Designation: C 457 – 06

Standard Test Method for Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete[1]

This standard is issued under the fixed designation C 457; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes procedures for microscopical determinations of the air content of hardened concrete and of the specific surface, void frequency, spacing factor, and paste-air ratio of the air-void system in hardened concrete (**1**).[2] Two procedures are described:

1.1.1 *Procedure A,* the linear-traverse method (**2, 3**).

1.1.2 *Procedure B,* the modified point-count method (**3, 4, 5, 6**).

1.2 This test method is based on prescribed procedures that are applied to sawed and lapped sections of specimens of concrete from the field or laboratory.

1.3 It is intended to outline the principles of this test method and to establish standards for its adequate performance but not to describe in detail all the possible variations that might be used to accomplish the objectives of this test method.

1.4 The values stated in SI units are to be regarded as the standard. The values in parentheses are provided for information purposes only.

1.5 *This standard does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements see 8.3 and 10.1.

2. Referenced Documents

2.1 *ASTM Standards:* [3]

C 42/C 42M Test Method for Obtaining and Testing Drilled Cores and Sawed Beams of Concrete

C 138/C 138M Test Method for Density (Unit Weight), Yield, and Air Content (Gravimetric) of Concrete

C 173/C 173M Test Method for Air Content of Freshly Mixed Concrete by the Volumetric Method

C 231 Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials

C 823 Practice for Examination and Sampling of Hardened Concrete in Constructions

C 856 Practice for Petrographic Examination of Hardened Concrete

D 92 Test Method for Flash and Fire Points by Cleveland Open Cup Tester

2.2 *American Concrete Institute Standards:*

201.2R Guide to Durable Concrete[4]

211.1 Recommended Practice for Selecting Proportions for Normal, Heavyweight, and Mass Concrete[4]

3. Terminology

3.1 *Definitions:*

3.1.1 *air content (A)*—The proportion of the total volume of the concrete that is air voids; expressed as percentage by volume.

3.1.2 *air void*—A space enclosed by the cement paste and that was filled with air or other gas prior to the setting of the paste.

3.1.2.1 *Discussion*—This term does not refer to voids of submicroscopical dimensions, such as the porosity inherent to the hardened-cement paste. Air voids are usually larger than a few micrometers in diameter. The term includes both entrapped and entrained voids.

3.1.3 *average chord length* ($\bar{l}$)—The average length of the chords formed by the transection of the voids by the line of traverse; the unit is a length.

3.1.4 *paste-air ratio (p/A)*—The ratio of the volume of hardened cement paste to the volume of the air voids in the concrete.

3.1.5 *paste content (p)*—The proportion of the total volume of the concrete that is hardened cement paste expressed as percentage by volume.

[1] This test method is under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.65 on Petrography.

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[2] The boldface numbers in parentheses refer to the list of references at the end of this test method.

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[4] American Concrete Institute of Concrete Practice, issued annually, available from American Concrete Institute (ACI), P.O. Box 9094, Farmington Hills, MI 48333.

3.1.5.1 *Discussion*—When this parameter is calculated, it is the sum of the proportional volumes of the cement, the net mixing water (including the liquid portions of any chemical admixtures), and any mineral admixtures present **(7, 8)**.

3.1.6 *spacing factor* ($\bar{L}$)—A parameter related to the maximum distance in the cement paste from the periphery of an air void, the unit is a length.

3.1.7 *specific surface* (α)—The surface area of the air voids divided by their volume, expressed in compatible units so that the unit of specific surface is a reciprocal length.

3.1.8 *void frequency* , *n*—Voids per unit length of traverse; the number of air voids intercepted by a traverse line divided by the length of that line; the unit is a reciprocal length.

3.1.8.1 *Discussion*—The value for void frequency *(n)* cannot be directly determined by the paste-air ratio method as this value refers to the voids per unit measure of traverse in the total concrete (including aggregate).

3.1.9 *water void*—A space enclosed by the cement paste that was occupied by water at the time of setting and frequently found under an aggregate particle or reinforcing bar. A water-void is usually identified by its irregular shape or evidence that a channel or cavity has been created by bleed water trapped in the concrete at the time it hardened.

4. Summary of Test Method

4.1 *Procedure A, Linear-Traverse Method*—This procedure consists of the determination of the volumetric composition of the concrete by summing the distances traversed across a given component along a series of regularly spaced lines in one or more planes intersecting the sample. The data gathered are the total length traversed (T_t), the length traversed through air voids (T_a), the length traversed through paste (T_p), and the number of air voids intersected by the traverse line *(N)*. These data are used to calculate the air content and various parameters of the air-void system. If only the air content is desired, only T_a and T_t need be determined.

4.2 *Procedure B, Modified Point-Count Method*—This procedure consists of the determination of the volumetric composition of the concrete by observation of the frequency with which areas of a given component coincide with a regular grid system of points at which stops are made to enable the determinations of composition. These points may be in one or more planes intersecting the sample. The data gathered are the linear distance between stops along the traverse *(I),* the total number of stops (S_t), the number of stops in air voids (S_a), the number of stops in paste (S_p), and the number of air voids *(N)* intersected by the line of traverse over which the component data is gathered. From these data the air content and various parameters of the air-void system are calculated. If only the air content is desired, only S_a and S_t need be determined.

4.3 *Paste-Air Ratio Modification*—In some instances the sample is not representative of the concrete as a whole, so T_t and S_t lose their significance and cannot be used as a basis for calculations. The most common examples are concrete with large coarse aggregate and samples from the finished surface region, for both of which the examined sample consists of a disproportionately large amount of the mortar fraction. In such instances the usual procedure must be changed, and the paste-air ratio modification must be used (see 5.7).

5. Significance and Use

5.1 The parameters of the air-void system of hardened concrete determined by the procedures described in this test method are related to the susceptibility of the cement paste portion of the concrete to damage by freezing and thawing. Hence, this test method can be used to develop data to estimate the likelihood of frost damage to concrete or to explain why it has occurred. The test method can also be used as an adjunct to the development of products or procedures intended to enhance the frost resistance of concrete **(1)**.

5.2 Values for parameters of the air-void system can be obtained by either of the procedures described in this test method.

5.3 No provision is made for distinguishing among entrapped air voids, entrained air voids, and water voids. Any such distinction is arbitrary, because the various types of voids intergrade in size, shape, and other characteristics. Reports that do make such a distinction typically define entrapped air voids as being larger than 1 mm in at least one dimension being irregular in shape, or both. The honey-combing that is a consequence of the failure to compact the concrete properly is one type of entrapped air void **(9, 10)**.

5.4 Water voids are cavities that were filled with water at the time of setting of the concrete. They are significant only in mixtures that contained excessive mixing water or in which pronounced bleeding and settlement occurred. They are most common beneath horizontal reinforcing bars, pieces of coarse aggregate and as channelways along their sides. They occur also immediately below surfaces that were compacted by finishing operations before the completion of bleeding.

5.5 For air-entrained concrete designed in accordance with ACI 201.2R and ACI 211.1, the paste-air ratio *(p/A)* is usually in the range 4 to 10, the specific surface (α) is usually in the range 24 to 43 mm^{-1}(600 to 1100 $in.^{-1}$), and the spacing factor ($\bar{L}$) is usually in the range 0.1 to 0.2 mm (0.004 to 0.008 in.).

5.6 The air-void content determined in accordance with this test method usually agrees closely with the value determined on the fresh concrete in accordance with Test Methods C 138, C 173, or C 231 **(11)**. However, significant differences may be observed if the sample of fresh concrete is consolidated to a different degree than the sample later examined microscopically. For concrete with a relatively high air content (usually over 7.5 %), the value determined microscopically may be higher by one or more percentage points than that determined by Test Method C 231.

5.7 Application of the paste-air ratio procedure is necessary when the concrete includes large nominal maximum size aggregate, such as 50 mm (2 in.) or more. Prepared sections of such concrete should include a maximum of the mortar fraction, so as to increase the number of counts on air voids or traverse across them. The ratio of the volume of aggregate to the volume of paste in the original mix must be accurately known or estimated to permit the calculation of the air-void systems parameters from the microscopically determined paste-air ratio.

5.8 Of the parameters determined with this test method, the spacing factor ($\bar{L}$) is generally regarded as the most significant indicator of the durability of the cement paste matrix to freezing and thawing exposure of the concrete. The maximum value of the spacing factor for moderate exposure of the concrete is usually taken to be 0.20 mm (0.008 in.). Somewhat larger values may be adequate for mild exposure, and smaller ones may be required for severe exposure, especially if the concrete is in contact with deicing chemicals. Care should be exercised in using spacing factor values in specifications since the standard deviation of that property has been found to approach one-fifth of the average when determinations are made in different laboratories. Hence, substantial differences in spacing factor may be caused solely by sampling and between laboratory variation. The factors affecting the variability of the test method are discussed in the section on Precision and Bias.

5.9 The air content and the parameters of the air-void system in hardened concrete depend primarily on the kind and dosage of the air entraining agent used, the degree of consolidation of the concrete, and its water-cement ratio. The values of the specific surface (α) and the void frequency *(n)* decrease rapidly with an increase of the water-cement ratio or the paste content if other conditions are not altered. Satisfactory values of specific surface (α) and spacing factor ($\bar{L}$) require that the void frequency be larger than about 315/m (8/in.). An increase in the water-cement ratio or the paste content must be accompanied by an increase in the air content, if the spacing factor ($\bar{L}$) is not to increase. The air content can be reduced substantially by extended vibration of the concrete, without a significant increase of the spacing factor ($\bar{L}$), provided the concrete was adequately air entrained originally. Extended vibration is not, however, recommended as a field practice because of the dangers of excessive bleeding and segregation.

5.10 The void frequency *(n)* is a critical parameter in determining the magnitude of the specific surface (α) and the spacing factor ($\bar{L}$). Consequently, utmost care must be taken in conducting either microscopical method to observe and record all air-void sections intersected by the line of traverse. Recognition of air-void sections of small size, for example, 10 µm (3.94 by 10^{-5} in.) is essential to securing a correct evaluation of these parameters. For this reason, care must be taken to prepare extremely smooth and plane sections, the magnification employed should be not less than 50×, and the index point in the cross hairs (or other reticle device) must be observed precisely in relation to the area and periphery of the air-void section.

5.11 Provided the value of the specific surface (α) or the void frequency *(n)* is sufficiently high, a suitable spacing factor ($\bar{L}$) will be obtained even when the air content is low. However, in order to obtain an air-void system that has both the volume capacity and the geometric parameters necessary to protect saturated mature cement paste during exposure to freezing, it is important to obtain concrete with an acceptably high air content *(A)* and a low enough spacing factor ($\bar{L}$) to provide protection (**12**).

5.12 For concrete exposed to freezing and thawing while critically saturated, a minimum-compressive strength must be developed prior to the freezing exposure, in addition to the securing of adequate air entrainment if the concrete is to be protected properly. Such compressive strength must be at least 28 MPa (4000 psi).

SAMPLING AND SECTION PREPARATION

6. Apparatus and Materials for Sample Preparation (for either procedure)

6.1 Apparatus and materials for the preparation of surfaces of concrete samples for microscopical observation are described in Practice C 856; other apparatus may be equally suitable.

NOTE 1—Apparatus for measurement of prepared samples is described in the two following procedures.

7. Sampling (for either procedure)

7.1 Samples of concrete can be obtained from specimens cast in the field or laboratory, or by coring, sawing, or otherwise removing concrete from structures or products. The procedure followed and the location from which the samples are obtained will depend on the objectives of the program. In general, secure samples of hardened concrete in accordance with Test Method C 42 or Practice C 823 or both. Provide at least the minimum area of finished surface given in Table 1 in each sample. A sample may be composed of any number of specimens.

7.2 For referee purposes or to determine the compliance of hardened concrete with requirements of specifications for the air-void system, obtain samples for analysis by this test method from at least three randomly selected locations over the area or throughout the body of concrete to be tested, depending upon the objectives of the investigation.

8. Preparation of Sections (for either procedure)

8.1 Unless the objectives of the program dictate otherwise, saw the section for observation approximately perpendicular to the layers in which the concrete was placed or perpendicular to the finished surface. Individual sections should be as large as can be ground and examined with the available equipment. The required area may consist of more than one prepared section. Spread the selected traverse length uniformly over the available surface so as to compensate for the heterogeneity of the concrete.

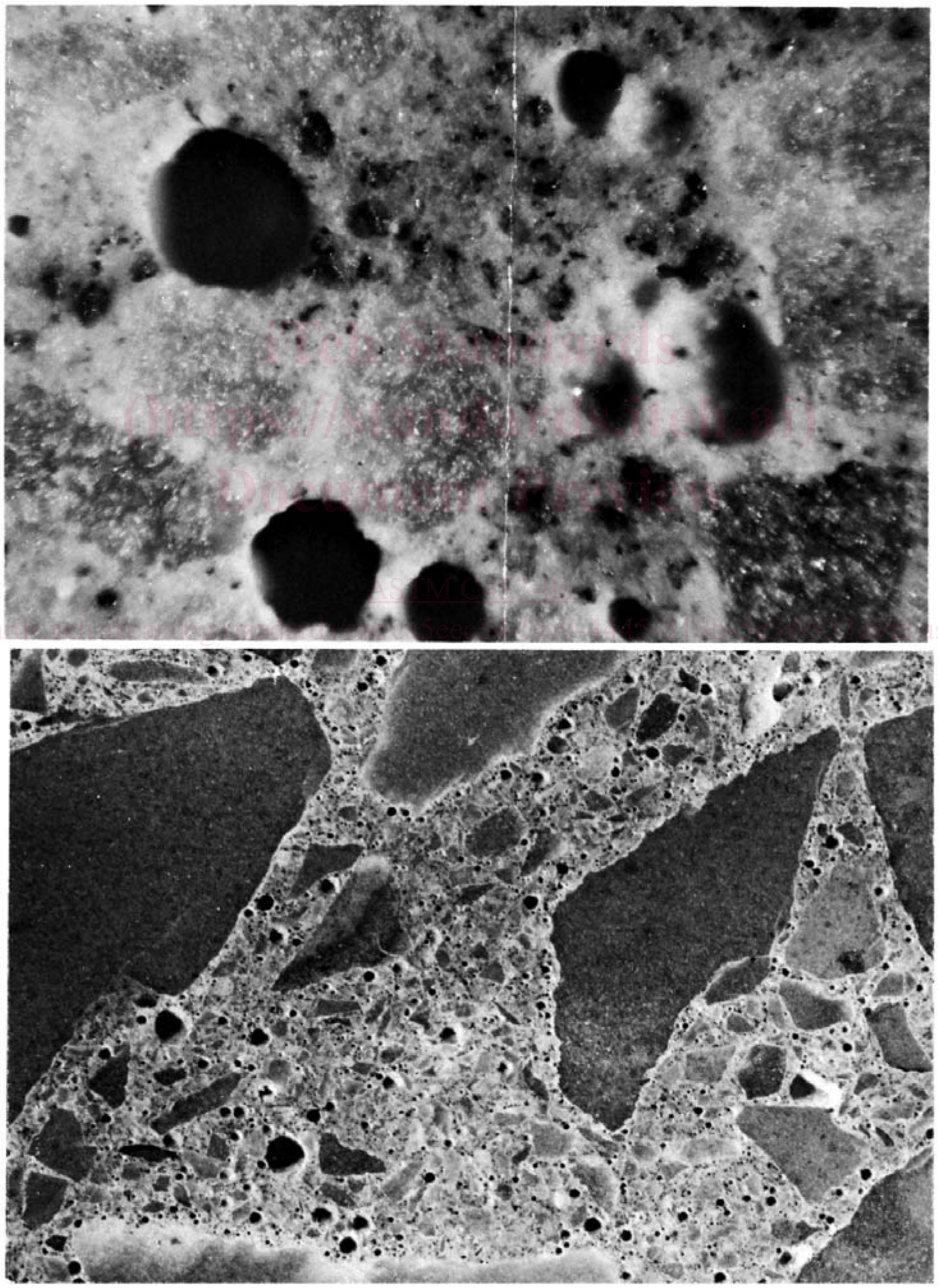
TABLE 1 Minimum Area of Finished Surface for Microscopical Measurement

Nominal or Observed Maximum Size of Aggregate in the Concrete, mm (in).	Total Area to be Traversed for Determination of α or $\bar{L}$ [A], min, cm² (in.²) Based on Direct Measurement of:	
	Total Air-Void Content,[A,B]	Paste-Air Ratio, *p/A*
150 (6)	1613 (250)	645 (100)
75 (3)	419 (65)	194 (30)
37.5 (1½)	24 (155)	97 (15)
25.0 (1)	77 (12)	77 (12)
19.0 (¾)	71 (11)	71 (11)
12.5 (½)	65 (10)	65 (10)
9.5 (⅜)	58 (9)	58 (9)
4.75 (No. 4)	45 (7)	45 (7)

[A] See Section 3 for the interpretation of symbols employed.

[B] The indicated values refer to reasonably homogeneous, well-compacted concrete. The microscopical measurement should be made on proportionately larger area of sections if the concrete is markedly heterogeneous in distribution of aggregate or large air voids. If more than one finished surface is taken from a single portion of the concrete, the finished surfaces shall be separated by a distance greater than one half of the nominal or observed maximum size of aggregate.

8.2 If gross irregularities are present, begin the surface preparation by lapping (grinding on a flat surface) with nominal 150 µm (No. 100) silicon carbide abrasive. Lap the surface with successively finer abrasives until it is suitable for microscopical observation. An appropriate series of abrasives would include nominal 75, 35, 17.5 and 12.5 µm grit sizes (No. 220, 320, 600, and 800, respectively), and perhaps 5-µm aluminum oxide (Note 2). From time to time during lapping, and when changing to a finer abrasive and when lapping is complete, clean all surfaces of the specimen gently and thoroughly to remove the grinding compound. Use of ultrasonic cleaners may be harmful to the surface. Such treatment should not be used without care and experimentation. Cleaning with a soft cosmetic brush under running water, or by a pressurized dental spray has been successful. A surface that is satisfactory for microscopical examination will show an excellent reflection of a distant light source when viewed at a low incident angle and there shall be no noticeable relief between the paste and the aggregate surfaces. Areas that are scratched or imperfect indicate the need for additional preparation; use special techniques if required (see 8.3). The edges of the sections of the air voids will be sharp and not eroded or crumbled, and air-void sections including those as small as 10 µm ($3.94\ 10^{-5}$ in.) in diameter will be clearly distinguishable.



(*a*) At 6×

FIG. 1 Photographs of a Satisfactory Surface

(See Fig. 1.) Do not include scratched or broken portions of the surface in the analyzed area. If needed to meet the requirements of Table 1, prepare additional surfaces.

NOTE 2—Grit numbers of abrasives can denote slightly different particle sizes, depending on the manufacturer. The suggested sizes will usually be appropriate, but others may be selected according to the experience of the user.

8.3 Sometimes difficulty will be encountered in preparing the lapped surfaces. The usual cause is a weak cement-paste matrix. The problem is manifested by the plucking of sand grains from the surface during the lapping, with consequent scratching of the surface, and by undercutting of the paste around the harder aggregate particles. Friable particles of aggregate can also cause difficulty. In such instances the following procedure is helpful. Heat the partially prepared specimen of concrete to about 150 °C (300 °F) in an oven. (**Warning**—If the specimen was sawn with a lubricant other than water, heating must be done so as to avoid inhaling the fumes and to preclude fire or explosion. Some lubricants have a flash point as low as 140 °C (285 °F). (The flash point of the lubricant may be found by use of Test Method D 92.) Unless other precautions are taken, the temperature must not be allowed to approach the flash point. If this cannot be avoided, heating must be done in the open air on a hot plate or in an explosion-proof hood.)

Remove the specimen from the oven and immediately brush melted carnauba wax that was heated to the same temperature onto the surface. Repeat the application as the wax is absorbed by the concrete, so that when the temperature of the concrete falls below the melting point of the wax, a perceptible film remains on the surface. After the specimen has cooled, scrape off any excess wax and repeat the lapping. After completion of lapping, remove the residue of wax from the surface air voids by reheating the concrete to about 150 °C (300 °F) to allow absorption of the molten wax into the specimen. Again take care to avoid approaching the flash point of the wax or of any cutting oil present. Protect the surface from dust during heating. The time to remove the wax from the surface air voids varies with the properties and thickness of the specimen, but heating for about an hour is usually sufficient. Exceptionally fragile concrete may require repetition of this process. Substances other than carnauba wax have been used successfully to impregnate and strengthen the surfaces of concrete specimens before grinding.

NOTE 3—If performing a microscopic examination of the cement paste, using Practice C 856, on the same test sample that will be coated with wax or other paste strengthening media, perform the microscopic examination prior to heating the sample and application of the strengthening materials. The application of such materials and exposure to oven temperatures of 150 °C will alter the physical characteristics and appearance of the cement paste.

8.4 If the parameters of the air-void system near a finished or formed surface are desired, then prepare the section examined in such a manner as to allow for the fact that the parameters of the air-void system may vary greatly with the distance from such a surface. Therefore, measure the distance between the section to be examined and the original surface accurately, to at least the nearest 1.2 mm (0.05 in.). Use the following procedure: (*1*) Prepare a specimen that includes a portion of the finished or formed surface to be investigated, and of convenient thickness, but not less than 12 mm (½ in.) or one-half of the nominal maximum size of the aggregate, whichever is greater. (*2*) Lap the surface with a coarse abrasive until the last portion of the original surface is just removed, then complete the lapping operation as described above. Use this surface as the reference plane, to which later measurements are referenced. (*3*) Lap the back surface of the specimen so as to produce a plane section. (*4*) Measure the thickness of the specimen to the nearest 1.2 mm (0.05 in.) at four or more points uniformly spaced around the periphery. Average the results, and record the average to the nearest 1.2 mm. (*5*) Determine the parameters of the air-void system on any plane desired or specified. If nearest surface values are desired, make the determination on the reference plane; if values for the bulk concrete are desired, make the determination on the back plane. If values for some other plane are desired, repeat the grinding process to the desired depth. Redetermine the thickness of the sample as specified above so that the parameters of the air-void system can be correlated with the distance of the examined surface from the reference plane.

8.5 The composition of the near-surface zone differs from that of the concrete as a whole. Therefore, whenever the design of the mixture is known, use the paste-air ratio method for the determination of the air-void system parameters in this region.

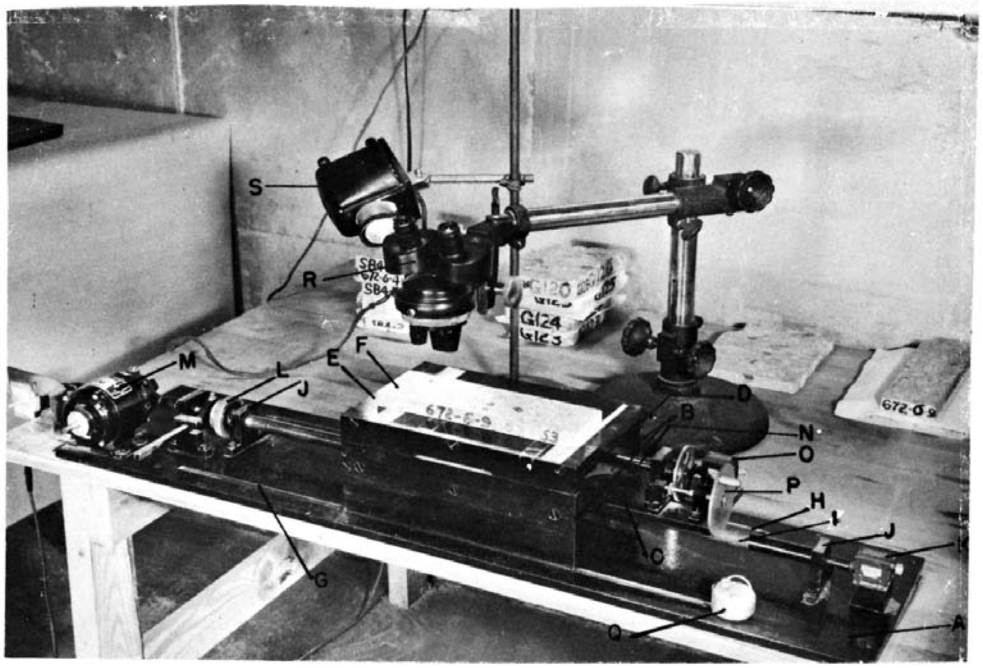
PROCEDURE A—LINEAR TRAVERSE METHOD

9. Apparatus for Measurement of Specimens

9.1 The apparatus listed in 9.1.1 to 9.1.5 comprises a recommended minimum selection. Apparatus other than that described has been used and may be equally satisfactory. Apparatus that uses electronic switches and totalizers has been constructed. Computerized apparatus is commercially available. Image analyzers have frequently been used.

9.1.1 *Linear-Traverse Device*—Provide a platform, on which the specimen is carried mounted on lead screws by means of which it can be smoothly translated in two perpendicular directions. Provide one lead screw for movement in the N-S direction and at least two for movement in the E-W direction (Note 4). One of these latter is called the "main" lead screw and the other(s) the "upper" lead screw(s). Ensure that the capacity of the main (E-W) lead screw is at least 100 mm (4 in.), that of each (E-W) lead screw at least 65 mm (2.5 in.), and that of the N-S lead screw at least 75 mm (3.0 in.). Ensure that the pitch of the upper lead screw does not exceed 0.265 mm (0.0105 in.) per revolution. Determine the pitch of all E-W lead screws to the nearest 0.03 mm (0.001 in.). Attach rotation counters readable to the nearest 0.01 of revolution to all E-W lead screws. Provide a manually operated tally counter. For the determination of the paste content, provide a third E-W lead screw complete with rotation counter, unless each traverse is to be repeated, that is, performed once for the air content and again for the paste content. Photographs of satisfactory linear-traverse devices are shown in Figs. 2 and 3.

NOTE 4—In the descriptions of the linear-traverse and point-count devices the term "E-W direction" refers to the direction from the operator's right to his left, and "N-S" means the direction perpendicular to



A = Base plate.
B = Front and back rails supporting the middle plate *C*
C = Middle plate.
D = Upper front and back rails carrying the stage *E*
E = Stage.
F = Concrete specimen
G = Rectangular front groove in the base plate.
H = V-shaped back groove in the base plate.
I = Main lead screw.
J = Two bearing blocks for the main lead screw.
K = Revolution counter on main lead screw.
L = Manually operated knurled wheel.
M = Electric motor for driving the main lead screw.
N = Upper lead screw.
O = Revolution counter for upper lead screw.
P = Hand-driven wheel for moving the stage.
Q = Ratchet counter to tally the number of air voids encountered.
R = Stereoscopic microscope.
S = Microscope lamp.

NOTE 1—Not shown are a third lead screw and a disengaging clutch; the former is necessary if a determination of the air-paste ratio is required, and the latter may be required (see 9.1.1).

FIG. 2 Photograph of a Linear-Traverse Device Meeting the Requirements of This Test Method

E-W, that is, the directions are analogous to those on a map.

9.1.2 *Stereoscopic Microscope and Support,* with objectives and eyepieces to give final magnification in the range from about 50× to about 125×. While it is possible to use a microscope with a single, fixed magnification, it is more convenient to be able to vary the magnification within the above range by replacing eyepieces or objectives or, better, by means of a zoom attachment. Arrange the microscope so as to permit continuous observations of the surface of the specimen mounted on the platform of the linear-traverse device. Include cross hairs, scale, or some other reticle device to provide an index point in one eyepiece. Since an index point is dimensionless it shall be a point such as the intersection of one pair of edges of the cross hairs or one corner of the end of a line of a scale. Use the same index point throughout any examination.

9.1.3 *Microscope Lamp*, spotlight-type, arranged to provide sufficient illumination at a low and variable incident angle to the surface. The spot of evenly lit area on the specimen surface should be slightly larger than the field of view of the microscope.

9.1.4 *Spirit Level*, the small circular type is convenient.

9.1.5 *Leveling Device*—Provide a means to level the examined surface. This can be done by the insertion of small pieces of modeling clay. A better way is by means of a platform that