

**Evaluation of Methods for Characterizing
Air-Void Systems in Wisconsin Paving Concrete**

Submitted by:

Transportation Materials Research Center
Michigan Technological University
1400 Townsend Dr.
Houghton, MI 49931

Principal Investigator:

Lawrence L. Sutter, Ph.D.
Michigan Technological University
School of Technology
1400 Townsend Dr.
Houghton, MI 49931

Co-Principal Investigators

Thomas Van Dam, Ph.D., PE
Michigan Technological University
Civil and Environmental Engineering
1400 Townsend Dr.
Houghton, MI 49931

Michael Thomas, Ph.D., PE
Professor, Department of Civil Engineering,
University of New Brunswick
P.O. Box 4400
Fredericton, N.B., Canada E3B 5A3

Problem Statement

Portland cement concrete (PCC) is an inherently durable material used for constructing roads and bridges. The properties and durability of hardened concrete can vary widely depending upon the relative volumes of cement, sand, aggregate, and water used along with the volumetric content of air. Properly sized and spaced entrained air bubbles have long been known to protect concrete from cyclic freezing and thawing. As a result, air entrained concrete is universally specified for road construction in the upper Midwest (e.g. Wisconsin, Michigan, Minnesota, Iowa). To be effective, a minimum volumetric content of air must be entrained and the bubbles must be within close enough proximity to one another to protect the paste from damage during freezing. Powers developed an expression called the spacing factor that describes, for the majority of the paste, the distance to the nearest air void (Snyder 1998). Typically, the entrained air bubbles should range in size from 50 to 200 μm (Mehta and Montiero 1993) and the spacing factor should be less than 0.200 mm (0.008 in) to be effective in protecting the hydrated cement paste from F-T damage (ASTM C-457). However, it is noted that if excessive air is entrained in the concrete, there is a commensurate loss of strength and an increase in permeability, both greatly negatively affecting the durability of the concrete.

To construct durable roads, it is necessary to monitor and control the addition of air entraining admixtures used in making concrete. This is generally accomplished by monitoring the total air content of fresh concrete delivered to a job site. The two most common methods used for assessing the air content of fresh concrete are ASTM C-231 *Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method* and ASTM C-173 *Test Method for Air Content of Freshly Mixed Concrete by the Volumetric Method*. Both methods have been widely applied for many years. It has been observed that these methods may not accurately measure the true volume of air entrained in the fresh concrete, especially if the entrained air bubbles are very small (Roberts 1994).

In the past, most air entraining admixtures were derived from naturally occurring vinsol resins. Recently, it is becoming more and more common that air entrainment is induced through the use of synthetic air entraining admixtures rather than those produced from vinsol resins. It is not well understood how changing this important admixture changes the resulting concrete microstructure. There is some concern that the resulting air-void system has a larger volume percent of smaller air voids with respect to the total air content. If a larger fraction of smaller air voids is coupled with a field test that is insensitive to these smaller air voids, artificially low air readings on the job site may result. Then, the mix plant will adjust to correct for the “low” measured air contents, ultimately producing concrete with an excess air content. This concrete is inherently weaker and more importantly, more permeable, contributing to low durability characteristics.

There are established alternatives to measuring the air content in fresh concrete. The principal alternative is ASTM C-457 *Standard Test Method for Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete* which is performed on polished slabs cut from concrete cores. The air-void system parameters are measured using an optical microscope and linear traverse or point counting methods. The process is tedious and requires a skilled operator to prepare the samples and perform the microscopic analysis. Even experienced operators may end up with different results when evaluating the same concrete depending upon their individual criteria for identifying phases (paste, aggregate, and air) and how they address other factors such as infilling of bubbles with secondary deposits (e.g. ettringite, portlandite). Although this method is generally viewed as being the most widely accepted characterization of PCC air-void systems, inherent difficulties associated with conducting the test in a repeatable manner makes it impractical for use as a routine quality control procedure.

As a result of the known relationship that exists between the air-void system parameters (e.g. total air content, spacing factor) and F-T durability, the true characteristics of the air-void system must be known to improve the durability of PCC used for paving in Wisconsin. Such a characterization study must establish the relationship(s) between the measured air content of fresh concrete made with different air entraining agents and the air-void system parameters measured in the hardened concrete using the approach described in ASTM C-457. Also, new methods of assessing the air-void system parameters in hardened concrete must be developed that reduce operator subjectivity and increase repeatability, providing an additional level of quality control to augment or calibrate measurements performed on fresh concrete. And finally, once procedures for accurately assessing the air-void system of PCC are established, best construction practices and materials selection must be employed to ensure that quality concrete pavements are constructed.

Research Objectives

1. Examine the relationships between various methods of measuring air content in fresh concrete and measurements of air-void system parameters using ASTM C-457 for mixtures made with vinsol resin and synthetic air entraining agents.
2. Evaluate three new emerging technologies that show promise for characterizing the air-void system in hardened concrete.
3. Correlate measured air-void system parameters collected using all techniques with freeze-thaw testing data obtained from ASTM C-666.
4. Perform a literature synthesis to document best construction practices and materials selection for producing a consistent, adequate air-void system in PCC used in pavement applications.

Background and Significance

Paste Freeze-Thaw Damage - When moist concrete is exposed to alternating cycles of freezing and thawing, internal deterioration can result. As this deterioration accumulates, it is referred to as freeze-thaw (F-T) damage. Damage related to freezing and thawing can occur in both the cement paste and aggregate phases of concrete. It has long been recognized (Powers 1945) that damage to the cement paste can occur internally or at the surface. Surface scaling most often occurs in the presence of deicers, which exacerbate the pressures generated through freezing and thawing. In both cases, the basic deterioration mechanism is identical, and the same paste air-void system is expected to protect the paste against damage. Aggregate F-T damage is not considered in this project, being primarily a function of the aggregate porosity, strength, and size.

Paste F-T damage in concrete is considered to be a physical phenomenon arising from excess internal pressures resulting from the freezing action of water. Currently, a general consensus on the exact mechanism(s) responsible for these excessive internal pressures does not exist. The most widely accepted theories consider either hydraulic or osmotic pressures (or a combination of the two) to be the primary causes. The role of entrained air voids is a central element in both of the theories. It is generally agreed that the magnitude of these internal pressures is dependent on the concrete pore structure, moisture content, pore water chemistry, rate of freezing and/or length of the freezing cycle. The temperature at which water will freeze is a function of the size of the pore in which it is contained and the concentration of dissolved species in the water. An excellent review of the literature related to these phenomena is provided by Marchand et al. (1994).

Powers (1945) first attributed F-T damage to excessive hydraulic pressures resulting from the expansion of ice. It was proposed that as ice gradually forms at discrete sites in a saturated capillary pore system, the resulting 9 percent volume expansion causes the surrounding unfrozen water to be expelled away from the freezing sites. Depending on the nature of the pore system, excessive internal stresses can be generated by hydraulic pressures resulting from resistance to this flow. The pressurized water moving away from the freezing sites can find relief at the air voids, where it will then presumably freeze without causing damage.

Based on experimental results, Powers recognized that the spacing between voids, rather than the total volume of air, was the better measure of resistance to F-T damage. Building on this, he developed equations that provided an average measure of the distance that water within the paste must travel to reach the surface of an air void (Powers 1949). He proposed the adoption of a void spacing factor (now known as the Powers spacing factor) as the basis of protecting the paste from F-T damage. It is interesting to note that this pioneering work based on the hydraulic pressure theory still forms the primary basis of specifying F-T resistant concrete (see ASTM C-457).

More recent theories (Powers 1975) consider osmotic potential to be the primary cause of excess internal stress. As previously mentioned, the temperature at which water will freeze in concrete is a function of the alkali concentration as well as the pore size in which it is contained. Freezing will only occur when the temperature becomes low enough to allow ice to form at the existing alkali concentration. Because of their relatively large size, air voids are likely initial freezing sites and as the pore water solution freezes, only pure water forms the ice. Thus, the remaining unfrozen liquid at the freezing sites becomes a more concentrated alkaline solution. The less concentrated alkaline solution in the surrounding paste is then drawn to the freezing sites to maintain thermodynamic equilibrium. The driving force for the movement of this solution is a function of the alkali concentration gradient. As the unfrozen solution at the freezing sites is diluted by the infusion of surrounding water, additional ice growth occurs. This progressive ice formation can occur at any solute concentration, including zero and is referred to as ice-accretion (Powers 1956, Powers 1975).

This process of pore water moving from the capillary system to contiguous air voids will continue until one of two possible conditions prevail. If adequate air-void space exists, sufficiently distributed throughout the paste, all of the freezable water will eventually diffuse to the freezing sites inside the air voids, eliminating any further fluid flow. This drains the surrounding capillary pore system eliminating the possibility of paste F-T damage. The other possible outcome is that the air-void system is inadequate to accommodate all of the surrounding unfrozen water. If this occurs, osmotic pressures will increase due to the remaining differences in alkali concentrations between the liquid in the air voids and the bulk solution within the capillary pores. Pressures of any kind, whether they are caused by loads, hydraulic forces, or osmotic forces, that approach or exceed the tensile strength of the hardened cement paste will naturally cause damage. Also, if the rate of temperature drop is too fast to allow all of the water to diffuse to the air voids, damage may occur.

Measurement of Air-Void System Parameters by ASTM C-457 - Concrete can be evaluated for susceptibility to damage by paste F-T damage by following ASTM C-457 procedures to assess the adequacy of the entrained air-void system (Walker 1992). Turnkey analytical systems to perform ASTM C-457 are not common, but available. Often researchers purchase the necessary hardware and write the necessary controlling software (Sutter 1998).

On normal strength concrete, entrained air-void systems with a Powers spacing factor of 0.008 in (0.20 mm) or less will typically provide good F-T protection. This value was empirically established primarily by laboratory F-T testing using ASTM C-666 (AASHTO T 161) (Philleo 1986). While the Powers spacing factor is not considered to be a truly definitive measure of F-T protection, it is still used as the standard method of quantifying the distribution of entrained air in concrete. Other measures for characterizing entrained air have been proposed (Walker 1980, Philleo 1983, Attiogbe 1993), but none have been adopted for general use.

The microscopic examination of the air-void system in hardened concrete can take the form of a point count, linear traverse, or an areal traverse. All three methods rely on measurements obtained from a polished plane surface of concrete. The general mathematical methods used to extrapolate measurements obtained from a two-dimensional surface to three-dimensional space are known as stereology (Underwood, 1970, Russ, 1986). Hilliard (1968) presents the mathematical derivations for volume fraction relationships based upon measurements in a two-dimensional slice of the volume, as is the case with a polished microscope specimen. The fundamental relationships leading to volume fraction estimation depend upon equivalence between the volume density of quantities measured on a plane section, including point, line, or area fractions. The fundamental relationships are shown below.

$$P_p = L_L = A_A = V_V \quad (1)$$

Where:

P_p = fraction of total points counted falling in phase of interest

L_L = fraction of total line length traversed falling in phase of interest

A_A = area fraction of phase of interest

V_V = volume fraction of phase of interest

Both methods of volume fraction estimation, point counting and linear traverse, have been applied to air void measurement in concrete.

In addition to the total volume of air in hardened concrete, ASTM C-457 details procedures for determining a Powers spacing factor. Powers and Willis (1949) developed two expressions for a spacing factor, both of which require a determination of the total air-void specific surface. They demonstrated that the total volume of air voids and their total specific surface could be estimated from the mean air-void intercept or chord length obtained from a linear traverse. Assuming all air voids to be spherical and using geometric probability concepts, the total specific surface, α , expressed in terms of the average chord length, \bar{l} , was shown to be:

$$\alpha = 4 / \bar{l} \quad (2)$$

Powers first spacing factor expression was obtained by simply calculating the volume of cement paste per unit area of air-void surface. This is given as:

$$\bar{L} = p / \alpha A \quad (3)$$

where: \bar{l} = spacing factor, in units of length
 α = total specific surface of the air voids, in consistent units of length⁻¹
 \bar{L} = spacing factor, in units of length
 p = paste content, in volume percent of concrete
 A = total volume of air voids, in volume percent of concrete

His second spacing factor expression is based on a hypothetical system of equal sized spherical voids uniformly distributed throughout the paste phase. The size of each of these hypothetical voids is determined by setting their specific surface (3/R) equal to the total measured specific surface of the true void system and then solving for the resulting sphere radius, R. By making the total air content of the hypothetical system of voids equal to the measured value of air content, the number of hypothetical voids is then determined. The Powers spacing factor thus obtained is:

$$\bar{L} = 3/\alpha[1.4(1 + p/A)^{1/3} - 1] \quad (4)$$

Powers recognized that neither expression for spacing factor provides a true measure of void spacing. Assuming that both expressions overestimate the true average void spacing, he recommended using the smaller spacing factor obtained from the two equations. Equation 3 yields a smaller factor for p/A less than 4.33, and Equation 4 gives the smaller value when p/A is greater than 4.33.

Emerging Technologies for Measuring Air-Void System Parameters - Research has continued on new methods of characterizing the air-void system parameters of both fresh and hardened concrete with an ultimate goal of making a more reproducible, less tedious method of determining these important material parameters.

The first technology, which has been under investigation for a number of years, is an alternative method for measuring the air-void system parameters for fresh concrete. The test equipment, known as the air-void analyzer (AVA) or the Danish air test, has received mixed reviews (Price 1996, Magura 1996). The basic principal of the AVA is to measure changes in buoyancy using a special buoyancy recorder that captures bubbles as they rise from the concrete, through a viscous liquid, and finally through water to the recorder. The viscous liquid retains the original bubble sizes and larger bubbles rise faster than smaller ones. Therefore, monitoring the change in buoyancy as a function of time allows for determination of the air-void size distribution, total air content, spacing factor, and specific surface.

Another emerging technology uses a high resolution flatbed scanner to collect a digital image of a polished cross-section through a concrete pavement. The procedures for the high resolution flatbed scanner technique are well documented in available publications (Peterson 2001a, 2001b, 2001c). The images obtained from this device have a maximum pixel resolution of 8 μm . Image processing software is used to classify each pixel in the image, differentiating it as either cement paste, aggregate, or air void. Once the image has been classified, a standard ASTM C-457 linear traverse or point count can be automatically performed using software. Other analysis procedures can also be applied. For example, the distance to the nearest air void can be calculated for each pixel classified as cement paste using an accumulated cost surface algorithm. The presence of aggregate is accounted for in the distance calculation since the accumulated cost surface algorithm allows pixels classified as aggregate to be treated as obstructions that must be navigated around before reaching the nearest air void, thereby modeling real water flow through the air-void system. Figure 1 shows a classified image obtained from a high resolution flat bed scanner.

Another emerging technology involves the application of high resolution industrial CT x-ray scans to measure air-void system parameters in concrete in three dimensions (Wiese 2000). The patent pending non-destructive technique uses small diameter samples of fresh or hardened concrete that can be scanned at resolutions of up to 10 microns. The resulting data set is a three dimensional representation of the sample showing the spatial distribution of aggregate, paste and air voids. This technique uses a well-established radiographic technique referred to as CT or computed tomography.

CT scanners typically produce multiple two-dimensional x-ray images, each representing cross sections or slices (also known as tomographs) through the specimen. The tomographs may be stacked one on top of one another to create a three-dimensional volume. In a linear array system, tomographic images are generated by projecting a thin-beam of x-rays through one plane of the specimen at many different angles. As x-ray photons pass through a specimen, some are absorbed while transmitted photons are measured by an x-ray detector. By collecting this data over many angles, a cross section representing the attenuation parameter of each element within the slice can be reconstructed by a computer.

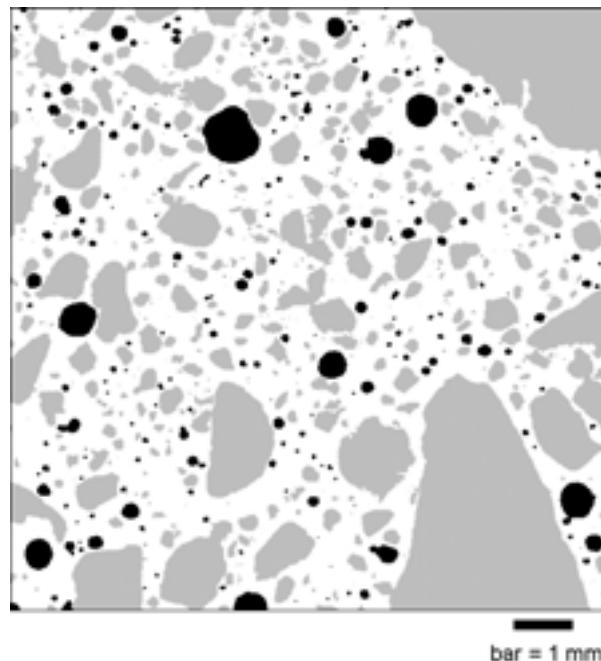


Figure 1: Classified image obtained from a high resolution flat bed scanner where air voids appear black, aggregates appear gray, and cement paste appears white.

In the more innovative volume CT system, the fan beam is extended to a cone beam and thereby collects multiple two-dimensional projections at once. Specialized reconstruction algorithms process the two-dimensional cone-beam projections into a volume that is the equivalent of many simultaneous, contiguous CT slices. The current patent pending system consists of a desktop microfocus industrial CT x-ray machine that uses cone beam technology. It is connected to a computer workstation that collects and reconstructs the volume. Visualization of the resulting three-dimensional matrix using volume rendering techniques is also done on a computer workstation.

A final emerging technology that has potential as a method characterizing the air-void system in PCC is the analytical x-ray microscope. The analytical x-ray microscope produces a "focussed" x-ray beam by passing the x-ray flux generated from an air-cooled x-ray tube through a fine collimator. The collimators are finely produced glass tubes that taper along their length to a final aperture size of 10 μm , 100 μm , or 300 μm , depending upon the collimator selected by the user. The final aperture size determines the point resolution of the image or the point analysis performed on the specimen analyzed. Using the x-ray microscope in a fluoresced x-ray mode, characteristic x-ray maps can be produced showing elemental distributions and point analyses can be performed to determine the composition of individual phases. When used in the transmitted x-ray mode, the x-ray microscope produces high-resolution (i.e. 10 $\mu\text{m}/\text{pixel}$) images of the internal specimen structure and may be used as a measure of bulk paste density. Cracks, voids, discontinuities, or areas of varying density can also be shown in this type of image. In addition, the incident x-rays from the x-ray source will fluoresce phases in the material and quantitative chemical analysis can be performed. All of these analyses are performed in air and as a result, no specimen degradation from desiccation occurs. Research remains to be performed to determine if slabs prepared with white wollastonite powder pressed into the air voids can be analyzed by x-ray mapping at 10 $\mu\text{m}/\text{pixel}$ using this instrument

Implementation

The findings from this research will fall into multiple categories with regards to implementation. A portion of the research will compare results from measurements of air content in fresh concrete and ASTM C-457 based analysis of the same concrete after hardening and curing. This may help establish or modify specifications regarding testing of fresh concrete for road construction. Another aspect of this research involves demonstration of concept for three emerging technologies for analyzing the air-void system of hardened concrete. None of these are intended to deliver a working instrument for implementation. However, with additional research, one or more of the concepts presented may develop

into a working approach implemented in the field or laboratory. Additional information gathered from comparing F-T data with measured air system parameters might in the future lead to new specifications for how to analyze PCC air-void systems. Finally, the synthesis of best construction practices and materials selection required to obtain an adequate air-void system will be delivered in a stand alone volume that can be readily circulated to field personnel for implementation.

Qualifications of Research Team

The faculty and staff of the Michigan Technological University Transportation Materials Research Center (TMRC) have had a strong research program in construction materials at Michigan Tech for many years. Externally funded research has been conducted for a diverse group of sponsors including the Michigan Department of Transportation, the Federal Highway Administration, the National Highway Cooperative Research Program, the National Science Foundation, and private industry. The TMRC possesses impressive capabilities for the investigation of portland cement concrete materials. This program is supported by full-time research staff and state-of-the-art equipment specifically chosen to provide thorough and rigorous analysis of portland cement based materials. The petrographic capabilities are housed in four new laboratories managed under the Non-Conductive/Volatile Materials Characterization Facility, which is co-directed by Drs. Lawrence Sutter and Thomas Van Dam. It is the unique combination of faculty, professional staff, and highly qualified students working with state-of-the-art facilities that make Michigan Tech's portland cement concrete characterization team an attractive research partner. Likewise, faculty and staff of the University of New Brunswick are world-renowned for their research in portland cement concrete. Dr. Thomas and his colleagues are leaders in ASTM, ACI, and TRB and his inclusion on this project greatly strengthens the overall research team. Qualifications of the individual researchers are presented below. A full statement of qualifications is included as Attachment B.

Lawrence L. Sutter, Ph.D.

Dr. Sutter is an Assistant Professor of Civil Engineering Technology at Michigan Tech University where he instructs on a variety of subjects including concrete technology, soil engineering, water and wastewater engineering. He has an extensive background in materials characterization and has done research on the characterization of construction materials including concrete and asphalt. He is currently involved in a number of projects for the Michigan Department of Transportation, the Federal Highway Administration, the National Cooperative Highway Research Program, and the National Science Foundation investigating concrete pavement durability and performance. Dr. Sutter's dissertation subject was the identification of materials related distress in portland cement concrete pavements. He is co-director of the University's *Facility for Analysis of Non-Conductive/Volatile Materials*, which includes a complete petrography lab, environmental scanning electron microscope, and newly developed x-ray microscope. He is an Officer of the International Cement Microscopy Association, Member of ASCE, ACI, and ASTM and an ACI Level 1 Concrete Technician Examiner.

Thomas J. Van Dam, Ph.D., P.E.

Dr. Van Dam is an Assistant Professor in transportation engineering, having specific interest in pavement materials, evaluation, design and performance. He is currently involved in a number of projects for the Michigan Department of Transportation, the Federal Highway Administration, the National Cooperative Highway Research Program, and the National Science Foundation investigating concrete pavement durability and performance. Dr. Van Dam is the Director of the Michigan Tech Transportation Materials Research Center and is the Co-Director of Michigan Tech's *Facility for Analysis of Non-Conductive/Volatile Materials*. Dr. Van Dam conducted doctoral work at the University of Illinois studying the design and performance of general aviation airport pavements. As a Project Manager for four years at ERES Consultants, Inc., he supervised the airport pavement evaluation and design services area. Dr. Van Dam's international experience includes two years of service in Tanzania, East Africa, as a U.S. Peace Corps volunteer and more recently working as a consultant to the Asian Development Bank in Malaysia. He is also co-director of the Peace Corps Masters International program in the Department of Civil and Environmental Engineering at Michigan Tech. He is an active member of ACI Committee 201, *Durability of Concrete*, and TRB Committee A2E01, *Concrete Durability*.

Michael D.A. Thomas, Ph.D., P.E.

Michael Thomas is a Professor in the Department of Civil Engineering at the University of New Brunswick. Between 1986 and 1991, Dr. Thomas worked at the Building Research Establishment in the U.K. under an Imperial College Research Fellowship. During this time he led a major research project investigating the influence of fly ash on concrete durability. In 1991 he accepted a position as a concrete materials engineer with Ontario Hydro in Canada, where he was responsible for the inspection and evaluation of concrete structures, and for conducting research in concrete materials.

From 1994 until 2002, he was on the faculty at the University of Toronto and joined the University of New Brunswick in July of 2002. Dr. Thomas's main research interests are concrete durability and the use of industrial by-products including pozzolans and slag. His studies on durability have included alkali-silica reaction, delayed ettringite formation, sulfate attack, chloride ingress and embedded steel corrosion. He is also active in the area of service life modeling. He has authored more than 100 technical papers and reports on these subjects. Dr. Thomas is active on technical committees within the American Concrete Institute, ASTM, RILEM and the Canadian Standards Association. He was a recipient of the ACI's Wason Medal for Materials Research in 1997 and the ACI Construction Practice Award in 2001. He is a member of ACI Committees 201 Durability, 222 Aggregates, 232 Fly Ash and Natural Pozzolans, 233 Slag, 234 Silica Fume, 236 Materials Science and chairs Committee 365 Service Life Prediction. He also serves on the Chapter Activities Committee and is past President of the ACI Ontario Chapter.

Karl Peterson

Mr. Karl Peterson is a Research Scientist with a focus on the microstructure of building materials. He is currently involved in a number of projects for the Michigan Department of Transportation, the Federal Highway Administration, the National Cooperative Highway Research Program, and the National Science Foundation focusing on concrete pavement durability and performance. Mr. Peterson is a well-published and respected petrographer whose creative talents and endless curiosity are of unmeasurable value to the Michigan Tech's research efforts. His Master's research focused on development of a methodology for performing ASTM C-457 with a high-resolution flatbed scanner.

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