Cement & Concrete Testing Workshop Part I:
An Introduction to Portland Cement and Slag Cement Specifications

Understanding ASTM International Test Procedures for Cement and Concrete - Staying Up to Standard

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December 14, 2016

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Cement & Concrete Testing Workshop Part I: Presentation Overview

ASTM C150 – Portland Cement

Physical Test Methods

- Fineness
  - ASTM C204 – Air Permeability Test
- Consistency
  - ASTM C1437 – Flow
  - ASTM C187 – Normal Consistency
- Chemical Requirements
  - Chemical analysis
  - Compound composition

ASTM C989 – Slag Cement

- Chemical composition
- Physical Requirements

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Hydraulic Cement Specifications

- Hydraulic Cements for General Concrete Construction
  - ASTM C10 – Natural Cement
  - ***ASTM C150 – Portland Cement***
  - ASTM C595 – Blended Hydraulic Cements
  - ASTM C1157 – Hydraulic Cement

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ASTM C150 Requirements

Chemical Requirements
Chemical analysis
Compound composition
Chemical limits

Physical Requirements
Fineness
Soundness
Consistency (Flow and Normal Consistency)
Setting Time
False set and flash set
Compressive strength
Heat of hydration
Loss on ignition
Density
Air content
Sulfate expansion

Table 3: Physical Requirements

Cement Type	Applicable Test Method	I	II	III	IV	V
Air content of mortar, volume % (Max)						
max	C185	12	12	12	12	12
min		…	…	…	…	…
Fineness, specific surface, m^2/kg						
max	C204	260	260	…	260	260
min		…	…	…	430	…
Autoclave expansion, max %	C151	0.80	0.80	0.80	0.80	0.80
Strength, not less than the values shown for the ages indicated as Compressive strength, Mpa (psi)						
1 day	C109	…	…	12.0 [1740]	…	…
3 days		12.0 [1740]	10.0 [1450]	24.0 [3480]	…	8.0 [1160]
7 days		19.0 [2760]	17.0 [2320]	…	7.0 [1020]	15.0 [2180]
28 days		…	…	…	17.0 [2470]	21.0 [3050]
Time of Setting, Vicat Test						
Time of Setting, min.	C191	45	45	45	45	45
Time of Setting, max.		375	375	375	375	375

Presentation Outline

ASTM C150 – Portland Cement

Physical Test Methods

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- Consistency
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ASTM C204 - Air Permeability Test

- Determine fineness of cement
- Very important physical property
  - Affects setting time, hydration rate, strength, shrinkage, heat of hydration, and permeability
- Increase in fineness
  - Increases rate of hydration, shortens setting time, increases early-age strength gain
  - Affects consistency of mixtures and admixture demand
- Required for ASTM C150 and C989

Fineness Standards

ASTM C204 – Blaine Surface Area

ASTM C115 – Turbidimeter

ASTM C430 – No. 325 Sieve

Scope of Test Method

- Determination of <u>fineness</u> using Blaine <u>air permeability apparatus</u>
- Result is in surface area of cm^2/g or m^2/kg
- Results are considered <u>relative</u> values, not absolute
- Known to work well for portland cement. Should be used with caution on other materials.

Nature of Apparatus

Calibration of Apparatus

- Calibration of the air permeability apparatus shall be made using the current lot of standard material:
  - NIST Standard Reference Material No. 114
- Determine Bulk Volume of compacted Bed of Powder by Physical Measurement:
  - Measure average diameter and average cell depth

$$\boldsymbol{Volume = \pi r^2 h}$$

Note: Bulk volume can also be determined by mercury displacement method. Not covered in this presentation.

Calibration of Apparatus

- Prepare sample by shaking in a sample jar, let stand for 2 minutes, gently stir the sample.
- Use equation to determine the mass of sample required to have a porosity of 0.500

$$\mathbf{W} = \boldsymbol{\rho}\mathbf{V}(\mathbf{1} - \varepsilon)$$

W = grams of sample required
ρ = density of sample for portland cement 3.15
V = bulk volume
ε = desired porosity of bed of cement 0.500 ± 0.005

Procedure: Preparation of Cement Bed

Procedure: Preparation of Cement Bed (1/3)

1. Seat Perforated Disc

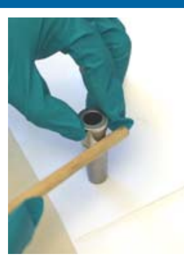
2. Place Filter Paper on Metal Disc

3. Press Edges Down with Rod

Procedure: Preparation of Cement Bed (2/3)

4. Measure Cement Mass to Nearest 0.001g

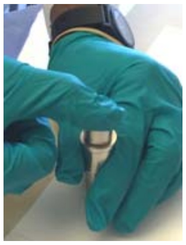
5. Place Cement Sample in Cell



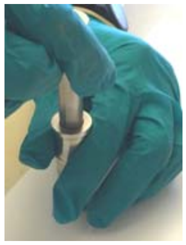
6. Tap Side to Level Cement

Procedure: Preparation of Cement Bed (3/3)

7. Place Filter Paper on Top of Cement



8. Compress Sample with Plunger Until Collar is in Contact with Top of Cell



9. Withdraw Short Distance, Rotate 90°, Recompress, and Withdraw

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Procedure: Permeability Test

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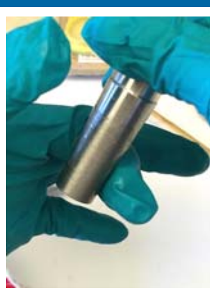
Procedure: Permeability Test (1/4)



1. Apply Stopcock Grease to Tapered Connection

2. Attach Cell to Manometer Tube

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Procedure: Permeability Test (2/4)

3. Evacuate Air from One Arm Until Liquid Reaches Top Mark

4. Close Air Valve

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Procedure: Permeability Test (3/4)

5. Start Timer when Liquid Reaches Next Mark Down from Top (2nd Mark)

6. Stop Timer when Liquid Reaches Next Mark Down from Top (3rd mark)

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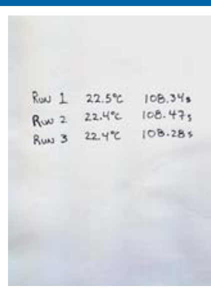
Procedure: Permeability Test (4/4)

7. Record Time Interval for Test in Seconds and Temperature at Time of Testing in °C.



8. Repeat 3 Times and Recalibrate No Less than Every 2.5 Years

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Procedure: About Cement and Other Materials

- Cement shall be at room temperature for test
- Same weight of sample as used with calibration sample:
  - For type III cement, or other fine ground cements, the bulk of material will be too great for thumb pressure on plunger to compress sample – use a porosity of 0.530 for sample mass calculation
  - For other materials, or cements where 0.500 and 0.530 won't work, adjust mass of sample such that a firm bed of material is produced with thumb pressure only. Plunger must not rebound once thumb is removed.

Calculation (section 6) and Report (section 7)

$$S = \frac{S_s \sqrt{T}}{\sqrt{T_s}}$$

- Round values to nearest 10 cm^2/g or nearest 1 m^2/kg
- Report results of single trial
- For high fineness materials report the average of 2 trials (if trials are within 2% of each other). If trials are not within 2% discard values and repeat until 2 values are within 2%

Fineness

- Requirements for Type I, II, IV & V
  - (No requirements for Type III)

	Air Permeability
Minimum, m^2/kg	280
Maximum, m^2/kg	400
Typical Values, m^2/kg	350-380 Type I 450-600 Type III

- No limits for blended cement (ASTM C 595), hydraulic cements (ASTM C 1157), or slag cement (ASTM C989) but values must be reported

Limitations and Errors

- Specific surface area does NOT characterize the particle size distribution of a cement.
- Watch for air leaks, continuous loss of pressure in the manometer.
- Care should be taken when testing other materials than portland cement.
- Reporting temperature and comparing it to calibration temperature.

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Presentation Outline

ASTM C150 – Portland Cement

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- Consistency
  - ***ASTM C1437 – Flow***
  - ASTM C187 – Normal Consistency
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ASTM C1437 – Flow of Mortar Samples

- Determine consistency (flow behavior) of a fresh mixture
- Consistency depends on:
  - Fineness, flocculation of cement and cement with aggregates, and setting times
- Consistency is used for:
  - Strength (C109), air content (C185) and Sulfate Expansion (C1038) [Optional]
  - Consistency used instead of w/cm to ensure quality samples.

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Apparatus: Flow Table, Flow Mold, and Caliper (1/2)

- Flow Table

Diameter: 255 ± 2.5 mm

Raised Height: 12.7 ± 0.13 mm

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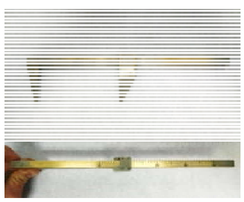
Apparatus: Flow Table, Flow Mold, and Caliper (2/2)

- Conical Mold and Caliper

Diameter Top: 70 ± 0.5 mm
Diameter Bottom: 100 ± 0.5 mm
Height: 50 ± 0.5 mm



Distance to Zero: 100 ± 0.25 mm

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Apparatus: Other

- **Tamper:** conforming to the requirements of Test Method C109. The tamping face of the tamper shall be flat and at right angles to the length of the tamper.
- **Steel Straightedge:** not less than 200 mm long and not less than 1.5 mm nor more than 3.5 mm in thickness.
- **Trowel:** steel blade 100 to 150 mm long

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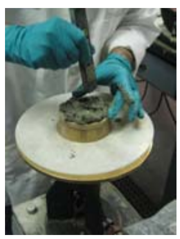
Procedure: Determination of Flow

Procedure: Determination of Flow (1/3)

1. Place 25 mm Layer

2. Tamp 20 Times. Incline Tamper when Near Perimeter.



3. Fill Mold and Tamp as Specified.

Procedure: Determination of Flow (2/3)

4. Cut off Mortar Plane with Sawing Motion with Straightedge or Trowel.

5. Wipe off Table Top.

Procedure: Determination of Flow (3/3)

6. Lift Mold 1 Minute after Mixing.

7. Immediately Drop the Table 25 times in 15s.

8. Measure the Diameter Across Four Scribed Lines.

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Procedure: Calculation

- Flow is the resulting increase in average base diameter of the mortar mass, expressed as a percentage of the original base.
- Using ASTM C230 Caliper, add the four readings, and record the total. This gives the flow in percent.

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Flow Requirements for Related Tests

- ASTM C109 – Compressive Strength
  - Record flow for portland cements and air-entraining portland
  - 110 ± 5 % for other than portland and air-entraining portland cements
- ASTM C185 – Air Content
  - Drop table 10 times
  - 87 ½ ± 7 ½ %
- ASTM C1038 – Sulfate Resistance (Optional)
  - 110 ± 5 %

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Limitations and Errors

- Gauging time should be strictly observed.
- Room temperature should be well maintained as per test requirement.
- All apparatus used should be clean.
- Uniformly compress and fill conical mold.
- Over-tamping causes water to extrude towards bottom of molds and causes increased (erroneous) flow.

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ASTM C150 – Portland Cement

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  - ***ASTM C187 – Normal Consistency***

Chemical Requirements

- Chemical analysis
- Compound composition

ASTM C989 – Slag Cement

- Chemical composition
- Physical requirements

Significance of ASTM C187 – Consistency

- Determine amount of water required to achieve a standard consistency
- Measures consistency through penetration resistance
- Required for:
  - ASTM C191 - Time of Setting (Vicat)
  - ASTM C151 - Autoclave

Scope/Significance and Use

- ***Scope:*** This test method covers the determination of the normal consistency of hydraulic cement
- ***Significance and Use:*** This test method is intended to be used to determine the amount of water required to prepare hydraulic cement pastes with normal consistency, as required for certain standard tests.

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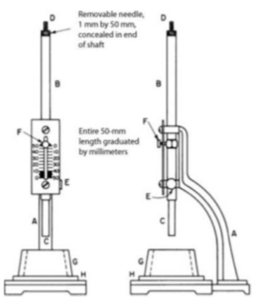
Apparatus: Vicat Apparatus



Source: ASTM C187

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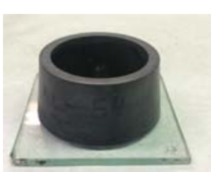
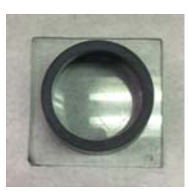
Apparatus: Ring

- Inside diameter of ring at bottom 70 ± 3 mm
- Inside diameter of ring at top 60 ± 3 mm
- Height of ring 40 ± 1 mm





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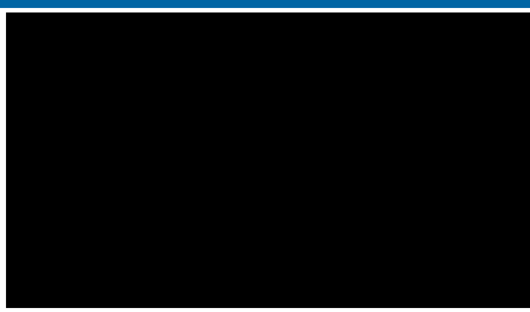
Procedure: Mixing

- Mix 650 g of cement with a measured quantity of water following the procedure prescribed in the Procedure for Mixing Pastes and Practice C305.
- The water shall conform to the numerical limits of Specification D1193 for Type III or Type IV grade of reagent water.

Procedure: Molding Test Specimens



Procedure: Molding Test Specimens (1/2)

1. Mix per ASTM C305

2. Toss 6 Times

3. Press Ball in Larger End of Conical Ring

Procedure: Molding Test Specimens (2/2)

4. Place Conical Ring on Base Plate

5. Slice Off Excessive Paste

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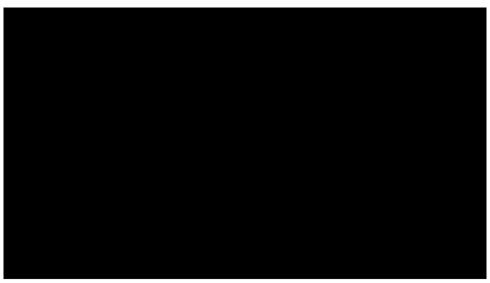
Procedure: Consistency Determination



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Procedure: Consistency Determination

1. Center Ring and Base Plate under Rod

2. Place Plunger in Contact with Paste and Zero Reading

3. Release the Plunger. Time not Exceeding 30s after Mixing.

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Calculation

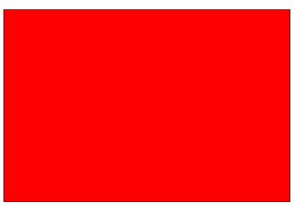
- Calculate the amount of water required for normal consistency as the mass of water divided by the mass of dry cement, expressed as a percentage.
- Calculate the mass ratio to the nearest 0.1% and report the mass ratio to the nearest 0.5%.

$$P = \frac{Quantity\ Water\ (g)}{Quantity\ Cement\ (g)}\ X\ 100$$

P = Percentage of Water(%)

Normal Consistency Requirements for Related Tests

- ASTM C151 – Time of Setting (Vicat)
- ASTM C191 – Autoclave Expansion



Limitations and Errors

- Gauging time should be strictly observed.
- Room temperature should be well maintained as per test requirement.
- All apparatus used should be clean.
- The experiment should be performed away from vibrations and other disturbances.
- Do not compress paste in conical ring.
- Mixtures containing SCMs (especially Silica Fume) normal consistency are very sensitive to water.

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Chemical Requirements

- *Chemical analysis*
- *Compound composition*

ASTM C989 – Slag Cement

- Chemical composition
- Physical requirements

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Portland Cement Types (ASTM C150)

- Type I – Normal
  - General-purpose portland cement
- Type II – Moderate Sulfate Resistance
  - Used where protection from sulfate is required for structures exposed to soil or ground water

Severity of potential exposure	Water-soluble sulfate (SO_4) in soil, % by mass*	Sulfate (SO_4)* in water, ppm	w/cm by mass, max.†‡	Cementitious material requirements
Class 0 exposure	0.00 to 0.10	0 to 150	No special requirements for sulfate resistance	No special requirements for sulfate resistance
Class 1 exposure	> 0.10 and < 0.20	> 150 and < 1500	0.50‡	C150 Type II or equivalent§
Class 2 exposure	0.20 to < 2.0	1500 to < 10,000	0.45‡	C150 Type V or equivalent§
Class 3 exposure	2.0 or greater	10,000 or greater	0.40‡	C150 Type V plus pozzolan or slag§

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Portland Cement Types (ASTM C150)

- Type III – High early strength
  - Chemically similar to Type I, but ground finer
  - Used in precast construction
- Type IV – Low heat of hydration
  - Mass concrete structures
  - Not commonly manufactured in North America
- Type V – High Sulfate resistance

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Composition

Chemical Name	Chemical Formula	Notation	Mass (%)
Tricalcium silicate	$3CaO{\bullet}SiO_2$	C_3S	50-70
Dicalcium silicate	$2CaO{\bullet}\ SiO_2$	C_2S	15-30
Tricalcium aluminate	$3CaO{\bullet}Al2O_3$	C_3A	5-10
Tetracalcium aluminoferrite	$4CaO{\bullet}Al_2O_3{\bullet}Fe_2O_3$	C_4AF	5-15
Calcium sulfate dihydrate	$CaSO_4.2H_2O$	CSH_2	~ 5

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Composition

- The relative quantities of each of these phases affects:
  - setting time
  - rate of strength development
  - overall strength
  - durability
  - color
- It is important, then, to know the composition of the cement.

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Chemical Analysis: Determine Composition

- X-Ray Fluorescence (XRF) Spectroscopy
  - Provides bulk elemental composition of materials
  - Results are used for Bogue calculations
- X-ray Powder Diffraction (XRD)
  - Rapid analytical technique used for phase identification of a crystalline material
  - Rietveld refinement used to analyze results and provide more precise portland cement phases

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Chemical Analysis

Oxide	Element
SiO_2	Silicon dioxide
Al_2O_3	Aluminum oxide
Fe_2O_3	Ferric oxide
CaO	Calcium oxide
MgO	Magnesium oxide
SO_3	Sulfur trioxide
LOI	Loss on ignition
Na_2O	Sodium oxide
K_2O	Potassium oxide
TiO_2	Titanium dioxide
P_2O_5	Phosphorus pentoxide
ZnO	Zinc oxide
Mn_2O_3	Manganic oxide
Sulfide sulfur	

ASTM C 114 - Standard Test Methods for Chemical Analysis of Hydraulic Cement

Major Components (SiO_2 through LOI)

Minor Components (Na_2O through Sulfide sulfur)

Chemical Analysis

Oxide	%
SiO_2	20.6
Al_2O_3	5.07
Fe_2O_3	2.90
CaO	63.9
MgO	1.53
SO_3	2.53
Na_2O	0.15
K_2O	0.73
LOI	1.58

90-95% (SiO_2 through MgO)

Oxide	Shorthand	Common Name
CaO	C	Lime
SiO_2	S	Silica
Al_2O_3	A	Alumina
Fe_2O_3	F	Ferric Oxide
MgO	M	Magnesia
K_2O	K	Alkalis
Na_2O	Na	Alkalis
SO_3	<u>S</u>	Sulfate
CO_2	<u>C</u>	Carbonate
H_2O	<u>H</u>	Water

Compound Composition

- Bogue Composition/Calculations

Alite (Tricalcium Silicate)

$C_3S = 4.07C – 7.60S – 6.72A – 1.43F – 2.85\underline{S}$

Belite (Dicalcium Silicate)

$C_2S = 2.87S – 0.75C_3S$

Aluminate Phase (Tricalcium Aluminate)

$C_3A = 2.65A – 1.69F$

Ferrite Compounds(Tetracalcium Aluminoferrite)

$C_4AF = 3.04F$

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Compound Composition: Example Bogue

Oxide	%
SiO_2	20.6
Al_2O_3	5.07
Fe_2O_3	2.90
CaO	63.9
MgO	1.53
SO_3	2.53
Na_2O	0.15
K_2O	0.73
LOI	1.58

Calculated Phase Composition

C_3S = 4.07(63.9) – 7.60(20.6) – 6.72(5.07) – 1.43(2.90) – 2.85(2.53) = **58.1**

C_2S = 2.87(20.6) – 0.75(58.1) = **15.6**

C_3A = 2.65(5.07) – 1.69(2.90) = **8.5**

C_4AF = 3.04(8.8) = **8.8**

Phase	%
C_3S	58
C_2S	16
C_3A	9
C_4AF	9

Bogue Composition Assumptions

- All 4 phases are pure
- All the F present occurs as C_4AF, and the quantities of A = 0.64(% F) and C = 1.40 (% F) are subtracted from the appropriate totals.
- The remaining Al_2O_3 is combined as C_3A and a further quantity of C = 1.65 (% Al_2O_3) is subtracted from the total remaining CaO.
- The SiO_2 combines initially with CaO to form C_2S giving a provisional C_2S figure. The CaO combining with SiO_2 = 2.87%(SiO_2) is subtracted from the total CaO figure, and the remaining CaO is then combined with a part of the C_2S = 4.07(%CaO) to form C_3S.

As a result, Bogue compositions may be "off" by as much as 10% compared to XRD-determined compositions.

Compound Composition: Example Equivalent Alkalis

Oxide	%
SiO_2	20.6
Al_2O_3	5.07
Fe_2O_3	2.90
CaO	63.9
MgO	1.53
SO_3	2.53
Na_2O	0.15
K_2O	0.73
LOI	1.58

Sodium equivalent, Na_2O_e

$Na_2O_e = Na_2O + (0.658 \times K_2O)$

$Na_2O_e = 0.15 + (0.658 \times 0.73)$

$Na_2O_e = 0.63\%$

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Chemical Limits

Cement Type	Applicable Test Method	I	II	III	IV	V
Al_2O_3, max, %	C114	---	6.0	---	---	---
Fe_2O_3, max, %	C114	---	6.0	---	6.5	---
MgO, max, %	C114	6.0	6.0	6.0	6.0	6.0
SO_3, max, %						
When $C_3A \leq 8\%$	C114	3.0	3.0	3.5	2.3	2.3
When $C_3A \geq 8\%$		3.5		4.5		
Loss on ignition, max, %						
When limestone is not an ingredient	C114	3.0	3.0	3.0	2.5	3.0
When limestone in an ingredient		3.5	3.5	3.5	3.5	3.5
Insoluble residue, max, %	C114	1.5	1.5	1.5	1.5	1.5
C_3S, max, %	Annex A1	---	---	---	35	---
C_2S, max, %	Annex A1	---	---	---	40	---
C_3A, max, %	Annex A1	---	8	15	7	5
$C_3S + 4.75C_3A$, max, %	Annex A1	---	---	---	---	---
C_3S, max, %	Annex A1	---	---	---		
$C_4AF + 2(C_3A)$, max, %	Annex A1	---	---	---	---	25

Presentation Outline

ASTM C150 – Portland Cement

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- Fineness
  - ASTM C204 – Air Permeability Test
- Consistency
  - ASTM C1437 – Flow
  - ASTM C187 – Normal Consistency

Chemical Requirements

- Chemical analysis
- Compound composition

ASTM C989 – Slag Cement

- ***Chemical composition***
- ***Physical requirements***

Nomenclature: Slag Cement vs. GGBFS

ASTM C989 – Slag Cement

- Glassy granular material formed when molten blast-furnace slag is rapidly chilled, as by immersion in water
- Non-metallic product, consisting of silicates and aluminosilicates of calcium and other bases

Component	Mass (%)
CaO	30-50
SiO_2	28-38
Al_2O_3	8-24
MgO	1-18

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Classification

- Slag is Classified as Grade 80, 100, and 120
- Determined from compressive strength
- ASTM C109 and ASTM C1437
- (2 Mixes)
  - Slag mixture 50/50 slag and reference cement
  - Reference mixture – only reference cement

Total Alkalies (Na_2O + 0.658 K_2O)	min %	0.60
	max %	0.90
Compressive Strength, MPa, min, 28 days[A]		35 [5000 psi]

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Classification

Slag Activity Index, % = (SP / P) X 100

SP = Average compressive strength of slag−cement mortar cubes, MPa
P = Average compressive strength of cement mortar cubes, MPa

	Average of Last Five Consecutive Samples	Any Individual Sample
Slag Activity Index		
28-Day Index, min.%		
Grade 80	75	70
Grade 100	95	90
Grade 120	115	120

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Physical Requirements

	Item
Fineness:	
Amount retained when wet screened on a 45-µm Sieve, max. %	20
Specific surface by air permeability, Test Methods C204 shall be determined and reported although no limits are requried.	…
Air Content of Slag Mortar, max. %	12

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Chemical Requirements

- Composition Depends mainly on the composition blast furnace oxides
- Variability between sources exist, but relatively low within the same plant
- ASTM C989 Limits
  - Sulfide sulfur content (S), to 2.55
  - Determined per ASTM C114

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Portland Cement and Slag Cement Standards

- Portland Cement Standards
  - Require more testing than slag cement
  - Variability within and between sources
  - Make up large percentage of concrete mixtures
- Slag Cement
  - Used increasing quantities throughout the world
  - Concern on titanium concentrations

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Questions & Answers