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## NATIONAL RESEARCH COUNCIL OF CANADA CONSEIL NATIONAL DE RECHERCHES DU CANADA

### Apparatus for Preparing Portland Cement Paste of High Water-Cement Ratio

by

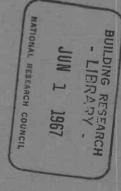
P. J. Sereda and E. G. Swenson

ANALYZED

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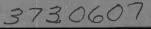
Technical Paper No. 244 of the Division of Building Research



OTTAWA April 1967

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APPAREIL POUR LA PREPARATION D'UNE PÂTE DE CIMENT DE PORTLAND À FORTE TENEUR EN EAU

### SOMMAIRE

Les auteurs ont préparé des échantillons de pâte de ciment de porosité uniforme et d'un rapport eau/ciment atteignant 1.1 à l'aide de l'appareil et selon la technique décrits dans le présent exposé. L'indice de porosité et la répartition des grosseurs des pores, déterminées à l'aide d'un porosimètre à mercure, n'ont montré que des écarts inférieurs à 5 pour cent.



### Apparatus for Preparing Portland Cement Paste of High Water-Cement Ratio\*

#### P. J. Sereda / E. G. Swenson

**REFERENCE:** P. J. Sereda and E. G. Swenson, "Apparatus for **Preparing Portland Cement Paste of High Water-Cement Ratio**," *Materials Research & Standards*, Vol 7, No. 4, April, 1967, pp. 152–154.

**ABSTRACT:** Samples of cement paste of uniform porosity having a water-cement ratio of up to 1.1 were prepared using the apparatus and technique described in this paper. Porosity and poresize distribution as determined by mercury porosimeter showed variations of less than 5 per cent.

**KEY WORDS:** portland cements, water-cement ratio, porosity, mixers, pore size distribution

<sup>2</sup> T. C. Powers, L. E. Copeland, and H. M. Mann, "Capillary Continuity or Discontinuity in Cement Pastes," Journal, Portland Cement Association Research and Development Laboratories, Vol 1, No. 2, May, 1959, pp. 38–48. For studies of the physical and mechanical properties of hardened portland cement paste it is desirable to have samples of practical size and uniform density and covering the widest range of water-cement (W/ C) ratio. The practical range of W/C ratio for mixes of Type I portland cement and water is about 0.25 to 0.5. Increasing the W/C ratio above the value of about 0.5 results in sedimentation and "bleeding" and a gradient in density as was shown by Powers et al.<sup>1</sup> Samples of cement paste having a high W/C ratio were prepared by Powers et al<sup>2</sup>, by using superfine portland cement.

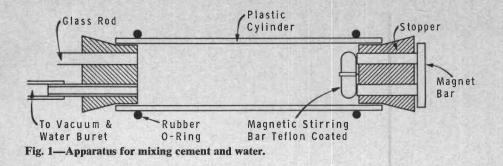
This paper describes a method for preparing a very homogeneous, highly porous sample of paste from portland cements of normal fineness.

The principal features of the proposed method are:

1. ability to evacuate the air from the dry powder,

<sup>\*</sup> This paper is a contribution from the Division of Building Research, National Research Council, Canada, and is published with the approval of the director of the Division.

<sup>&</sup>lt;sup>1</sup>T. C. Powers, L. E. Copeland, J. C. Hayes, and H. M. Mann, "Permeability of Portland Cement Paste," Proceedings, Am. Concrete Inst., Vol 51, 1954, pp. 285–298. <sup>2</sup>T. C. Powers, L. E. Copeland, and H. M. Mann, "Capillary Continuity: an Discontinuity Con-



2. introduction of the measured quantity of water into the evacuated powder,

3. ability to mix the water with the powder adequately, and

4. provision for hydration to proceed while the solid is maintained in a dispersed state in the water.

Requirements 1 and 2 are achieved by employing a well-known method for evacuation and introduction of water from a buret. Mixing is achieved by a relative motion between a magnetic stirring bar (Teflon coated) which remains fixed in the field of a large permanent magnet and a cylindrical plastic tube, which is moved by hand.

Requirement 4 is met by orienting the tube (with the mix inside) with its long axis horizontal and rotating it about this axis at such a rate that each particle is required to travel in a small elliptical orbit and thus maintain its relative position in the bulk. The rate selected should give a low component of centrifugal force relative to the gravitational force. Because the particle-size distribution of the powder and even its density distribution are not known, mathematical analyses to determine optimum speed of rotation are not possible. A further complication is the changing fluidity of the system with time because of the hydration products being formed.

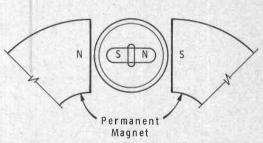
#### Apparatus

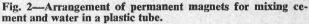
The mixer is shown in Fig. 1. The plastic cylinder, 3.2 cm in diameter and 14 cm long, is fitted with a two-hole rubber stopper at each end. One rubber

stopper retains two steel bars joined by a bar magnet 6 by 6 mm by 2.5 cm long which serves to hold the mixing magnet in place at one end during the hydration period. The other stopper carries a glass tube to serve as connection through a two-way valve-to-vacuum system and source of water in a buret, and a glass rod to adjust for the correct volume of water by displacement and to displace any accidentally trapped air. The amount of water can also be adjusted by changing the location of the stopper in the tube.

Figure 2 shows the mixer in position in the field of the horseshoe magnet of the type to give a magnetic flux density of about 2400 gauss (an electromagnet of similar magnetic strength should be equally suitable).

Figure 3 shows the roller device for rotating the mixer during the hydration of the cement. The rollers are 3.8 cm in diameter and spaced 5 cm apart (center to center). One of the four rollers is driven at  $8\frac{1}{2}$  rpm by a small motor. O-rings are fitted at each end of the mixer cylinder to facilitate smooth rolling. Driving





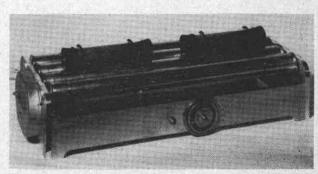
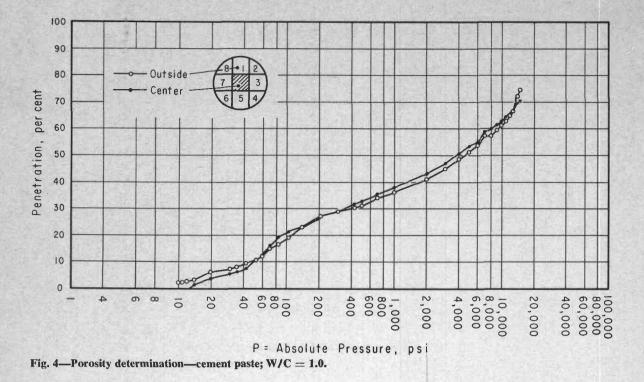


Fig. 3—Apparatus for rotating mixes of cement and water during setting.

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E. G. Swenson is a graduate of the University of Saskatchewan, with a Master's degree in chemistry. He did several years of postgraduate research on the chemistry of cement. In 1950, Mr. Swenson joined the Division of Building Research of the National Research Council as research officer in the field of cement and concrete. From 1959 to 1963 he carried out resarch in the cement and concrete industry in Montreal. He then rejoined the Division to continue his studies in the same field. Mr. Swenson is the author of some twenty papers on cement, concrete, and concrete aggregates. He is a member of ASTM Committees C-1 and C-9, of CSA Committees A5 on Cement and A23 on Concrete, and several committees of the Highway Research Board.



of other rollers is achieved through the O-rings on the two cylinders placed between each two rollers.

#### Procedure for Making the Samples

The mixing tube is fitted with the stopper carrying the two steel rods (without the bar magnet) and is placed at an angle of less than 45 deg. A weighed sample of portland cement screened through a 100-mesh sieve is placed in the mixing tube, and the magnetic stirring bar is placed on top of the cement. The rubber stopper is fitted into the top of the tube, and a connection to vacuum is made through a trap to prevent any powder from entering the vacuum pump during accidental lifting of the powder with rapid removal of air. This action is largely prevented when the tube is inclined at an angle of 45 deg or less. Evacuation by a rotary pump is continued for about 1 hr. Boiled distilled water is introduced from a buret to give the desired W/C ratio. The only important requirement of this operation is the exclusion of all the air. This can be achieved by adequate evacuation, and, when the boiled distilled water is added, the glass rod or the rubber stopper can be lowered so as to exclude all the free space from the mixer for final adjustment of the volume of water. The mixer is disconnected from the buret and vacuum connection and the short rubber tube is stoppered with a piece of glass rod. At this stage there must not be any bubbles of air.

The mixing is accomplished by moving by hand the mixer tube horizontally between the pole faces of the permanent magnet while rotating it at the same time. The tube is moved horizontally the full length and back about once every second and it is rotated one revolution in about two seconds. The mixing action is continued for a minimum of 5 min. For low W/C ratios it is extended to 15 min. This can be judged by the operator.

When mixing is stopped, the magnetic stirring bar is left at the rubber stopper that is equipped with iron rods. The mixer is removed from the magnetic field and a small bar magnet is placed across the two iron rods to retain the stirring magnet in place. The mixer is then placed on the rollers and rotated, usually for a period of 48 hr, at which time the rubber stoppers are removed and the sample is pushed out of the plastic tube using a wooden mandrel and a simple hand press. The time for demolding will depend on the type of mix.

The sample is stored under water in a stoppered plastic tube of 3.7 cm inside diameter.

#### Results

Samples of cement paste made from Type I portland cement of Blaine fineness  $3300 \text{ cm}^2/\text{g}$  having W/C ratios of 0.6, 0.8, 0.9, 1.0, and 1.1 were prepared. These were cut in such a way as to compare the porosity and pore-size distribution from end to end of the cylinder and radially from center to edge.

The pore-size distribution was determined, using a 15,000 psi mercury porosimeter, of sections of paste 5 by 10 by 10 mm taken from different parts of the cylindrical samples. The results indicate that samples of paste having a uniform density and pore-size distribution can be made by this method. A typical porosity curve is shown in Fig. 4. Porosity and pore-size distribution variations were less than 5 per cent.