that obstructions are not the only cause of poor sampling points, as explained later. Moreover, plants cannot always be designed to give the required length of straight pipe.

A common method for investigating the adequacy of the cross section selected for sampling is to determine the velocity contours by passing a pitot tube along several diameters. The isovelocity contour lines should be circular and the maximum velocity should be in the center. Drew[11] gives an example of such a test and shows how irregular such contour lines can be even after 20 diameters of straight pipe length. Yet, good velocity contour lines do not prove uniform distribution of dust. Prof. W. E. Gibbs[12] suggests a method for visually observing dust flow if the gas is nearly saturated with water vapor, but the only reliable method of universal applicability is to take dust samples at numerous points across the section to learn the variation in dust concentration. This is tedious but may be well worthwhile if many determinations are to be made at the section concerned. As suggested by Fritton and Sayles,[13] it may even be possible to find a point of mean concentration to eliminate the need for sampling at numerous points every time a determination is made. However, the position of the point of mean concentration is likely to vary if the type or particle size of the dust is changed and probably will be completely disturbed near an obstruction.

Poor isoconcentration contours are not always due to insufficient lengths of straight pipe. Gas velocities may be so low as to cause segregation of the larger particles in the stream. This may have little effect on the result if dust counts are desired because the large particles are relatively few in number but will cause great errors if the weight concentration of dust in the gas stream is desired, since most of the weight is in the larger sizes. In the latter case results may be improved by using smaller diameter ducts or by selecting vertical runs, although White[14] states that solid particles tend to move gradually to the peripheries in vertical as well as horizontal ducts.

Straightening vanes or baffles will improve results. Fritton and Sayles,[15] for example, found that a nozzle restricting half the area gave a fairly good distribution 6 diameters downstream. Stairmand[16] found mixing baffles satisfactory 3 to 6 diameters downstream, the pressure drop being 5 velocity heads. Evans and Sarjant[17] used a calming section containing screens to obtain normal turbulent flow without superimposed flow patterns, but this might not be suitable for dust-laden gases.

Thus, selection of the sampling point was found to be the most important factor in satisfactory dust determination. It should preferably be in the center of a long vertical run in which the gas velocity is high. No definite length of run can be confidently specified as satisfactory in all cases. The adequacy of the sampling

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11/ Drew, W. N., Gas Volume and Dust Concentration Determination in Connection with the Cottrell Process: Jour. Am. Soc. Mech. Eng., Vol. 37, 1915, p. 677.

12/ Gibbs, W. E., Clouds and Smokes: P. Blakiston's Son & Co., Philadelphia, Pa., 1924, p. 119.

13/ Fritton, A., and Sayles, C. P., The Collection of a Representative Flue-Dust Sample: Engineering, Vol. 173, No. 4491, Feb. 22, 1952, pp. 224-230; Vol. 173, No. 4492, Feb. 29, 1952, pp. 261-263.

14/ White, A. H., Technical Gas and Fuel Analysis: McGraw-Hill Book Co., Inc., New York, 1920, p. 138.

15/ See footnote 13.

16/ Stairmand, C. J., The Sampling of Dust-Laden Gases: Trans. Inst. Chem. Eng., Vol. 29, No. 1, 1951, p. 15.

17/ Evans, S. I., and Sarjant, R. S., Heat Transfer and Turbulence in Gases Flowing Inside Tubes: Jour. Inst. Fuel, Vol. 24, No. 139, September 1951, p. 218.

point can be ascertained by determining velocity contours or, preferably, dust-concentration contours. Some investigations have found that straightening vanes or baffles improve results. It was found that the proper points for sampling are especially difficult to find in pilot plants because of short runs and low velocities.

Sampling Methods

For accurate results, especially when stratification is suspected, samples for dust determination should be taken at several points that are centers of equal areas at the selected cross section of the duct. These samples need not be handled separately as when obtaining dust-concentration contours. A faster procedure is to make traverses along several diameters at the selected cross section. At every point, however, the velocity in the sampling tube should equal that in the duct at the particular point being sampled at the time. British standard specifications[18] call for a minimum of 24 sampling points. Stairmand[19] gives design details for sampling tubes.

The equality of gas velocities in sampling tube and duct is extremely important.[20] Hardie[21] presents a graph of the results of 3 other independent investigators (Fahrbach, Zimmerman, and Caldwell) which shows that a 10-percent error in velocity causes a 6-percent error in dust-concentration determination and a 20-percent error causes a 10- or 15-percent error.

Identical velocity is usually obtained by increasing the flow in the sampling line until the static pressure inside the sampling tube becomes equal to that in the duct at the same cross section. This causes the velocity pressure head (and hence velocity) inside and outside the sampling tube to be equal, because the total pressure is the same along any one cross section of the duct. The velocity at any one point, of course, will vary with the distance from the duct wall and should be at a maximum in the center if a good sampling section has been chosen. A variety of sampling tubes for this purpose is shown in ASME Power Test Code 21-1941.

The method discussed cannot be used if the gas velocity in the pipe is very low, because the static pressure becomes so close to the total pressure that the difference cannot be determined with sufficient accuracy. A total flow method is then used. The rate of sampling is regulated until

$$f = F \frac{a}{A} ,$$

in which f is the rate of flow in the sampling tube, F is the rate in the pipe, a is the cross-sectional area of the sampling tube, and A the cross-sectional area of the pipe in any consistent units.

If this method is used, the point of mean velocity in the pipe should be used as sampling point. Thus, the method is unreliable if any stratification exists,

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18/ British Standard Specifications No. 893: The Methods of Testing Extraction Plants and the Emission of Solids from Chimneys of Electric Power Stations, British Standards Institution, 128 Victoria St., London S.W., England, April 1940, p. 10.

19/ See work cited in footnote 16, p. 20.

20/ Brady, Williams, and Touzalin, L. A., The Determination of Dust in Blast-Furnace Gas: Jour. Ind. Eng. Chem., September 1911, pp. 662-670.

21/ Hardie, P. H., Résumé of Methods for the Measurement of Flue Dust: Trans. Am. Soc. Mech. Eng., Vol. 59, 1937, p. 355.