Numerous other methods are available. The American Society for Testing Materials has found air permeability satisfactory for testing the finest portland cement dust for size consist and has set up a standard method.[59] For routine tests on flue dust, the Roller analyzer, 325-mesh screen or oil elutriation are commonly used. Screens and air elutriation were satisfactorily applied to this work.[60] Comprehensive discussions of the various methods for particle-size analysis are given by Ivey,[61] Bangham[62] and Skinner.[63]

In a general way, the methods may be divided into two classes: Those suitable for practical engineering and control work, which are rapid and convenient but often inaccurate or unreliable, and methods that are accurate but lengthy and tedious. The latter classification includes particle-size determination by microscopic examination.[64] This is probably the most accurate method[65] and has the additional advantage of giving the true sizes of the particles instead of fictitious sizes interpreted from some law. The errors of such interpretation increase as the particle shapes deviate from spheres. The microscope can be used for any size down to about 2 microns and the electron microscope for smaller particles. Good sampling, always difficult, is especially important in this case because of the small samples examined. Each method of collection presents different problems. For example, filters imbed some particles so that they cannot be removed for examination, whereas impingers may break up the larger particles or may collect certain particles selectively because of density, size, adhesiveness, or other properties. Following the collection of samples, their reduction to a suitable representative quantity for microscopic examination presents other problems. Mixing is an important factor in this operation, yet several procedures that are intended to mix the particles may actually segregate them.

Dispersion

A further problem is separation of the individual particles from each other to determine their true sizes. Fine particles tend to agglomerate owing to electrical or other effects and complete dispersion without attrition of the larger particles is difficult. Dispersion for microscopic or electron microscopic examination is often achieved by mixing the dust with turpentine or a viscous liquid, such as glycerine, and rubbing it on the microscope slide with a glass rod.[66] Considerable experience

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59/ American Society for Testing Materials, designation C-204-46T, pt. 3, 1949, p. 103.
60/ See footnote 23.
61/ Ivey, F. E., Jr., The Particle-Size Analysis of Fluidized Cracking Catalyst: Petrol. Refiner, vol. 30, 1951, pp. 127-132.
62/ Bangham, D. H., Progress in Coal Science: Butterworth Sci. Pub., London, 1950.
63/ Skinner, D. G., and others, Determination of Particle Size in the Sub-Sieve Range: British Colliery Owners Research Assoc., General Buildings, Aldwych, London, W.E. 2.
64/ American Society for Testing Materials, Tentative Recommended Practice for Analysis by Microscopical Methods for Particle Size Distribution of Particulate Substances of Sub-Sieve Sizes: Designation E-20-48T, pt. 3, 1949, p. 1250; pt. 4, p. 1200; pt. 5, p. 1570; and pt. 6, p. 1294.
65/ Hawksley, P.G.W., The Physics of Particle-Size Measurement, Part I, Fluid Dynamics and the Stokes Diameter: British Coal Utilization Research Assoc., Monthly Bull. 15, No. 4, April 1951, p. 106.
66/ Green, Henry, A Photographic Method for the Determination of Particle Size of Paint and Rubber Pigments: Jour. Franklin Inst., vol. 192, 1921, p. 637.

is required to break up all the agglomerates without appreciable attrition of the particles, and a thoroughly satisfactory uniform dispersion, which is necessary if a large variety of particle sizes is present, is extremely difficult to obtain.

Some dusts disperse well in specific liquids. Absolute alcohol is commonly used for this purpose but is unsuitable for dry dispersions because it usually picks up enough moisture from the air during drying to cause agglomeration in the last stages. Webb states that isopropyl alcohol is the best dispersing agent for certain cracking catalysts.67/ Experiments at this station showed redistilled benzene to be suitable for coal and pilot-plant residues. It is probably best to try several liquids and use the one that gives the smallest particle size, assuming this to prove complete dispersion. If dry dispersions are used, no such assumption is necessary because it is possible to examine the particles with light from above to detect any agglomeration. O'Brien68/ dispersed with water but found considerable agglomeration on drying. He found wetting agents to be unsatisfactory. O'Brien also described an interesting electrostatic dispersion apparatus. Dispersion by milling into pale crepe rubber and adding xylene has been suggested69/ as a standard method.

For particle counts in industrial hygiene work, an alcohol suspension of the dust is placed in a covered cell 1 mm. deep on the microscope slide and permitted to settle for a specified time then viewed under the microscope. As the objective lens must remain some distance from the dust layer, low powers must be used.

Particle-size determinations usually are made on dry dispersions, although no completely satisfactory method for obtaining them is known. Samples for any work under the electron microscope, of course must be dry. By the usual methods a large number of dry dispersions are prepared and the best is selected by visual comparison. A satisfactory dry dispersion cannot be made by merely removing the cover glass to evaporate the liquid from a suspension because agglomeration occurs almost immediately.

After considerable experimentation the device shown in figure 14 was developed. The dispersion in liquid is placed in a circular glass dish of flat bottom made by sealing a 1/2-inch-long section of 2-inch glass tubing to a thin circular glass plate. The seat for the dish was made from 1/2-inch copper tubing cut along its length and flared into 4 narrow flat strips, as shown on the drawing. The liquid from the dispersion is evaporated by air circulated between the dish and glass cover at a controlled rate of flow.

A typical dispersion obtained by this method is shown at two different magifications in figures 15 and 16. Figure 17 shows a slight agglomeration of very fine particles. Serious agglomeration, however, is due to convection in the liquid caused by drafts and eddy currents in the air above, which, in turn, are induced by evaporation of the liquid, occurring at specific spots equally spaced across the area. This was eliminated by placing a solid surface closely above the liquid surface, as shown in figure 14. Also, the meniscus causes a concentration of particles a few millimeters from the side of the dish, as seen in figure 16. This effect was eliminated by increasing the diameter of the dish until it no longer appeared in the central section where determinations were made. It is important that the bottom of the dish be perfectly flat and level.

67/ Webb, G. M., Particle Size Distribution of Cracking Catalysts: Petrol. Proc., vol. 2, July 1947, pp. 497-8, 500-502.
68/ O'Brien, H. C., Jr., Pigment Dispersion Methods for Electro Microscopy: Jour. Appl. Phys., vol. 16, 1945, pp. 370-372.
69/ American Society for Testing Materials, Proposed Method of Measurement of Average Particle Diameter of Subsieve Particulated Substances by Dark Field Methods: Proc., vol. 35, pt. 1, 1935, pp. 505-507.

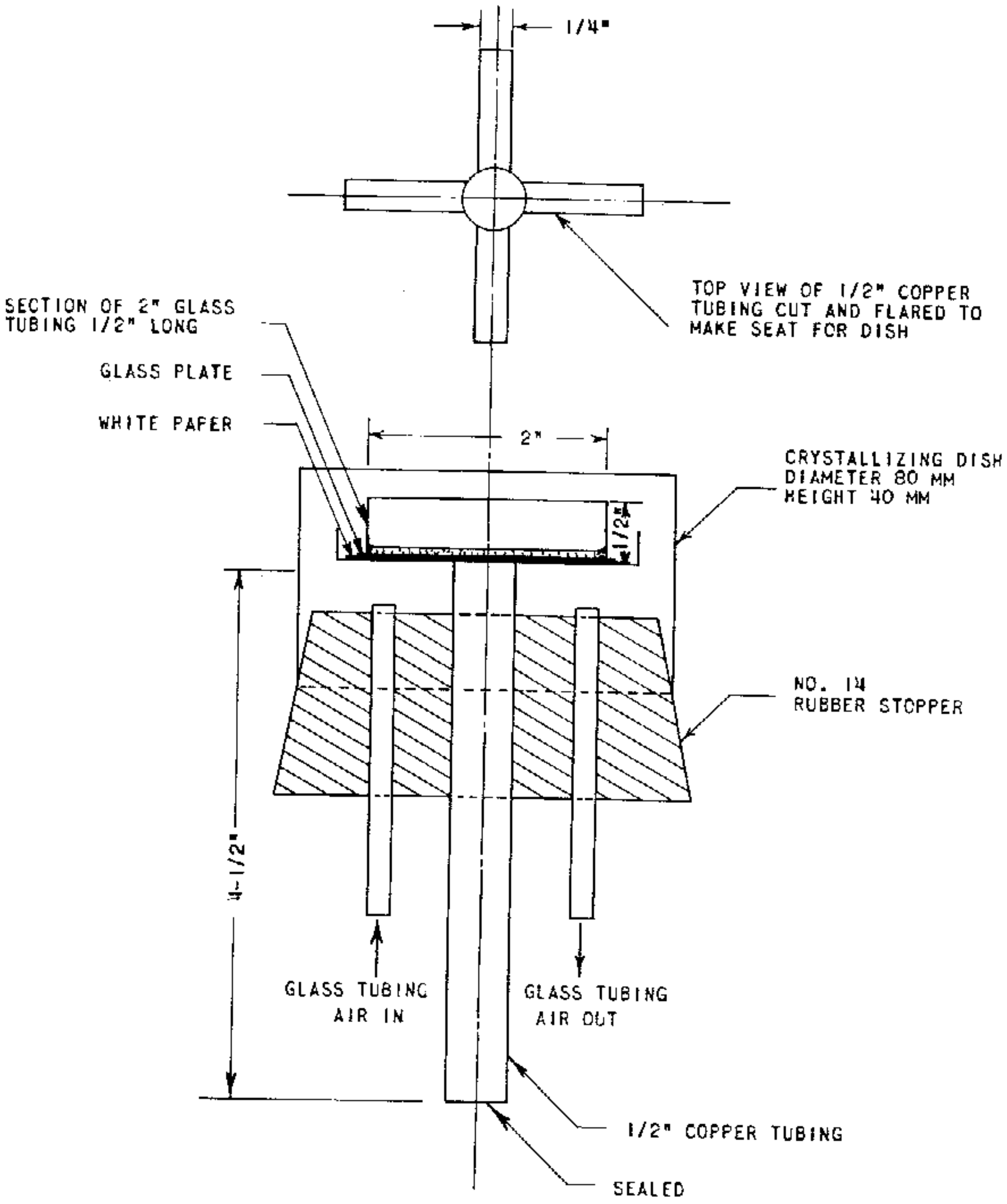

Figure 14. - Apparatus for preparation of dry dust dispersions.

Figure 15. - Typical dust dispersion.

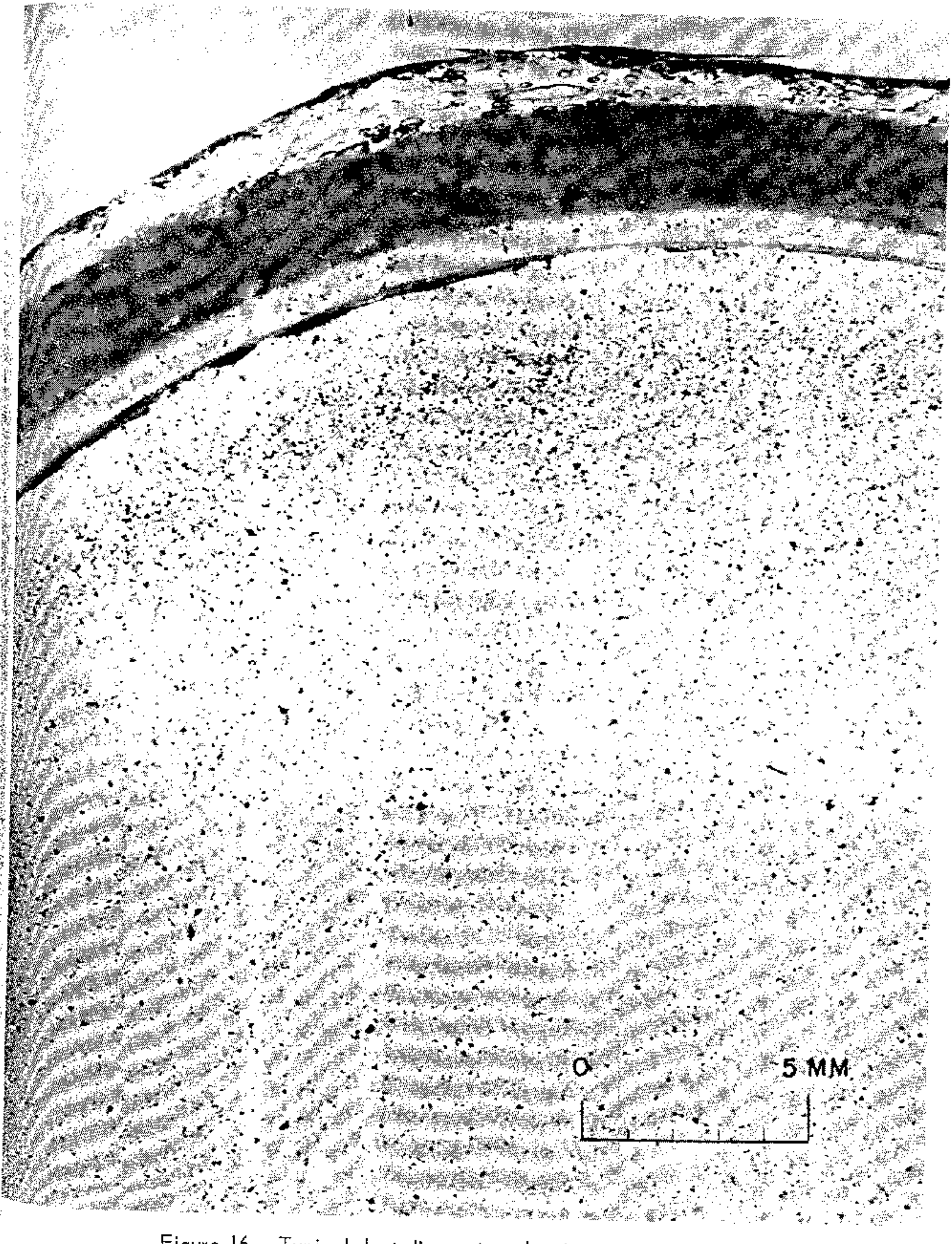

Figure 16. - Typical dust dispersion showing meniscus effect.

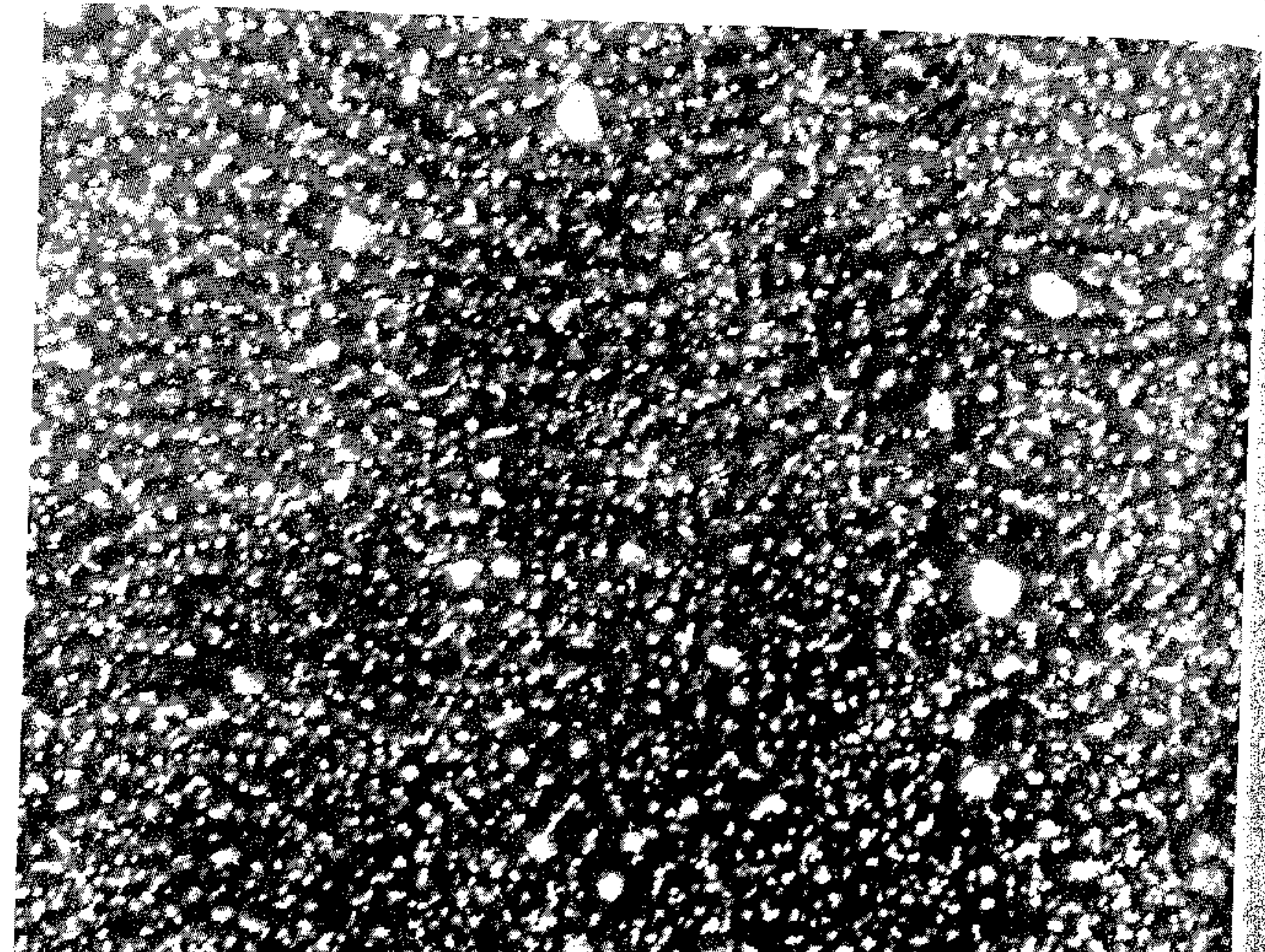

Figure 17. - Dispersion of fine particles showing agglomeration.