

Designation: C457/C457M - 16

Standard Test Method for Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete¹

This standard is issued under the fixed designation C457/C457M; 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 pasteair ratio of the air-void system in hardened concrete. Three procedures are described:
 - 1.1.1 Procedure A—Linear-traverse method.
 - 1.1.2 *Procedure B*—Modified point-count method.
 - 1.1.3 *Procedure C*—Contrast enhanced method.
- 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 either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.
- 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 Note 9 and Note 12.
- 1.6 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Documents

- 2.1 ASTM Standards:²
- C42/C42M Test Method for Obtaining and Testing Drilled Cores and Sawed Beams of Concrete
- C125 Terminology Relating to Concrete and Concrete Aggregates
- C138/C138M Test Method for Density (Unit Weight), Yield, and Air Content (Gravimetric) of Concrete
- C173/C173M Test Method for Air Content of Freshly Mixed Concrete by the Volumetric Method
- C231/C231M Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method
- C666/C666M Test Method for Resistance of Concrete to Rapid Freezing and Thawing
- C670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials
- C672/C672M Test Method for Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals
- C823/C823M Practice for Examination and Sampling of Hardened Concrete in Constructions
- C856 Practice for Petrographic Examination of Hardened Concrete d6-face 766b 77 / astm-c457-c457m-16
- D92 Test Method for Flash and Fire Points by Cleveland Open Cup Tester
- 2.2 American Concrete Institute Standards:³
- 201.2R Guide to Durable Concrete
- 211.1 Standard Practice for Selecting Proportions for Normal, Heavyweight, and Mass Concrete

3. Terminology

- 3.1 For definitions of terms used in this test method, refer to Terminology C125.
 - 3.2 Definitions of Terms Specific to This Standard:
- 3.2.1 average chord length (\bar{l}) , n—the average length of the chords formed by the transection of the voids by the line of

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² 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.

³ Available from American Concrete Institute (ACI), P.O. Box 9094, Farmington Hills, MI 48333-9094, http://www.aci-int.org.

traverse; the unit is a length.

- 3.2.2 *binary image*, *n*—formed by segmenting an image using only one threshold with the resulting image having only areas of black or white.
- 3.2.3 digital image, n—an image captured using a computer-based storage method where the information presented in the image can be seen visually, like a traditional photographic image, but can also be extracted in a numeric form that can be used for additional analysis.
- 3.2.4 *paste-air ratio* (*p/A*), *n*—the ratio of the volume of hardened cement paste to the volume of the air voids in the concrete.
- 3.2.5 paste content (p), n—the proportion of the total volume of the concrete that is hardened cement paste expressed as percentage by volume.
- 3.2.5.1 *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 supplementary cementitious materials present.
- 3.2.6 *pixel*, *n*—the smallest definable point of a digital image that has an assigned value representing the brightness of that component in an image.
- 3.2.6.1 *Discussion*—Typically a pixel will have the same aspect ratio as the overall image and will have an assigned integer value occurring in the range 0-255. Multiple pixels are arranged contiguously in two-dimensional arrays to form a digital image.
- 3.2.7 *segment*, *v*—the process of placing image pixels into classes or like-groupings using any number of thresholds.
- 3.2.8 spacing factor (\bar{L}) , n—a parameter related to the maximum distance in the cement paste from the periphery of an air void, the unit is a length. Og standards/sist/50146ac
- 3.2.9 *specific surface* (α), n—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.
- 3.2.10 *threshold*, *n*—a value used to discriminate pixels into more than one class or like grouping.
- 3.2.11 *void frequency (n)*, *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.
- 3.2.11.1 *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).
- 3.2.12 water void, n—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 watervoid 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 specimen. The data gathered are the total length traversed $(T_{\rm t})$, the length traversed through air voids $(T_{\rm a})$, the length traversed through paste $(T_{\rm p})$, 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 $T_{\rm a}$ and $T_{\rm t}$ 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 specimen. 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 Procedure C, Contrast Enhanced Method—This procedure consists of the determination of the volumetric composition of the concrete by summing distances measured in digital images of a prepared concrete surface using a series of regularly spaced lines in one or more digital images obtained from one or more planes intersecting the specimen. The specimen is prepared exactly as described for Procedures A and B with the additional steps of darkening the specimen surface and filling the air voids with a fine particle size white powder. The data gathered are the total length measured (T_t) , the length measured through air voids (T_a) , and the number of air voids intersected by the measurement lines (N). These data are used to calculate the air content and various parameters of the air-void system, except the paste-air ratio and spacing factor that require determination of the paste content (T_p) as described in 16.1. If only the air content is desired, only T_a and T_t need be determined.
- 4.4 Paste-Air Ratio Modification—In some instances the specimen is not representative of the concrete as a whole, so $T_{\rm t}$ and $S_{\rm t}$ lose their significance and cannot be used as a basis for calculations. The most common examples are concrete with large coarse aggregate and specimens from the finished surface region, for both of which the examined specimen 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 5.5).

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 damage due to cyclic freezing and thawing 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 resistance of concrete to cyclic freezing and thawing.

5.2 Values for parameters of the air-void system can be obtained by any of the procedures described in this test method. The selection of which one of the three methods to be used shall be subject to agreement of the user and provider of the determination

Note 1—Because Procedure C requires darkening the paste and aggregate, its use must occur after other tests if the analyst is also gathering petrographic data in addition to the measurements 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.
- 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 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.

Note 2—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 C138/C138M, C173/C173M, or C231/C231M. However, significant differences may be observed if the sample of fresh concrete is consolidated to a different degree than the specimen 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 C231/C231M.

SAMPLING AND SECTION PREPARATION

6. Apparatus and Materials for Specimen Preparation

- 6.1 Apparatus and Materials for All Procedures—
- 6.1.1 Apparatus and materials for the preparation of surfaces of concrete speciemens for microscopical observation are described in Practice C856; other apparatus may be equally suitable.

- 6.2 Materials for Procedure C—
- 6.2.1 *Opaque Permanent Black Ink*, wide felt-tipped marker, black ink stamp pad, or black ink roller, or similar.
- 6.2.2 *White Powder*, barium sulfate, wollastonite, or titanium dioxide with a median particle size of 2-3 µm, or similar. 6.2.3 *Light Oil*, light mineral oil, or similar.

Note 3—Apparatus for measurement of prepared specimens is described in the three following procedures.

7. Sampling (for all procedures)

- 7.1 Specimens of concrete can be obtained from concrete 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 specimens are obtained will depend on the objectives of the program. In general, secure samples of hardened concrete in accordance with Test Method C42/C42M or Practice C823/C823M or both. Provide at least the minimum area of finished surface given in Table 1 in each specimen. 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

- 8.1 Preparation of Sections for All Procedures—
- 8.1.1 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

TABLE 1 Minimum Area of Finished Surface for Microscopical Measurement $^{\!A}$, $^{\!B}$

Nominal or Observed Maximum Size of Aggregate in the Concrete, mm [in.]	Total Area to be Traversed ^C for	
	Determination of p , ^{D}A , α , or \bar{L} , min, cm ²	
	[in. ²]	
	Based on Direct Measurement of:	
	Total Air-Void Content	Paste-Air Ratio, p/A
150 [6]	1613 [250]	645 [100]
75 [3]	419 [65]	194 [30]
37.5 [1½]	155 [24]	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 [3/8]	58 [9]	58 [9]
4.75 (No. 4)	45 [7]	45 [7]

^AThe indicated values refer to reasonably homogeneous, well-compacted concrete. The microscopical measurement shall 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.

 D When p is determined, it shall be determined by analyzing the same area to be traversed for determination of A, α , or \bar{L} .

^B See Section 3 for the interpretation of symbols employed.

^CWhen performing a point count to determine p, A, α , or \bar{L} , the analysis points shall be distributed evenly over the area to be traversed.

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.

8.1.2 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 (No. 2500 grit) aluminum oxide.

Note 4—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.

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 Note 9 and Note 12). 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 [0.0004 in.] in diameter will be clearly distinguishable. (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.

8.1.3 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

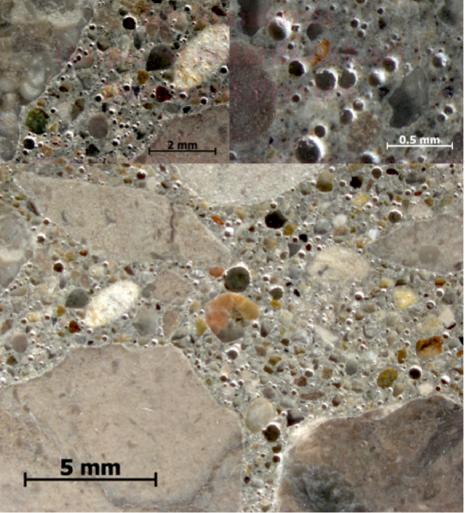


FIG. 1 Photographs of a Satisfactory Surface

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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 D92.) 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 5—If performing a microscopic examination of the cement paste, using Practice C856, on the same test specimen that will be coated with wax or other paste strengthening media, perform the microscopic examination prior to heating the specimen and application of the strengthening materials. The application of such materials and exposure to oven temperatures of 150 °C [300 °F] will alter the physical characteristics and appearance of the cement paste.

8.1.4 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 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 [1/2 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 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 mm [0.05 in]. (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 specimen 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.

- 8.1.5 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.
 - 8.2 Additional Preparation Required for Procedure C—
 - 8.2.1 Prepare specimens in accordance with 8.1.

Note 6—If paste-air ratio and spacing factor are to be determined, it is necessary to provide a determination of the specimen paste content. If this determination is to be made by point counting or another procedure requiring visually distinguishing aggregate from paste, such determinations must be performed prior to blackening the specimen surface.

8.2.2 Apply an even layer of opaque, permanent, black ink to the surface of the prepared specimen so the entire surface is rendered uniformly black.

Note 7—Black markers have successfully been used as has black ink applied with a roller such as that used in offset printing.

- 8.2.3 After the ink has dried, distribute a layer of white powder over the blackened surface and gently press it into the voids using a suitable implement such as a rubber stopper, glass rod, or petrographic slide. Avoid abrading the surface by minimizing rubbing the powder over the surface.
- 8.2.4 Remove excess powder by scraping it from the surface with a suitable implement (e.g., a sharp, new single-sided razor blade). Avoid abrading the ink thereby exposing cement or aggregate grains.
- 8.2.5 Inspect the darkened and filled surface. Areas where ink has been abraded should be re-touched with a local, fresh application of ink. Porous aggregate grains should likewise be re-touched with a local, fresh application of ink such that internal pores in the aggregate are rendered black. Blacken areas filled in by white powder that are identified as pullouts occurring during specimen preparation. Likewise, blacken areas filled in with white powder that are identified as coarse cracks or other defects in the specimen not classified as part of the air-void system.
- 8.2.6 With a very lightly oiled finger or thumb, remove the last remnants of white powder from the surface, leaving a shiny black surface without the presence of unfilled voids.

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

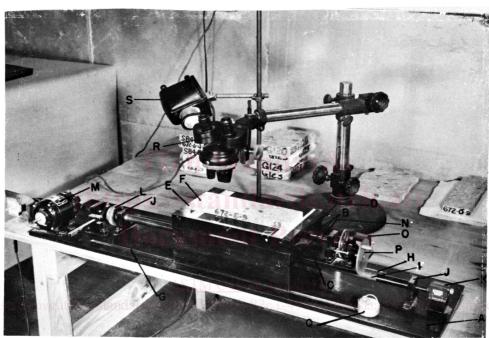
means of which it can be smoothly translated in two perpendicular directions. Provide one lead screw for movement in the N-S direction and at least two for movement in the E-W direction.

Note 8—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 E-W, that is, the directions are analogous to those on a map.

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 0.025 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 3.

9.1.2 Stereoscopic Microscope and Support, with objectives and eyepieces to give final magnification in the range from about $50 \times$ to about $125 \times$. While it is possible to use a



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- 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