Low Temperature Calorimetry Studies of Hydrating Portland Cement Pastes



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Abstract

This report presents nearly 200 low temperature calorimetry (LTC) scans performed on hydrating portland cement pastes between 2002 and 2005. Because the LTC scans provide valuable information on the percolation of various size pore networks within the hydrating cement pastes as well as the quantity of freezable water as a function of temperature, it was decided to present a compilation of the plots of all of the LTC results obtained over the course of several research projects. Variables include water-cement mass ratio (w/c), curing time, curing temperature, saturation, and limestone and alkali additions. Each plot is characterized by the portland cement used, the mixing solution used, the w/c, the curing temperature, the measured degree of hydration (when available), the curing conditions with respect to saturation, the specimen age when tested, the specimen mass, the filename of the raw LTC data, and the testing date of the LTC run. The experimental procedures used to prepare and evaluate the samples are briefly presented, along with a set of observations obtained from interpretation of the numerous individual data sets.

Keywords: Building technology; curing; freezing; hydration; low temperature calorimetry; percolation.

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Introduction

Low temperature calorimetry (LTC) can be conveniently used to examine the percolation of various size pore networks in hydrating cement pastes [1]. During a freezing scan, as the temperature is lowered, water will freeze in progressively smaller pores. For hydrating cement pastes, as shown on the cover of this report and duplicated in Figure 1 below, up to three peaks may be observed in a typical freezing scan, corresponding to percolated networks of capillary pores, open gel pores, and dense gel pores, respectively, adopting the naming convention introduced in [1]. This experimental technique has been utilized extensively in several recent studies to provide valuable information on the influence of curing conditions and alkali additions on the depercolation (and sometimes the repercolation) of the capillary porosity in hydrating portland cement pastes [2-5]. During the course of these studies, nearly 200 individual LTC scans were conducted on a wide variety of hydrated cement paste specimens. Because most of these data could not be included in references [2-5], in this report, all of the plots generated for these scans are presented individually. It is envisioned that this graphical database will be of use for comparison purposes to other researchers utilizing this technique on cement-based materials and may suggest further avenues of research employing LTC.



Figure 1 Typical LTC scans for a hydrating portland cement paste.

Experimental Procedure

Cement pastes were prepared by mixing Cement and Concrete Reference Laboratory (CCRL) proficiency cement samples (either CCRL cement 140 or CCRL cement 152) with either distilled water or a solution of alkali compounds dissolved in distilled water, using a temperature-controlled high speed blender at either 20 °C or 40 °C. Details on the particle size distributions and phase compositions of the two cements can be found in an online database available at http://ciks.cbt.nist.gov/bentz/phpct/database/images. CCRL cement 152 was used for the studies where curing conditions (temperature and saturation) and water-cement mass ratio (w/c=0.35 or w/c=0.45) were the major variables [2,5], while CCRL cement 140 was used in a study (w/c=0.40) where the alkali type and content of the cement paste were varied [3, 4]. The alkali solutions were prepared by adding the appropriate compounds (see Table 1) to distilled water and stirring with a glass rod until complete dissolution. Cement 140 is a low-alkali cement, containing only 0.093 % Na₂O and 0.186 % K₂O per unit mass of cement. The additional alkalis prepared for each mixture are listed in Table 1; their masses were selected to provide the same number of moles of additional cations $(K^+, Na^+, and Li^+)$ in each mixture. After mixing, cast cylindrical wafers (≈ 5 g) of the pastes were placed in sealed plastic vials. A small quantity of water was added to the tops of some of the wafers to maintain saturated curing conditions. The capped vials were placed either in a walk-in environmental chamber maintained at 20 °C or in a water bath maintained at 40 °C.

Material	Mixture 1	Mixture 2	Mixture 3	Mixture 4	Mixture 5
Cement	300 g				
Water	120 g				
K_2SO_4		2.79 g			
Na_2SO_4		2.28 g			
KOH [*]			2.02 g		
NaOH			1.30 g		
LiOH				2.7 g	
LiNO ₃					4.42 g

Table 1: Mixture proportions of the CCRL proficiency cement sample 140 paste mixtures prepared with various alkali additions.

*89 % purity as supplied by chemical company.

For the CCRL cement 152 specimens with w/c=0.45 cured at 20 °C, the vials were opened after 4 h of curing and any accumulated bleed water was removed using a small pipette. This resulted in a nominal achieved w/c=0.435 for these pastes. Three curing conditions were employed for the CCRL cement 152 studies. In saturated curing, a small amount of distilled water was placed on top of the paste wafers (after removing the bleed water). (All of the CCRL cement 140 paste specimens were also cured under saturated conditions). In sealed curing, the wafers were simply sealed in their plastic vials after removal of the bleed water. In sealed/saturated curing, the wafers were cured under sealed conditions for 7 d, then the plastic vials were opened and a small amount of distilled water was added on top of the wafers. At various ages, specimens of the pastes were removed from the vials for further analysis. For some of the LTC studies to be detailed below, many of the specimens cured under sealed conditions were crushed to smaller pieces and resaturated for a few days in an attempt to refill any pores that had been emptied during self-desiccation.

For many of the specimens, degree of hydration was estimated based on measurement of their non-evaporable water content. The non-evaporable water content w_n of a sample was determined as the mass loss between 105 °C and 1000 °C divided by the mass of the ignited sample, corrected for the loss-on-ignition (LOI) of the unhydrated cement powder, determined in a separate LOI measurement. Previously, the expanded uncertainty in the calculated w_n has been estimated to be 0.001 g/g cement, assuming a coverage factor of 2 [6]. The values of w_n were converted to estimated degrees of hydration based on the phase compositions of the cements and published coefficients for the non-evaporable water contents of the various cement clinker phases [7]. Based on a propagation of error analysis, the estimated uncertainty in the calculated degree of hydration is 0.004.

Small pieces of the hydrated cement pastes were used in the LTC experiments. Sample mass was typically between 30 mg and 90 mg. For each LTC experiment, one small piece of the relevant cement paste was surface dried and placed in a small open stainless steel pan. The pan with the sample, along with an empty reference pan of similar mass to the empty sample pan, was placed in the calorimeter cell. Using a protocol developed previously [1], a freezing scan was conducted between 5 °C and -55 °C at a scan rate of -0.5 °C/min. For temperatures between -100 °C and 500 °C, the equipment manufacturer has specified a constant calorimetric sensitivity of \pm 2.5 % and a root-mean-square baseline noise of 1.5 μ W. The peaks observed in a plot of heat flow (normalized to the mass of the sample) versus temperature correspond to water freezing in pores with various size entryways (pore necks). The smaller the pore entryway, the more the freezing peak is depressed. Thus, the presence of, absence of, or change in peaks can be used to infer information concerning the characteristic sizes of the "percolated" (connected) water-filled pores in the microstructure of the hydrating cement pastes.

One advantage of LTC over mercury intrusion porosimetry, and other techniques for assessing pore size and connectivity, is that specimens are evaluated without any external drying that might damage their pore structure. Of course, the LTC technique can only assess the size and connectivity of **water-filled** pores. For non-saturated curing conditions, it is assumed that the "empty" pores formed due to self-desiccation will not contain any freezable water and thus will not show up on the LTC scans. A further complication for LTC studies with variable alkali contents is the change in freezing point depression due to the variable ionic concentration of the (freezing) pore solution. For the experiments presented here, the initial dosages of added alkalis in the cement paste mixtures would be expected to depress the freezing point of bulk water between about 1 °C and 2 °C [8].

Table 2 on the following page is provided to give some guidance as to the mixtures and curing conditions corresponding to the various sets of graphs.

Cement	w/c or w/s	Curing	Starting Figure	Ending Figure
		Temperature (°C)		
152	0.25	20	4	30
152	0.35	20	31	86
152	0.435	20	87	94
152 + 20 % limestone	0.35	20	95	104
152	0.25	40	105	120
152	0.35	40	121	140
152	0.45	40	141	152
140	0.40	20	153	192

 Table 2: Plots corresponding to different sets of cement paste mixtures.

Processed Data (Graphs)

Control Materials

Curing: N/A
Age when tested: N/A
Sample mass: 45.1 mg
Filename: c140powder
Date tested: April 18, 2005



Figure 2 LTC scan for CCRL Cement 140 (dry) powder.



Figure 3 LTC scan for Gaithersburg tap water at two different scan rates.

CCRL Cement 152

Cement: CCRL Cement 152 Curing: Saturated Solution: Distilled water Age when tested: 1 d *w/c*: 0.25 Sample mass: 75.0 mg Filename: c152w025T20Csat1d Temperature: 20 °C Degree of hydration: 0.284 Date tested: May 17, 2005 Temperature (°C) -50 -45 -40 -35 -30 -25 -20 -15 -10 + 0.30 0.24 Heat Flow (W/g) 0.18 0.12 0.06 ± 0.00

Figure 4 LTC scan for CCRL Cement 152, w/c=0.25, cured for 1 d under saturated conditions at 20 °C.

Curing: Sealed

Cement: CCRL Cement 152

Solution: Distilled water Age when tested: 1 d *w/c*: 0.25 Sample mass: 57.3 mg Temperature: 20 °C Filename: c152w025T20Cseal1d Degree of hydration: 0.285 Date tested: May 17, 2005 Temperature (°C) -50 -40 -35 -30 -25 -45 -20 -15 -10 + 0.0250.020 Heat Flow (W/g) 0.015 j 0.010 I 0.005





Figure 6 LTC scan for CCRL Cement 152, w/c=0.25, cured for 2 d under saturated conditions at 20 °C.

Curing: Saturated Age when tested: 2 d Sample mass: 43.7 mg Filename: c152w025T20Cseal2d Date tested: May 18, 2005





Curing: Sealed 1 d/resaturated Age when tested: 2 d Sample mass: 78.4 mg Filename: c152w025T20Csealresat1t2d Date tested: May 18, 2005



Figure 8 LTC scan for CCRL Cement 152, *w/c*=0.25, cured at 20 °C for 1 d under sealed conditions, then resaturated for 1 d.

Cement: CCRL Cement 152 Solution: Distilled water *w/c:* 0.25 Temperature: 20 °C Degree of hydration: 0.456 Curing: Saturated Age when tested: 3 d Sample mass: 58.6 mg Filename: c152w025T20Csat3d Date tested: May 19, 2005



Figure 9 LTC scan for CCRL Cement 152, w/c=0.25, cured for 3 d under saturated conditions at 20 °C.



Figure 10 LTC scan for CCRL Cement 152, w/c=0.25, cured for 3 d under sealed conditions at 20 °C.

Curing: Sealed 2 d/resaturated Age when tested: 3 d Sample mass: 68.1 mg Filename: c152w025T20Csealresat2t3d Date tested: May 19, 2005



Figure 11 LTC scan for CCRL Cement 152, w/c=0.25, cured at 20 °C for 2 d under sealed conditions, then resaturated for 1 d.

Curing: Sealed 3 d/resaturated Age when tested: 4 d Sample mass: 59.1 mg Filename: c152w025T20Csealresat3t4d Date tested: May 20, 2005



Figure 12 LTC scan for CCRL Cement 152, w/c=0.25, cured at 20 °C for 3 d under sealed conditions, then resaturated for 1 d.







Figure 14 LTC scan for CCRL Cement 152, w/c=0.25, cured for 7 d under sealed conditions at 20 °C.

Curing: Sealed 7 d/resaturated Age when tested: 8 d Sample mass: 59.2 mg Filename: c152w025T20Csealresat7t8d Date tested: May 24, 2005



Figure 15 LTC scan for CCRL Cement 152, w/c=0.25, cured at 20 °C for 7 d under sealed conditions, then resaturated for 1 d.



Figure 16 LTC scan for CCRL Cement 152, w/c=0.25, cured for 15 d under saturated conditions at 20 °C.

Curing: Sealed Age when tested: 15 d Sample mass: 45.2 mg Filename: c152w025T20Cseal15d Date tested: May 31, 2005





Curing: Sealed 15 d/resaturated Age when tested: 16 d Sample mass: 59.2 mg Filename: c152w025T20Csealresat15t16d Date tested: June 1, 2005



Figure 18 LTC scan for CCRL Cement 152, w/c=0.25, cured at 20 °C for 15 d under sealed conditions, then resaturated for 1 d.



Figure 19 LTC scan for CCRL Cement 152, w/c=0.25, cured for 21 d under saturated conditions at 20 °C.



Figure 20 LTC scan for CCRL Cement 152, w/c=0.25, cured for 21 d under sealed conditions at 20 °C.

Curing: Sealed 21 d/resaturated Age when tested: 22 d Sample mass: 71.6 mg Filename: c152w025T20Csealresat21t22d Date tested: June 7, 2005



Figure 21 LTC scan for CCRL Cement 152, w/c=0.25, cured at 20 °C for 21 d under sealed conditions, then resaturated for 1 d.

Curing: Saturated Age when tested: 36 d Sample mass: 72.0 mg Filename: c152w025T20Csat36d Date tested: June 21, 2005



Figure 22 LTC scan for CCRL Cement 152, w/c=0.25, cured for 36 d under saturated conditions at 20 °C.



Figure 23 LTC scan for CCRL Cement 152, w/c=0.25, cured for 37 d under sealed conditions at 20 °C.

Curing: Sealed 37 d/resaturated Age when tested: 38 d Sample mass: 92.1 mg Filename: c152w025T20Csealresat37t38d Date tested: June 23, 2005



Figure 24 LTC scan for CCRL Cement 152, w/c=0.25, cured at 20 °C for 37 d under sealed conditions, then resaturated for 1 d.

Cement: CCRL Cement 152 Solution: Distilled water *w/c:* 0.25 Temperature: 20 °C Degree of hydration: 0.708 Curing: Saturated Age when tested: 56 d Sample mass: 84.3 mg Filename: c152w025T20Csat56d Date tested: July 11, 2005



Figure 25 LTC scan for CCRL Cement 152, w/c=0.25, cured for 56 d under saturated conditions at 20 °C.

Curing: Sealed Age when tested: 56 d Sample mass: 79.8 mg Filename: c152w025T20Cseal56d Date tested: July 11, 2005



Figure 26 LTC scan for CCRL Cement 152, w/c=0.25, cured for 56 d under sealed conditions at 20 °C.

Cement: CCRL Cement 152 Solution: Distilled water *w/c:* 0.25 Temperature: 20 °C Degree of hydration: N/A Curing: Sealed 56 d/resaturated Age when tested: 57 d Sample mass: 69.3 mg Filename: c152w025T20Csealresat56t57d Date tested: July 12, 2005



Figure 27 LTC scan for CCRL Cement 152, w/c=0.25, cured at 20 °C for 56 d under sealed conditions, then resaturated for 1 d.



Figure 28 LTC scan for CCRL Cement 152, w/c=0.25, cured for 91 d under saturated conditions at 20 °C.

Curing: Sealed Age when tested: 91 d Sample mass: 60.9 mg Filename: c152w025T20Cseal91d Date tested: August 15, 2005



Figure 29 LTC scan for CCRL Cement 152, w/c=0.25, cured for 91 d under sealed conditions at 20 °C.


Figure 30 LTC scan for CCRL Cement 152, w/c=0.25, cured at 20 °C for 91 d under sealed conditions, then resaturated for 1 d.

Cement: CCRL Cement 152 Solution: Distilled water <i>w/c:</i> 0.35 Temperature: 20 °C Degree of hydration: 0.316					Cur Age San File Dat	Curing: Saturated Age when tested: 1 d Sample mass: 52.2 mg Filename: cem152w35sat1d Date tested: Nov. 2, 2004			
Temperature (°C)									
-50	-45	-40	-35	-30	-25	-20	-15	-10	
								0.35	
								0.30	
								0.25 🚖	
								0 20 6	
								0.20	
							$- \parallel$	0.15 e	
							$-\parallel$	— 0.10 ម	
							-	0.05	
								0.00	

Figure 31 LTC scan for CCRL Cement 152, w/c=0.35, cured for 1 d under saturated conditions at 20 °C.

Curing: Sealed Age when tested: 1 d Sample mass: 38.5 mg Filename: cem152w35seal1d Date tested: Nov. 2, 2004



Figure 32 LTC scan for CCRL Cement 152, *w/c*=0.35, cured for 1 d under sealed conditions at 20 °C.



Figure 33 LTC scan for CCRL Cement 152, w/c=0.35, cured for 2 d under saturated conditions at 20 °C.

Curing: Sealed Age when tested: 2 d Sample mass: 61.2 mg Filename: cem152w35seal2d Date tested: Nov. 3, 2004



Figure 34 LTC scan for CCRL Cement 152, w/c=0.35, cured for 2 d under sealed conditions at 20 °C.





Curing: Sealed Age when tested: 2 d Sample mass: 64.2 mg Filename: cem152w35seal2d1221 Date tested: Dec. 22, 2004



Figure 36 LTC scan for CCRL Cement 152, *w/c*=0.35, cured for 2 d under sealed conditions at 20 °C (replicate mixture).



Figure 37 LTC scan for CCRL Cement 152, w/c=0.35, cured for 3 d under saturated conditions at 20 °C.



Figure 38 LTC scan for CCRL Cement 152, w/c=0.35, cured for 3 d under sealed conditions at 20 °C.

Curing: Sealed 2 d/resaturated Age when tested: 3 d Sample mass: 51.3 mg Filename: cem152w35sealresat2d1222 Date tested: Dec. 23, 2004



Figure 39 LTC scan for CCRL Cement 152, w/c=0.35, cured at 20 °C for 2 d under sealed conditions, then resaturated for 1 d.



Figure 40 LTC scan for CCRL Cement 152, w/c=0.35, cured for 4 d under saturated conditions at 20 °C.



Figure 41 LTC scan for CCRL Cement 152, w/c=0.35, cured for 4 d under sealed conditions at 20 °C.



Figure 42 LTC scan for CCRL Cement 152, w/c=0.35, cured at 20 °C for 3 d under sealed conditions, then resaturated for 1 d.

Curing: Sealed 4 d/resaturated Age when tested: 5 d Sample mass: 58.2 mg Filename: cem152w35sealresat4d Date tested: Nov. 6, 2004



Figure 43 LTC scan for CCRL Cement 152, w/c=0.35, cured at 20 °C for 4 d under sealed conditions, then resaturated for 1 d.



Figure 44 LTC scan for CCRL Cement 152, w/c=0.35, cured for 5 d under saturated conditions at 20 °C.

Curing: Sealed Age when tested: 5 d Sample mass: 74.2 mg Filename: cem152w35seal5d Date tested: Nov. 6, 2004



Figure 45 LTC scan for CCRL Cement 152, w/c=0.35, cured for 5 d under sealed conditions at 20 °C.



Figure 46 LTC scan for CCRL Cement 152, w/c=0.35, cured for 7 d under saturated conditions at 20 °C.



Figure 47 LTC scan for CCRL Cement 152, w/c=0.35, cured for 7 d under sealed conditions at 20 °C.

Curing: Sealed 5 d/resaturated Age when tested: 7 d Sample mass: 60.4 mg Filename: cem152w35sealresat5d Date tested: Nov. 8, 2004





Cement: CCRL Cement 152 Curing: Sealed 7 d/resaturated Solution: Distilled water Age when tested: 9 d Sample mass: 42.1 mg *w/c*: 0.35 Temperature: 20 °C Filename: cem152w35sealresat7d Date tested: Nov. 10, 2004 Degree of hydration: N/A Temperature (°C) -50 -45 -40 -30 -25 -20 -15 -35 -10 0.025 0.020 (0.00 Heat flow (M/g) 0.010

Figure 49 LTC scan for CCRL Cement 152, *w/c*=0.35, cured at 20 °C for 7 d under sealed conditions, then resaturated for 2 d.



Figure 50 LTC scan for CCRL Cement 152, *w/c*=0.35, cured at 20 °C for 7 d under sealed conditions, frozen during an LTC scan (Figure 17), then resaturated for 1 d.



Figure 51 LTC scan for CCRL Cement 152, w/c=0.35, cured for 9 d under saturated conditions at 20 °C.

Curing: Sealed Age when tested: 9 d Sample mass: 50.1 mg Filename: cem152w35seal9d Date tested: Nov. 10, 2004



Figure 52 LTC scan for CCRL Cement 152, w/c=0.35, cured for 9 d under sealed conditions at 20 °C.





Figure 53 LTC scan for CCRL Cement 152, w/c=0.35, cured for 14 d under saturated conditions at 20 °C.

Curing: Sealed Age when tested: 14 d Sample mass: 46.7 mg Filename: cem152w35seal14d Date tested: Nov. 15, 2004



Figure 54 LTC scan for CCRL Cement 152, *w/c*=0.35, cured for 14 d under sealed conditions at 20 °C.



Figure 55 LTC scan for CCRL Cement 152, w/c=0.35, cured at 20 °C for 14 d under sealed conditions, then resaturated for 1 d.

Curing: Sealed 14 d/resaturated Age when tested: 18 d Sample mass: 35.3 mg Filename: cem152w35sealresatb14d Date tested: Nov. 19, 2004



Figure 56 LTC scan for CCRL Cement 152, *w/*c=0.35, cured at 20 °C for 14 d under sealed conditions, then resaturated for 4 d.



Figure 57 LTC scan for CCRL Cement 152, w/c=0.35, cured for 18 d under saturated conditions at 20 °C.

Curing: Sealed Age when tested: 18 d Sample mass: 47.3 mg Filename: cem152w35seal18d Date tested: Jan. 7, 2005



Figure 58 LTC scan for CCRL Cement 152, w/c=0.35, cured for 18 d under sealed conditions at 20 °C.

Curing: Sealed 17 d/resaturated Age when tested: 18 d Sample mass: 69.0 mg Filename: cem152w35sealresat18d Date tested: Jan. 7, 2005



Figure 59 LTC scan for CCRL Cement 152, w/c=0.35, cured at 20 °C for 17 d under sealed conditions, then resaturated for 1 d.

Cement: CCRL Cement 152 Solution: Distilled water *w/c:* 0.35 Temperature: 20 °C Degree of hydration: N/A Curing: Sealed 14 d/resaturated Age when tested: 21 d Sample mass: 43.6 mg Filename: cem152w35sealresatc14d Date tested: Nov. 22, 2004



Figure 60 LTC scan for CCRL Cement 152, w/c=0.35, cured at 20 °C for 14 d under sealed conditions, then resaturated for 7 d.

Curing: Sealed 14 d/resaturated Age when tested: 29 d Sample mass: 66.5 mg Filename: cem152w35sealresatd14d Date tested: Nov. 30, 2004



Figure 61 LTC scan for CCRL Cement 152, w/c=0.35, cured at 20 °C for 14 d under sealed conditions, then resaturated for 15 d.



Figure 62 LTC scan for CCRL Cement 152, w/c=0.35, cured for 30 d under saturated conditions at 20 °C.



Figure 63 LTC scan for CCRL Cement 152, w/c=0.35, cured for 30 d under sealed conditions at 20 °C.



Figure 64 LTC scan for CCRL Cement 152, w/c=0.35, cured at 20 °C for 30 d under sealed conditions, then resaturated for 2 d.

Curing: Sealed 30 d/resaturated Age when tested: 35 d Sample mass: 91.4 mg Filename: cem152w35sealresatb30d Date tested: Dec. 6, 2004



Figure 65 LTC scan for CCRL Cement 152, w/c=0.35, cured at 20 °C for 30 d under sealed conditions, then resaturated for 5 d.

Curing: Sealed 30 d/resaturated Age when tested: 42 d Sample mass: 34.1 mg Filename: cem152w35sealresatd30d Date tested: Dec. 13, 2004



Figure 66 LTC scan for CCRL Cement 152, w/c=0.35, cured at 20 °C for 30 d under sealed conditions, then resaturated for 12 d.

Cement: CCRL Cement 152 Solution: Distilled water *w/c:* 0.35 Temperature: 20 °C Degree of hydration: N/A Curing: Sealed 30 d/resaturated Age when tested: 49 d Sample mass: 74.2 mg Filename: cem152w35sealresate30d Date tested: Dec. 20, 2004



Figure 67 LTC scan for CCRL Cement 152, w/c=0.35, cured at 20 °C for 30 d under sealed conditions, then resaturated for 19 d.



Figure 68 LTC scan for CCRL Cement 152, w/c=0.35, cured for 50 d under saturated conditions at 20 °C.

Curing: Sealed Age when tested: 50 d Sample mass: 75.1 mg Filename: cem152w35seal50d Date tested: Dec. 21, 2004



Figure 69 LTC scan for CCRL Cement 152, w/c=0.35, cured for 50 d under sealed conditions at 20 °C.





Curing: Sealed 50 d/resaturated Age when tested: 52 d Sample mass: 50.2 mg Filename: cem152w35sealresatb50d Date tested: Dec. 23, 2004



Figure 71 LTC scan for CCRL Cement 152, w/c=0.35, cured at 20 °C for 50 d under sealed conditions, then resaturated for 2 d.



Figure 72 LTC scan for CCRL Cement 152, w/c=0.35, cured at 20 °C for 50 d under sealed conditions, then resaturated for 13 d.

Curing: Sealed 88 d/resaturated Age when tested: 91 d Sample mass: 52.2 mg Filename: cem152w35sealresat91d Date tested: Jan. 31, 2005



Figure 73 LTC scan for CCRL Cement 152, w/c=0.35, cured at 20 °C for 88 d under sealed conditions, then resaturated for 3 d.





Curing: Sealed Age when tested: 91 d Sample mass: 91.3 mg Filename: cem152w35sealb91d Date tested: Jan. 31, 2005



Figure 75 LTC scan for CCRL Cement 152, w/c=0.35, cured for 90 d under sealed conditions at 20 °C.





Curing: Sealed Age when tested: 182 d Sample mass: 53.4 mg Filename: cem152182dseal Date tested: Oct. 25, 2004



Figure 77 LTC scan for CCRL Cement 152, *w/c*=0.35, cured for 182 d at 20 °C under sealed conditions.

Curing: Sealed 7 d/resaturated Age when tested: 182 d Sample mass: 58.6 mg Filename: cem152182dsealsat Date tested: Oct. 25, 2004



Figure 78 LTC scan for CCRL Cement 152, *w/c*=0.35, cured at 20 °C for 7 d under sealed conditions, then resaturated for 175 d.

Cement: CCRL Cement 152 Solution: Distilled water *w/c:* 0.35 Temperature: 20 °C Degree of hydration: N/A Curing: Sealed 182 d/resaturated Age when tested: 183 d Sample mass: 54.3 mg Filename: cem152182dsealresat Date tested: Oct. 26, 2004



Figure 79 LTC scan for CCRL Cement 152, *w/c*=0.35, cured at 20 °C for 182 d under sealed conditions, then resaturated for 1 d.



Figure 80 LTC scan for CCRL Cement 152, w/c=0.35, cured for 204 d at 20 °C under sealed conditions.

Curing: Sealed 203 d/resaturated Age when tested: 204 d Sample mass: 51.1 mg Filename: c152w35sealresat204d Date tested: Nov. 16, 2004



Figure 81 LTC scan for CCRL Cement 152, w/c=0.35, cured at 20 °C for 203 d under sealed conditions, then resaturated for 1 d.

Curing: Sealed 203 d/resaturated Age when tested: 207 d Sample mass: 51.0 mg Filename: c152w35sealresatb204d Date tested: Nov. 19, 2004



Figure 82 LTC scan for CCRL Cement 152, *w/c*=0.35, cured at 20 °C for 203 d under sealed conditions, then resaturated for 4 d.

Cement: CCRL Cement 152 Solution: Distilled water *w/c:* 0.35 Temperature: 20 °C Degree of hydration: 0.799 Curing: Sealed 203 d/resaturated Age when tested: 224 d Sample mass: 66.0 mg Filename: c152w35sealresatc204d Date tested: Dec. 6, 2004



Figure 83 LTC scan for CCRL Cement 152, *w/c*=0.35, cured at 20 °C for 203 d under sealed conditions, then resaturated for 21 d.



Figure 84 LTC scan for CCRL Cement 152, *w/c*=0.35, cured at 20 °C for 203 d under sealed conditions, then resaturated for 35 d.





Curing: Sealed 7 d/resaturated Age when tested: 228 d Sample mass: 68.1 mg Filename: c152w35sealsat228d Date tested: Dec. 10, 2004



Figure 86 LTC scan for CCRL Cement 152, *w/c*=0.35, cured for 7 d at 20 °C under sealed conditions, then resaturated for 221 d.

Curing: Saturated Age when tested: 214 d Sample mass: 48.7 mg Filename: c152w45sat214d Date tested: Oct. 29, 2004



Figure 87 LTC scan for CCRL Cement 152, *w/c*=0.435, cured for 214 d at 20 °C under saturated conditions.

Cement: CCRL Cement 152 Solution: Distilled water *w/c:* 0.435 Temperature: 20 °C Degree of hydration: N/A Curing: Sealed Age when tested: 214 d Sample mass: 55.2 mg Filename: c152w45seal214d Date tested: Oct. 29, 2004



Figure 88 LTC scan for CCRL Cement 152, w/c=0.435, cured for 214 d at 20 °C under sealed conditions.

Curing: Sealed 7 d/resaturated Age when tested: 214 d Sample mass: 54.0 mg Filename: c152w45sealsat214d Date tested: Oct. 29, 2004





Cement: CCRL Cement 152 Solution: Distilled water *w/c:* 0.435 Temperature: 20 °C Degree of hydration: N/A Curing: Sealed 214 d/resaturated Age when tested: 218 d Sample mass: 60.0 mg Filename: c152w45sealresat214d Date tested: Nov. 2, 2004



Figure 90 LTC scan for CCRL Cement 152, *w/c*=0.435, cured for 214 d at 20 °C under sealed conditions, then resaturated for 4 d.

Curing: Sealed 214 d/resaturated Age when tested: 255 d Sample mass: 51.6 mg Filename: c152w45sealresatb214d Date tested: Dec. 9, 2004



Figure 91 LTC scan for CCRL Cement 152, *w/c*=0.435, cured for 214 d at 20 °C under sealed conditions, then resaturated for 41 d.

Cement: CCRL Cement 152 Solution: Distilled water *w/c:* 0.435 Temperature: 20 °C Degree of hydration: N/A Curing: Sealed 204 d/resaturated Age when tested: 238 d Sample mass: 75.3 mg Filename: c152w35seal204resat238d Date tested: Dec. 20, 2004



Figure 92 LTC scan for CCRL Cement 152, w/c=0.435, cured for 204 d at 20 °C under sealed conditions, then resaturated for 34 d.





Figure 93 LTC scan for CCRL Cement 152, w/c=0.435, cured for 256 d at 20 °C under sealed conditions.

Curing: Saturated Age when tested: 276 d Sample mass: 62.2 mg Filename: c152w435sat276d Date tested: Dec. 29, 2004



Figure 94 LTC scan for CCRL Cement 152, w/c=0.435, cured for 276 d at 20 °C under saturated conditions.

Cement: Cement 152 + 20 % limestoneCuring: SaturatedSolution: Distilled waterAge when tested: 91 dw/s: 0.35Sample mass: 59.6 mgTemperature: 20 °CFilename: c152w35lf20sat91dDegree of hydration: 0.855Date tested: Jan. 24, 2005





Cement: Cement 152 + 20 % limestone Solution: Distilled water *w/s:* 0.35 Temperature: 20 °C Degree of hydration: 0.763 Curing: Sealed Age when tested: 91 d Sample mass: 36.5 mg Filename: c152w35lf20seal91d Date tested: Jan. 24, 2005



Figure 96 LTC scan for CCRL Cement 152 with 20 % limestone filler substitution, *w/s*=0.35, cured for 91 d at 20 °C under sealed conditions.
Cement: Cement 152 + 20 % limestone
Solution: Distilled waterCuring: Sealed 91 d/resaturated
Age when tested: 92 d
Sample mass: 64.7 mg
Filename: c152w35lf20sealresat91d
Date tested: Jan. 25, 2005Temperature: 20 °C
Degree of hydration: N/ATemperature: 20 °C
Degree of hydration: N/A



Figure 97 LTC scan for CCRL Cement 152 with 20 % limestone filler substitution, *w/s*=0.35, cured under sealed conditions for 91 d at 20 °C, then resaturated for 1 d.

Cement: Cement 152 + 20 % limes	stone Curing: Sealed 91 d/resaturated						
Solution: Distilled water	Age when tested: 183 d						
<i>w/s</i> : 0.35	Sample mass: 40.5 mg						
Temperature: 20 °C	Filename: c152lf20w35sealresat91t182d						
Degree of hydration: N/A	Date tested: April 26, 2005						
Temperature (°C)							
	05 00 15 10						



Figure 98 LTC scan for CCRL Cement 152 with 20 % limestone filler substitution, w/s=0.35, cured under sealed conditions for 91 d at 20 °C, then resaturated for 92 d.





Figure 99 LTC scan for CCRL Cement 152 with 20 % limestone filler substitution, *w/s*=0.35, cured for 183 d at 20 °C under saturated conditions.

Cement: Cement 152 + 20 % limestone Solution: Distilled water *w/s:* 0.35 Temperature: 20 °C Degree of hydration: 0.828

Curing: Sealed Age when tested: 183 d Sample mass: 65.8 mg Filename: c152lf20w35seal182d Date tested: April 26, 2005



Figure 100 LTC scan for CCRL Cement 152 with 20 % limestone filler substitution, w/s=0.35, cured for 183 d at 20 °C under sealed conditions.



Figure 101 LTC scan for CCRL Cement 152 with 20 % limestone filler substitution, w/s=0.35, cured under sealed conditions for 183 d at 20 °C, then resaturated for 1 d.

Cement: Cement 152 + 20 % limestone
Solution: Distilled water
<i>w/s:</i> 0.435
Temperature: 20 °C
Degree of hydration: N/A

Curing: Saturated Age when tested: 213 d Sample mass: 55.0 mg Filename: c152w45lf20sat213d Date tested: Dec. 23, 2004



Figure 102 LTC scan for CCRL Cement 152 with 20 % limestone filler substitution, *w/s*=0.435, cured for 213 d at 20 °C under saturated conditions.





Cement: Cement 152 + 20 % limestone Solution: Distilled water *w/s:* 0.435 Temperature: 20 °C Degree of hydration: N/A Curing: Sealed 218 d/resaturated Age when tested: 220 d Sample mass: 67.5 mg Filename: c152w45lfseal219d Date tested: Dec. 29, 2004



Figure 104 LTC scan for CCRL Cement 152 with 20 % limestone filler substitution, *w/s*=0.435, cured under sealed conditions for 218 d at 20 °C, then resaturated for 1 d.



Figure 105 LTC scan for CCRL Cement 152, w/c=0.25, cured for 8 h under saturated conditions at 40 °C.

Curing: Saturated Age when tested: 1 d Sample mass: 56.8 mg Filename: c152w025T40Csat1d Date tested: June 1, 2005







Figure 107 LTC scan for CCRL Cement 152, w/c=0.25, cured for 1 d under sealed conditions at 40 °C.

Curing: Saturated Age when tested: 2 d Sample mass: 56.3 mg Filename: c152w025T40Csat2d Date tested: June 2, 2005







Figure 109 LTC scan for CCRL Cement 152, w/c=0.25, cured for 2 d under sealed conditions at 40 °C.

Curing: Sealed 1 d/resaturated Age when tested: 2 d Sample mass: 50.5 mg Filename: c152w025T40Csealresat1t2d Date tested: June 2, 2005



Figure 110 LTC scan for CCRL Cement 152, *w/c*=0.25, cured for 1 d at 40 °C under sealed conditions, then resaturated for 1 d.

Curing: Saturated Age when tested: 3 d Sample mass: 44.0 mg Filename: c152w025T40Csat3d Date tested: June 3, 2005



Figure 111 LTC scan for CCRL Cement 152, w/c=0.25, cured for 3 d under saturated conditions at 40 °C.

Cement: CCRL Cement 152 Solution: Distilled water *w/c:* 0.25 Temperature: 40 °C Degree of hydration: 0.467 Curing: Sealed Age when tested: 3 d Sample mass: 40.0 mg Filename: c152w025T40Cseal3d Date tested: June 3, 2005





Curing: Sealed 2 d/resaturated Age when tested: 3 d Sample mass: 40.3 mg Filename: c152w025T40Csealresat2t3d Date tested: June 3, 2005



Figure 113 LTC scan for CCRL Cement 152, w/c=0.25, cured for 2 d at 40 °C under sealed conditions, then resaturated for 1 d.

Cement: CCRL Cement 152 Solution: Distilled water *w/c:* 0.25 Temperature: 40 °C Degree of hydration: N/A Curing: Sealed 3 d/resaturated Age when tested: 4 d Sample mass: 35.2 mg Filename: c152w025T40Csealresat3t4d Date tested: June 4, 2005

Temperature (°C)								
C	-45	-40	-35	-30	-25	-20	-15	-10
								0.14
								0.12
								0.10 🞅
								<u>0.08</u>
								0.06 Ē
								— 0.04 —
_							— Л	0.02
							~ L	0.00

Figure 114 LTC scan for CCRL Cement 152, w/c=0.25, cured for 3 d at 40 °C under sealed conditions, then resaturated for 1 d.

Curing: Saturated Age when tested: 14 d Sample mass: 48.9 mg Filename: c152w025T40Csat14d Date tested: June 22, 2005



Figure 115 LTC scan for CCRL Cement 152, *w/c*=0.25, cured for 14 d under saturated conditions at 40 °C.

Cement: CCRL Cement 152 Solution: Distilled water *w/c:* 0.25 Temperature: 40 °C Degree of hydration: N/A Curing: Sealed Age when tested: 14 d Sample mass: 55.0 mg Filename: c152w025T40Cseal14d Date tested: June 22, 2005



Figure 116 LTC scan for CCRL Cement 152, w/c=0.25, cured for 14 d under sealed conditions at 40 °C.

Curing: Sealed 14 d/resaturated Age when tested: 15 d Sample mass: 58.7 mg Filename: c152w025T40Csealresat14t15d Date tested: June 23, 2005





Cement: CCRL Cement 152 Solution: Distilled water *w/c:* 0.25 Temperature: 40 °C Degree of hydration: N/A Curing: Saturated Age when tested: 28 d Sample mass: 61.8 mg Filename: c152w025T40Csat28d Date tested: July 6, 2005



Figure 118 LTC scan for CCRL Cement 152, *w/c*=0.25, cured for 28 d under saturated conditions at 40 °C.

Curing: Sealed Age when tested: 28 d Sample mass: 43.4 mg Filename: c152w025T40Cseal28d Date tested: July 6, 2005



Figure 119 LTC scan for CCRL Cement 152, w/c=0.25, cured for 28 d under sealed conditions at 40 °C.

Cement: CCRL Cement 152 Solution: Distilled water *w/c:* 0.25 Temperature: 40 °C Degree of hydration: N/A Curing: Sealed 28 d/resaturated Age when tested: 29 d Sample mass: 59.8 mg Filename: c152w025T40Csealresat28t29d Date tested: July 7, 2005



Figure 120 LTC scan for CCRL Cement 152, w/c=0.25, cured for 28 d at 40 °C under sealed conditions, then resaturated for 1 d.



Figure 121 LTC scan for CCRL Cement 152, w/c=0.35, cured for 1 d under saturated conditions at 40 °C.

Curing: Sealed Age when tested: 1 d Sample mass: 43.5 mg Filename: c152w35T40seal1d Date tested: Jan. 12, 2005







Figure 123 LTC scan for CCRL Cement 152, *w/c*=0.35, cured for 1 d at 40 °C under sealed conditions, then resaturated for 1 d.

Cement: CCRL Cement 152 Solution: Distilled water *w/c:* 0.35 Temperature: 40 °C Degree of hydration: N/A Curing: Sealed 1 d/resaturated Age when tested: 3 d Sample mass: 85.7 mg Filename: c152w35T40sealresatb1d Date tested: Jan. 14, 2005



Figure 124 LTC scan for CCRL Cement 152, *w/c*=0.35, cured for 1 d at 40 °C under sealed conditions, then resaturated for 2 d.



Figure 125 LTC scan for CCRL Cement 152, w/c=0.35, cured for 2 d under saturated conditions at 40 °C.

Curing: Sealed Age when tested: 2 d Sample mass: 77.3 mg Filename: c152w35T40seal2d Date tested: Jan. 13, 2005







Figure 127 LTC scan for CCRL Cement 152, w/c=0.35, cured for 7 d under saturated conditions at 40 °C.

Curing: Sealed Age when tested: 7 d Sample mass: 50.9 mg Filename: c152w35T40seal7d Date tested: Jan. 18, 2005





Curing: Sealed 2 d/resaturated Age when tested: 7 d Sample mass: 76.8 mg Filename: c152w35T40sealresat2t7d Date tested: Jan. 18, 2005



Figure 129 LTC scan for CCRL Cement 152, w/c=0.35, cured for 2 d under sealed conditions at 40 °C, then resaturated for 5 d.

Cement: CCRL Cement 152 Solution: Distilled water *w/c:* 0.35 Temperature: 40 °C Degree of hydration: N/A Curing: Sealed 7 d/resaturated Age when tested: 13 d Sample mass: 52.7 mg Filename: c152w35T40sealresat7t13d Date tested: Jan. 24, 2005



Figure 130 LTC scan for CCRL Cement 152, w/c=0.35, cured for 7 d under sealed conditions at 40 °C, then resaturated for 6 d.

Curing: Saturated Age when tested: 14 d Sample mass: 60.8 mg Filename: c152w35T40sat14d Date tested: Jan. 25, 2005



Figure 131 LTC scan for CCRL Cement 152, *w/c*=0.35, cured for 14 d under saturated conditions at 40 °C.

Cement: CCRL Cement 152 Solution: Distilled water w/c: 0.35Temperature: 40 °C Degree of hydration: 0.645 Curing: Sealed Age when tested: 14 d Sample mass: 62.7 mg Filename: c152w35T40seal14d Date tested: Jan. 25, 2005



Figure 132 LTC scan for CCRL Cement 152, w/c=0.35, cured for 14 d under sealed conditions at 40 °C.



Figure 133 LTC scan for CCRL Cement 152, *w/c*=0.35, cured for 28 d under saturated conditions at 40 °C.

Cement: CCRL Cement 152 Solution: Distilled water *w/c:* 0.35 Temperature: 40 °C Degree of hydration: 0.681 Curing: Sealed Age when tested: 28 d Sample mass: 80.2 mg Filename: c152w035a40Cseal28d Date tested: Feb. 8, 2005



Figure 134 LTC scan for CCRL Cement 152, w/c=0.35, cured for 28 d under sealed conditions at 40 °C.

Curing: Sealed 14 d/resaturated Age when tested: 29 d Sample mass: 51.3 mg Filename: c152w35T40seal14resat29d Date tested: Feb. 9, 2005



Figure 135 LTC scan for CCRL Cement 152, *w/c*=0.35, cured for 14 d under sealed conditions at 40 °C, then resaturated for 15 d.

Cement: CCRL Cement 152 Solution: Distilled water *w/c:* 0.35 Temperature: 40 °C Degree of hydration: N/A Curing: Sealed 28 d/resaturated Age when tested: 29 d Sample mass: 61.5 mg Filename: c152w35T40Csealresat28d Date tested: Feb. 9, 2005



Figure 136 LTC scan for CCRL Cement 152, *w/c*=0.35, cured for 28 d under sealed conditions at 40 °C, then resaturated for 1 d.



Figure 137 LTC scan for CCRL Cement 152, *w/c*=0.35, cured for 58 d under saturated conditions at 40 °C.

Curing: Sealed Age when tested: 58 d Sample mass: 51.3 mg Filename: c152w035T40Cseal58d Date tested: March 10, 2005



Figure 138 LTC scan for CCRL Cement 152, w/c=0.35, cured for 58 d under sealed conditions at 40 °C.

Curing: Sealed 57 d/resaturated Age when tested: 58 d Sample mass: 62.1 mg Filename: c152w035T40Csealresat58d Date tested: March 10, 2005



Figure 139 LTC scan for CCRL Cement 152, *w/c*=0.35, cured for 57 d under sealed conditions at 40 °C, then resaturated for 1 d.

Cement: CCRL Cement 152 Solution: Distilled water *w/c:* 0.35 Temperature: 40 °C Degree of hydration: N/A Curing: Sealed 57 d/resaturated Age when tested: 63 d Sample mass: 71.5 mg Filename: c152w035T40Cresat58t63d Date tested: March 15, 2005



Figure 140 LTC scan for CCRL Cement 152, w/c=0.35, cured for 57 d under sealed conditions at 40 °C, then resaturated for 6 d.





Curing: Sealed Age when tested: 3 d Sample mass: 55.2 mg Filename: c152w45T40Cseal3d Date tested: Feb. 17, 2005

0.015

0.010







Figure 143 LTC scan for CCRL Cement 152, w/c=0.45, cured for 8 d under saturated conditions at 40 °C.

Curing: Sealed Age when tested: 8 d Sample mass: 51.4 mg Filename: c152w45T40Cseal8d Date tested: Feb. 22, 2005



Figure 144 LTC scan for CCRL Cement 152, w/c=0.45, cured for 8 d under sealed conditions at 40 °C.



Figure 145 LTC scan for CCRL Cement 152, w/c=0.45, cured for 3 d under sealed conditions at 40 °C, then resaturated for 5 d.



Figure 146 LTC scan for CCRL Cement 152, *w/c*=0.45, cured for 14 d under saturated conditions at 40 °C.

Cement: CCRL Cement 152 Solution: Distilled water *w/c:* 0.45 Temperature: 40 °C Degree of hydration: 0.799 Curing: Sealed Age when tested: 14 d Sample mass: 44.7 mg Filename: c152w45T40Cseal14d Date tested: Feb. 28, 2005



Figure 147 LTC scan for CCRL Cement 152, w/c=0.45, cured for 14 d under sealed conditions at 40 °C.



Figure 148 LTC scan for CCRL Cement 152, *w/c*=0.45, cured for 3 d under sealed conditions at 40 °C, then resaturated for 11 d.

Cement: CCRL Cement 152 Solution: Distilled water *w/c:* 0.45 Temperature: 40 °C Degree of hydration: N/A Curing: Sealed 8 d/resaturated Age when tested: 14 d Sample mass: 68.6 mg Filename: c152w45T40sealres8t14d Date tested: Feb. 28, 2005



Figure 149 LTC scan for CCRL Cement 152, *w/c*=0.45, cured for 8 d under sealed conditions at 40 °C, then resaturated for 6 d.



Figure 150 LTC scan for CCRL Cement 152, *w/c*=0.45, cured for 29 d at 40 °C under saturated conditions.

Cement: CCRL Cement 152 Solution: Distilled water *w/c:* 0.45 Temperature: 40 °C Degree of hydration: 0.826 Curing: Sealed Age when tested: 29 d Sample mass: 56.7 mg Filename: c152w045T40Cseal28d Date tested: March 15, 2005



Figure 151 LTC scan for CCRL Cement 152, *w/c*=0.45, cured for 29 d at 40 °C under sealed conditions.

Curing: Sealed 28 d/resaturated Age when tested: 29 d Sample mass: 52.2 mg Filename: c152w045T40Cresat28t29d Date tested: March 15, 2005



Figure 152 LTC scan for CCRL Cement 152, *w/c*=0.45, cured for 28 d under sealed conditions at 40 °C, then resaturated for 1 d.

CCRL Cement 140

Cement: CCRL Cement 140 Solution: Distilled water *w/c:* 0.40 Temperature: 20 °C Degree of hydration: N/A Curing: Saturated Age when tested: 2 d Sample mass: 64.7 mg Filename: c140w4nalk2d Date tested: Dec. 30, 2004



Figure 153 LTC scan for CCRL Cement 140, w/c=0.40, prepared with distilled water and cured for 2 d at 20 °C under saturated conditions.



Figure 154 LTC scan for CCRL Cement 140, w/c=0.40, prepared with a solution of alkali sulfates and cured for 2 d at 20 °C under saturated conditions.

Cement: CCRL Cement 140	Curing: Saturated								
Solution: Alkali hydroxide solution	Age when tested: 2 d								
<i>w/c</i> : 0.40	Sample mass: 62.8 mg								
Temperature: 20 °C	Filename: c140w4walkoh2d								
Degree of hydration: N/A	Date tested: Dec. 30, 2004								
Temperature (°C)									
D 4E 40 DE DO 4	DE 20 4E 40								



Figure 155 LTC scan for CCRL Cement 140, w/c=0.40, prepared with a solution of alkali hydroxides and cured for 2 d at 20 °C under saturated conditions.



Figure 156 LTC scan for CCRL Cement 140, w/c=0.40, prepared with a LiOH solution and cured for 2 d at 20 °C under saturated conditions.

Cement: CCRL Cement 140 Solution: LiNO₃ solution w/c: 0.40 Temperature: 20 °C Degree of hydration: 0.472 Curing: Saturated Age when tested: 2 d Sample mass: 48.0 mg Filename: c140wlino3a2d Date tested: Feb. 3, 2005



Figure 157 LTC scan for CCRL Cement 140, w/c=0.40, prepared with a LiNO₃ solution and cured for 2 d at 20 °C under saturated conditions.



Figure 158 LTC scan for CCRL Cement 140, w/c=0.40, prepared with a LiOH solution and cured for 4 d at 20 °C under saturated conditions.

Cement: CCRL Cement 140 Solution: LiNO₃ solution *w/c*: 0.40 Temperature: 20 °C Degree of hydration: N/A Curing: Saturated Age when tested: 4 d Sample mass: 81.0 mg Filename: c140w04lino3a4d Date tested: Feb. 5, 2005







Figure 160 LTC scan for CCRL Cement 140, w/c=0.40, prepared with a LiOH solution and cured for 7 d at 20 °C under saturated conditions.

Cement: CCRL Cement 140 Solution: LiNO₃ solution *w/c*: 0.40 Temperature: 20 °C Degree of hydration: 0.686 Curing: Saturated Age when tested: 7 d Sample mass: 67.3 mg Filename: c140w04lino3a7d Date tested: Feb. 8, 2005



Figure 161 LTC scan for CCRL Cement 140, w/c=0.40, prepared with a LiNO₃ solution and cured for 7 d at 20 °C under saturated conditions.



Figure 162 LTC scan for CCRL Cement 140, w/c=0.40, prepared with distilled water and cured for 8 d at 20 °C under saturated conditions.

Cement: CCRL Cement 140 Solution: Alkali sulfate solution w/c: 0.40 Temperature: 20 °C Degree of hydration: 0.631 Curing: Saturated Age when tested: 8 d Sample mass: 42.5 mg Filename: c140w4walk8d Date tested: Jan. 5, 2005



Figure 163 LTC scan for CCRL Cement 140, w/c=0.40, prepared with a solution of alkali sulfates and cured for 8 d at 20 °C under saturated conditions.

Cement: CCRL Cement 140 Solution: Alkali hydroxide solution <i>w/c:</i> 0.40 Temperature: 20 °C Degree of hydration: 0.661							Curing: Saturated Age when tested: 8 d Sample mass: 61.0 mg Filename: c140w4walkoh8d Date tested: Jan. 5, 2005			
	Temperature (°C)									
-5	0 -4	15 -4	40 -3	35 -3	30 -2	25 -2	20 -1	15 -1	0	
ŀ					1				- 0.030	
-		/ 1							- 0.025	
-		7 3 F 3	<u></u>						- 0.020	
	*		91 By em	en des <u>re</u> des	an an 19 = .	¹⁰ ¹⁰ ¹⁰ ¹⁰ ¹⁰	· · · · ·		- 0.015 eat flo	
-									- 0.010 [±]	
Į									- 0.005	

Figure 164 LTC scan for CCRL Cement 140, w/c=0.40, prepared with a solution of alkali hydroxides and cured for 8 d at 20 °C under saturated conditions.







Figure 166 LTC scan for CCRL Cement 140, w/c=0.40, prepared with a solution of LiNO₃ and cured for 8 d at 20 °C under saturated conditions.

Curing: Saturated Age when tested: 14 d Sample mass: 58.9 mg Filename: c140w04nalk14d Date tested: Jan. 11, 2005




C S W T L	Ceme Soluti v/c: (Temp Degre	ent: CCl ion: All 0.40 perature ee of hy	RL Cen kali sult : 20 °C rdration	nent 14 fate solu : 0.655	Curin Age Samp Filen Date	Curing: Saturated Age when tested: 14 d Sample mass: 48.7 mg Filename: c140w04walk14d Date tested: Jan. 11, 2005				
				Tempe	rature (°C)				
-50	-4	-5 -4	40 -	35 -	-25	-20	-15	-10		
						1			0.030	
									0.025	
	-	/ `,							0.020	δ///) w
						~ ~			0.015	eat flo
								100	0.010	T
									0.005	

Figure 168 LTC scan for CCRL Cement 140, w/c=0.40, prepared with a solution of alkali sulfates and cured for 14 d at 20 °C under saturated conditions.



Figure 169 LTC scan for CCRL Cement 140, w/c=0.40, prepared with a solution of alkali hydroxides and cured for 14 d at 20 °C under saturated conditions.



Figure 170 LTC scan for CCRL Cement 140, *w/c*=0.40, prepared with a solution of LiOH and cured for 14 d at 20 °C under saturated conditions.

Cement: CCRL Cement 140 Solution: LiNO₃ solution w/c: 0.40 Temperature: 20 °C Degree of hydration: 0.736 Curing: Saturated Age when tested: 14 d Sample mass: 53.1 mg Filename: c140w04lino3a14d Date tested: Feb. 15, 2005



Figure 171 LTC scan for CCRL Cement 140, w/c=0.40, prepared with a solution of LiNO₃ and cured for 14 d at 20 °C under saturated conditions.



Figure 172 LTC scan for CCRL Cement 140, w/c=0.40, prepared with distilled water and cured for 30 d at 20 °C under saturated conditions.



Figure 173 LTC scan for CCRL Cement 140, w/c=0.40, prepared with a solution of alkali sulfates and cured for 30 d at 20 °C under saturated conditions.

	Ceme Solut w/c: (Temp	ent: CC ion: All 0.40 perature	RL Cen cali hyd : 20 °C	nent 140 roxide s	Curing: Saturated Age when tested: 30 d Sample mass: 75.5 mg Filename: c140w04walkoh30d				
	Degre	ee of hy	dration	0.738	Date tested: Jan. 27, 2005				
				Temper	C)				
50	-4	-4	40 -3	35 -3	30 -2	25 -2	20 -1	15 -1	10
									- 0.030

Figure 174 LTC scan for CCRL Cement 140, w/c=0.40, prepared with a solution of alkali hydroxides and cured for 30 d at 20 °C under saturated conditions.

Cement: CCRL Cement 140 Solution: LiOH solution *w/c:* 0.40 Temperature: 20 °C Degree of hydration: 0.761 Curing: Saturated Age when tested: 30 d Sample mass: 57.9 mg Filename: c140w04lioh30d Date tested: March 3, 2005

0.025

0.020

0.015

0.010

0.005

Heat flow (W/g)







Figure 176 LTC scan for CCRL Cement 140, w/c=0.40, prepared with a solution of LiNO₃ and cured for 30 d at 20 °C under saturated conditions.

Cement: CCRL Cement 140 Solution: Distilled water *w/c:* 0.40 Temperature: 20 °C Degree of hydration: 0.82 Curing: Saturated Age when tested: 63 d Sample mass: 66.8 mg Filename: C140w4nalksat63d Date tested: March 1, 2005







Figure 178 LTC scan for CCRL Cement 140, w/c=0.40, prepared with a solution of alkali sulfates and cured for 63 d at 20 °C under saturated conditions.

	Ceme Solut w/c: Temp Degre	ent: CC ion: All 0.40 perature ee of hy	RL Cen kali hyd : 20 °C rdration	nent 140 roxide s : 0.815	Curing Age w Sampl Filena Date t	g: Satura when test e mass: me: C14 ested: N	ated ted: 63 o 48.5 m 40w4wa farch 1,	d g ılkohsat63d 2005	
				Temper	C)				
50	_4	-4	40 -3	35 -3	30 -2	25 -2	20 -1	5 -1	0
F									- 0.030
									- 0.025
_	= r ^p =	1 1 15	* *	= = ₁₀ gr	⁹ = = =	=			- 0.020 (6/))
							8 10 AL AL A	1 80 1 10 10 10	eat ilo
									т - 0.010

Figure 179 LTC scan for CCRL Cement 140, w/c=0.40, prepared with a solution of alkali hydroxides and cured for 63 d at 20 °C under saturated conditions.

0.005



Figure 180 LTC scan for CCRL Cement 140, w/c=0.40, prepared with a solution of LiOH and cured for 63 d at 20 °C under saturated conditions.



Figure 181 LTC scan for CCRL Cement 140, w/c=0.40, prepared with a solution of LiNO₃ and cured for 63 d at 20 °C under saturated conditions.



Figure 182 LTC scan for CCRL Cement 140, w/c=0.40, prepared with distilled water and cured for 90 d at 20 °C under saturated conditions.



Figure 183 LTC scan for CCRL Cement 140, w/c=0.40, prepared with distilled water and cured for 91 d at 20 °C under saturated conditions.



Figure 184 LTC scan for CCRL Cement 140, w/c=0.40, prepared with a solution of alkali sulfates and cured for 90 d at 20 °C under saturated conditions.

Cement: CCRL Cement 140 Solution: Alkali sulfate solution *w/c:* 0.40 Temperature: 20 °C Degree of hydration: N/A Curing: Saturated Age when tested: 91 d Sample mass: 74.6 mg Filename: c140w04walk91d Date tested: March 29, 2005



Figure 185 LTC scan for CCRL Cement 140, w/c=0.40, prepared with a solution of alkali sulfates and cured for 91 d at 20 °C under saturated conditions.



Figure 186 LTC scan for CCRL Cement 140, w/c=0.40, prepared with a solution of alkali hydroxides and cured for 90 d at 20 °C under saturated conditions.



Figure 187 LTC scan for CCRL Cement 140, w/c=0.40, prepared with a solution of LiOH and cured for 90 d at 20 °C under saturated conditions.



Figure 188 LTC scan for CCRL Cement 140, w/c=0.40, prepared with a solution of LiNO₃ and cured for 90 d at 20 °C under saturated conditions.

Cement: CCRL Cement 140 Solution: Distilled water *w/c:* 0.40 Temperature: 20 °C Degree of hydration: N/A Curing: Saturated Age when tested: 101 d Sample mass: 56.4 mg Filename: c140p101d Date tested: July 18, 2002



Figure 189 LTC scan for CCRL Cement 140, w/c=0.40, prepared with distilled water and cured for 101 d at 20 °C under saturated conditions.





Cement: CCRL Cement 140				
Solution: Alkali sulfate solution				
<i>w/c</i> : 0.40				
Temperature: 20 °C				
Degree of hydration: N/A				

Curing: Saturated Age when tested: 101 d Sample mass: 64.2 mg Filename: c140walk101d Date tested: July 18, 2002



Figure 191 LTC scan for CCRL Cement 140, w/c=0.40, prepared with a solution of alkali sulfates and cured for 101 d at 20 °C under saturated conditions.

Cement: CCRL Cement 140 Solution: Alkali sulfate solution w/c: 0.40 Temperature: 20 °C Degree of hydration: N/A	Curing: Saturated Age when tested: 110 d Sample mass: 60.6 mg Filename: c140walk110d Date tested: July 29, 2002				
Temperature	e (°C)				
-50 -45 -40 -35 -30	-25 -20 -15 -10				
	0.040				
	0.035				
	0.030 g				
	0.025 💐				
	0.020				
	0.015 🛱				
	0.010				
	0.005				

Figure 192 LTC scan for CCRL Cement 140, w/c=0.40, prepared with a solution of alkali sulfates and cured for 110 d at 20 °C under saturated conditions.

Observations

- 1) For low w/c, sealed curing first depercolates capillary pores, but then later repercolates them due to self-desiccation stresses, internal shrinkage, and possibly microcrack formation [2]. The sealed/resaturated scans for cement 152, w/c = 0.35 in Figures 39, 42, 43, 48, 49, 55, 59, 64, 71, 73, 79, and 81 illustrate this process for curing at 20 °C. For the small cement paste specimens employed in these studies, continuing resaturation of the specimens cured first under sealed conditions did result in a second depercolation of the repercolated capillary (size) pores; this is illustrated in Figures 67, 72, and 83. In Figures 83 and 84, however, the repercolated open gel (size) pores did not exhibit a second depercolation upon extended resaturation from 204 d to 224 d or 238 d. For curing at 40 °C, equivalent depercolation/repercolation behavior is indicated in Figures 124, 130, 135, 136, and 140. For cement pastes with a 20 % by mass fraction limestone substitution, equivalent repercolation behavior was observed (Figures 97 and 101), but without any evidence of a subsequent second depercolation of the capillary pores (Figure 98), perhaps due to the higher effective w/c in the specimens with the limestone substitution.
- 2) For extremely low w/c = 0.25 at 20 °C, this repercolation was observed both for specimens cured under sealed conditions and then resaturated (Figures 8, 11, 15, 18, 21, 24, 27, and 30) and for specimens cured under nominally "saturated" (water ponded on top) conditions (Figures 4, 6, 9, 13, 16, 19, 22, 25, and 28). Most likely, for this extremely low w/c, it was not possible to maintain saturated conditions using the techniques employed in this study. Similar repercolation observations apply for the 40 °C curing for the w/c = 0.25 cement pastes, as indicated in Figures 117, 118, and 120.
- 3) For saturated curing, **more** time (and hydration) is required to depercolate the pores in a w/c = 0.35 cement 152 paste when cured at 40 °C, as opposed to at 20 °C, in spite of the fact that the higher temperature curing significantly accelerates the cement hydration reactions. This suggests the formation of a coarser capillary pore structure when curing at higher temperatures in agreement with conventional wisdom [9]. For 20 °C curing under saturated conditions, depercolation of the capillary size pores occurred between 3 d (Figure 37) and 4 d (Figure 40) of curing. Conversely, for 40 °C saturated curing, this same depercolation occurred between 7 d (Figure 127) and 14 d (Figure 131).
- 4) For intermediate w/c (e.g., 0.40 to 0.45), it appears that some length of sealed curing followed by resaturation may be superior to saturated curing from the time of initial casting, in terms of providing an earlier depercolation of the capillary pores. Evidence for this can be found in Figures 146, 148, and 149 for the cement 152 w/c=0.45 cement pastes cured at 40 °C for 14 d and in Figures 87, 89, and 90 for the cement 152 w/c=0.435 cement pastes cured at 20 °C. This effect could be due to the fact that under the initial sealed curing conditions, cement hydration will be localized in the pore entryways and smaller pores and will not be occurring in the larger pores that are emptying due to self-desiccation (from the chemical shrinkage accompanying the hydration reactions). Figure 193 provides a simple 4-particle model of cement hydration illustrating this hypothesis.

5) For the low-alkali cement 140 pastes with w/c = 0.40, repercolation of the capillary and open gel pores was observed to occur at later ages even under saturated curing conditions, as exemplified by the LTC scans in Figures 172, 177, 182, 183, 189, and 190. It is likely that the C-S-H gel formed in this hydrating low-alkali cement paste is very amorphous with a highly random morphology; thus, subsequent rearrangement to a more ordered (nano)structure could be responsible for local shrinkage and the observed repercolation of the open gel and capillary (size) pores [3, 4]. Conversely, the addition of alkalis (lithium, sodium, or potassium) results in an earlier depercolation of the capillary pores as indicated by comparing Figures 162 to 166 for specimens cured for 8 d under saturated conditions. Furthermore, limited evidence of a later age repercolation is observed only for the specimens with potassium and sodium sulfate additions, as shown in Figures 191 and 192. The specimens with alkali hydroxide or lithium nitrate additions cured for 90 d under saturated conditions exhibited no evidence of either an open gel or capillary pore repercolation (Figures 186, 187, and 188).



Figure 193 Four particle model for hydrating cement paste microstructure, indicating hydration under saturated and sealed curing conditions. Dark grey is unhydrated cement grains, textured material and light grey are hydration products, white is water-filled porosity, and black is empty porosity, from reference [2].

Conclusions and Future Research

Low temperature calorimetry has been shown to be a valuable tool for characterizing the porosity of hydrating cement pastes, both in terms of the presence/absence of freezable water as a function of temperature and the percolation state of pore networks with various size entryways (dense gel, open gel, and capillary). Numerous examples of the influences of cement type, w/c, curing temperature, curing time, curing saturation conditions, and the additions of alkali compounds or limestone fillers have been presented in this report. Future efforts will focus on extending these LTC and hydration studies to blended cements to examine the influence of additions of silica fume, slag, fly ash, and the like on the developing porosity. In addition, studies are also underway on the influence of several different shrinkage-reducing admixture (SRA) types and dosages on the freezable water content of hydrating cement pastes.

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