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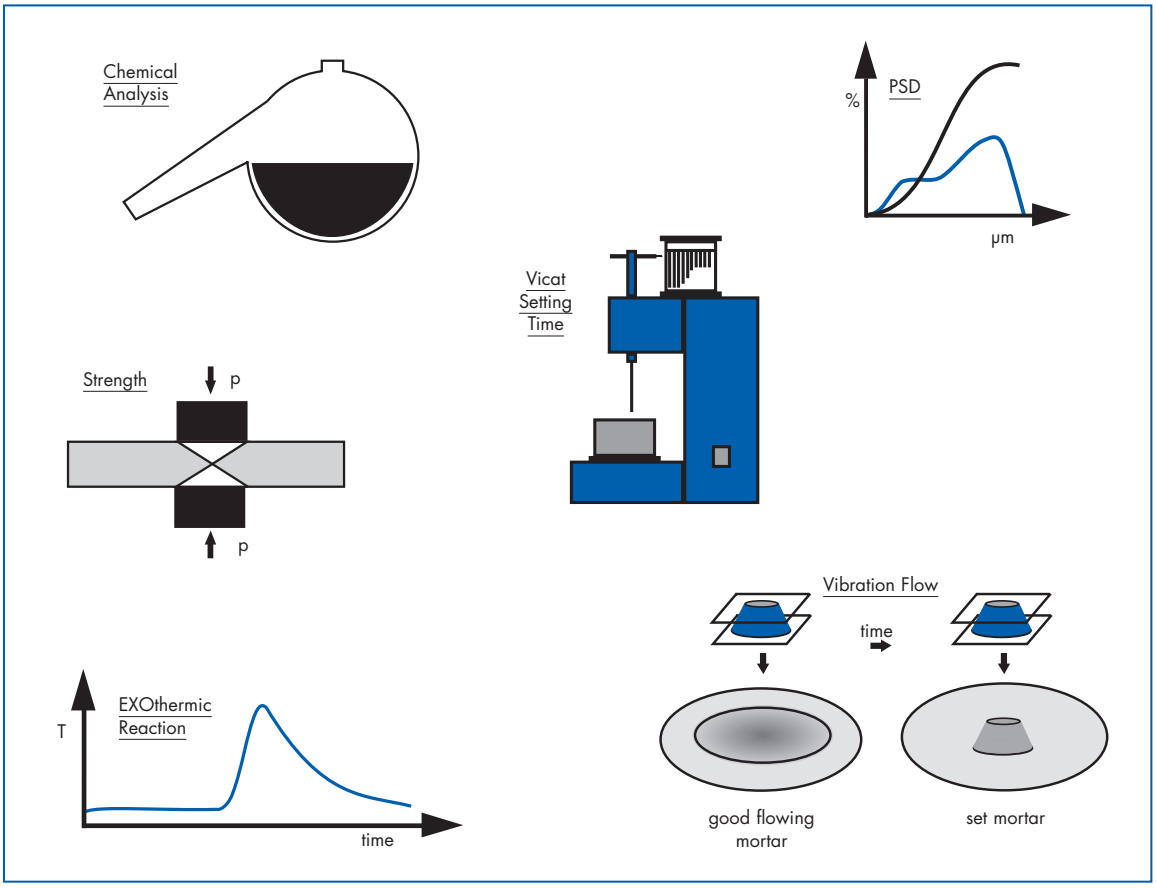
PREMIUM ALUMINA

Global Product Data

Calcium Aluminate Cements

Cement Test Methods



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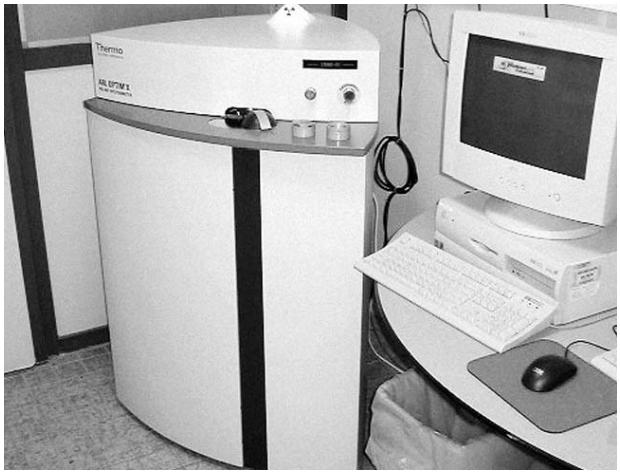
Calcium Aluminate Cements

Cement Test Methods



Chemical Analysis by X-Ray Fluorescence (XRF)

Vicamat – Measurement for Setting Time

Cilas – Measurement for Particle Size Distribution

Calcium Aluminate Cements

Cement Test Methods

Introduction

This brochure explains the Almatis cement test methods for Calcium Aluminate Cements which are used to provide the data on the product data sheet. Test descriptions for analysing chemical composition and fineness of pure cement as well as determining setting, exothermic reaction, flow and strength properties in a cement test mortar are also given.

The principle for our testing is the European Norm EN-196 "Methods of Testing Cement", designed for testing Portland cements. The EN-196 Normsand test grog was replaced by a Tabular Alumina grog (NORTAB).
This was done because Silica, the raw material for Normsand, is not a major component in refractory concretes. Also cement setting in sand-based concretes is generally faster than with Tabular, thus covering up differences in cement hydration behavior.

NORTAB test grog has a similar Particle Size Distribution (PSD) as Normsand. Using NORTAB brings our Calcium Aluminate Cement testing, based on EN-196, closer to the field requirements. NORTAB mortar is composed of 80 % test grog and 20 % cement plus individual water additions for each cement type as required for good working consistency: 10 % water for CA-14 cements, CA-25 R and CA-25 M and 9 % water for CA-270 and CA-25 C.

This latest revision of the CAC test methods brochure is reflecting the recent change in overall amount of mortar prepared for the testing and the mixing procedure. The NORTAB grog and the cement are dry mixed before the water is added.

Contents List

Calcium Aluminate Cements

Cement Test Methods

Chemical Analysis

1. Objective

This procedure describes the Almatis method for the chemical analysis of Calcium Aluminate Cement.
Conditions are based on the European Standard EN-ISO 12677:2003

2. Principal features of the method

This method determines the chemical analysis of Calcium Aluminate Cement for CaO, Al_2O_3, Fe_2O_3, Na_2O, SiO_2 and MgO. The chemical composition is measured with Wavelength Dispersive X-Ray Fluorescence spectrometry (WD-XRF). This method utilizes the element specific X-Ray wavelength, which is created by an electron beam directed on the sample. The intensity of the X-Ray radiation of the specific element provides the concentration of that element in the sample.

3. Laboratory and equipment

Laboratory conditions are according to the EN-ISO 12677:2003.
A Wavelength Dispersive XRF Spectrometer, Sequential-Simultaneous configuration is used.
The instrument is fitted with a SmartGonio™ for the elements Al (Z=13) to U (Z=92) and fixed channels for the elements Na (Z=11) and Mg (Z=12).

4. Procedure

10.000 g Calcium Aluminate Cement and 0.6 gram wax binder is ground in a laboratory disk mill to less than 50 microns to reduce particle size effects. The obtained homogeneous mixture is pressed into a 30 mm steel ring with a hydraulic press at approx. 15 MPa. The binder is used to obtain better pressed pellets.
This pressed pellet is placed into the Wavelength Dispersive - XRF for the determination of the concentration of Ca, Al, Fe, Na, Si, and Mg. The results are provided as CaO, Al_2O_3, Fe_2O_3, Na_2O, SiO_2 and MgO.

Reference curves are made from guaranteed reference sample concentrations, which are obtained from an external certified laboratory and cover the full reference concentration range shown in the table. A reference curve is made for each element oxide using Multi-Variable-Regression.

All samples and reference curves are measured with X-Ray Fluorescence spectrometry (WD-XRF). The concentration in the cement sample is determined with the reference curves.

Oxide	Crystal/ Detector	Line	Reference curve concentration [wt%]
CaO	Lif200 / FPC	CaKa1,2	0 – 33.0
Al_2O_3	PET / FPC	AlKa1,2	0 – 99.0
Fe_2O_3	Lif200 / FPC	FeKa1,2	0 – 7.70
Na_2O	Fixed	NaKa_m	0 – 0.90
SiO_2	PET / FPC	SiKa1,2	0 – 1.30
MgO	Fixed	MgKa_M	0 – 0.65

5. Calculation

The sum of the oxides CaO, Al_2O_3, Fe_2O_3, Na_2O, SiO_2 and MgO will be reported as a fixed total of 100% based on moisture and loss on ignition free material.

Calcium Aluminate Cements

Cement Test Methods

Fineness by Laserdiffractometer

1. Objective

This procedure describes the Almatis method of determining the Particle Size Distribution of Calcium Aluminate Cement. Conditions are as nearly as possible based on the European Standard EN-196 part 1, May 2005.

2. Principal features of the method

This method involves the determination of the Particle Size Distribution (PSD) of Calcium Aluminate Cement, measured by the laserdiffractometer Cilas 1064. The PSD is analysed by ultrasonic dispersion of the Calcium Aluminate Cement in Isopropyl alcohol. The liquid is passed through the measuring cell of the laserdiffractometer. Two laser beams are sent through the measuring cell to a detector. The dispersed powder causes scattering of the laser beams. The scattering pattern is a function of the size of the particles as well as the quantity of a given particle size. The scattering pattern is measured by the detector and translated into a PSD reported as a sum curve and as a cumulative curve.

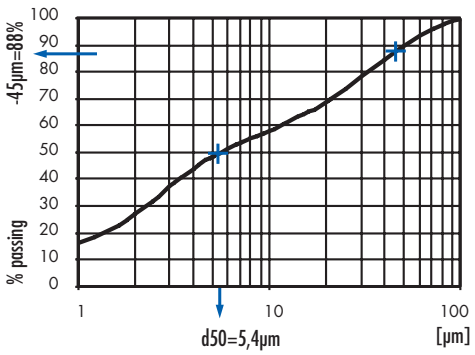
3. Laboratory and equipment

Laboratory conditions complying with 4.1 of EN-196 part 1, May 2005.
Laserdiffractometer (Quantachrome, Cilas Laserdiffractometer 1064).
Isopropyl alcohol.

4. Procedure

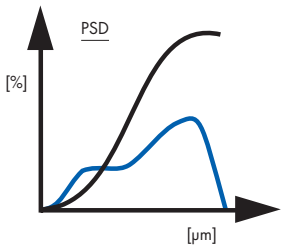
The laserdiffractometer is calibrated by using a standardised sample with a defined PSD. As all cement grades have their typical PSD characteristics, standard samples with a defined PSD are used for calibration before each testing.

Cilas PSD sum curve with d50 and -45µm points (Example CA-270)



The sample of 0.3 – 0.5 g Calcium Aluminate Cement is filled. The material is dispersed for 180 sec in an ultrasonic bath of Isopropyl alcohol, followed by the actual PSD measurement.

The PSD is expressed in d90, d50, d10, reflecting the particle diameter of the material where 90 %, 50 %, or 10 % of the powder is smaller than the listed diameter in µm. Recorded is the d50.



The weight percentage of material being smaller -45 µm (-325 mesh) is also calculated by the device and recorded.

Calcium Aluminate Cements

Cement Test Methods

Vicat Setting Time

1. Objective

This procedure describes the Almatis method of determining the setting properties of Calcium Aluminate Cement mortar. Conditions are as nearly as possible based on the European Standard EN-196 part 3, May 2005.

2. Principal features of the method

This method involves the determination of the setting properties of cement mortar filled in Vicat moulds. The Vicat moulds are of truncated conical form, 40 mm high, internal diameter at top of 80 mm and bottom of 90 mm.

The mortar contains 20 wt % of Calcium Aluminate Cement and 80 wt % of a standard Tabular Alumina T60/T64 grog (NORTAB). It has a water/cement ratio of 0.5 for CA-14 cements, for CA-25 R and CA-25M, 0.45 for CA-270 and CA-25 C (see table below).

The mortar is prepared by mechanical mixing and cast in the Vicat moulds without compacting or vibration. The filled moulds are kept in a moist atmosphere. The setting behaviour is determined by observing the decrease of penetration depth of a Vicat needle into the mortar. Initial and final setting times are taken when the Vicat needle is supported 10 or 30 mm above the base plate.

3. Laboratory and equipment

Laboratory conditions complying with 4.1 of EN-196 part 1, May 2005.
Test sieves complying with 4.3 of EN-196 part 1, May 2005.
Hobart Mixer (~5 litre bowl volume) complying with 4.4 of EN-196 part 1, May 2005.
Vicat moulds complying with 5.1 of EN-196 part 3, May 2005.
Vicat apparatus complying with 5.1 of EN-196 part 3, May 2005.
Demineralized water.

4. Mortar

NORTAB grog (made of pure Tabular Alumina T60/T64) is used to determine Vicat setting time of Calcium Aluminate Cement according to this procedure. For NORTAB grog sieve analysis and mortar composition see table opposite. NORTAB grog is applied as a premix of 2800 ± 2 g.

Particle Size Distribution (PSD) of NORTAB grog and Composition of mortar

NORTAB PSD	
Square mesh size [mm]	Sieve Residue [%]
+ 2.0	3 ± 2
+ 1.4	13 ± 3
+ 1.0	14 ± 5
+ 0.5	35 ± 5
+ 0.125	29 ± 5
+ 0.063	4 ± 3
– 0.063	2 ± 2
Raw Material: Tabular Alumina T60/T64	

NORTAB Mortar Composition:
80 % NORTAB and 20 % cement plus

- 10 % H_2O for CA-14 cements
- 10 % H_2O for CA-25 R and CA-25M
- 9 % H_2O for CA-270
- 9 % H_2O for CA-25 C

Calcium Aluminate Cements

Cement Test Methods

5. Procedure

NOTE Set up of Vicat apparatus: Use of an automatic Vicat tester device with
- cylindrical Vicat needle, effective length = 50 ± 1 mm, diameter = 1.13 ± 0.05 mm;
- total mass of the moving parts is 1000 ± 1 g.

Composition of the mortar	for CA-14 cements, CA-25 R and CA-25 M	for CA-270, CA-25 C
	2800 ± 2 g NORTAB	2800 ± 2 g NORTAB
	700 ± 1 g cement	700 ± 1 g cement
	350 ± 1 g water	315 ± 1 g water

Fill the NORTAB grog in the mixing bowl and add the cement. Start the mixer at low speed, note the starting timer as zero. After 1 min. add the demineralized water. Stop mixer after total mixing time of 5 min.

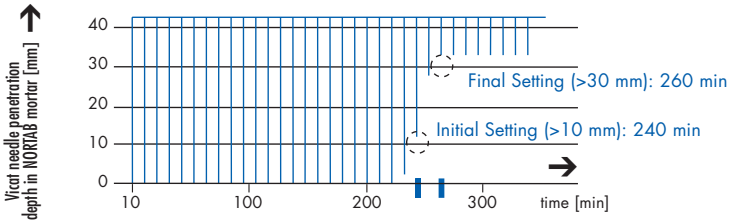
Fill the mortar immediately into a Vicat mould without compacting or vibration, large end facing up, cover and invert. Place the mould in upright position in a holder (pan). Cover the mould in the holder with a thin layer of demineralized water (~1-2 mm) to prevent drying out of mortar surface during testing. Transfer the holder to the calibrated automatic Vicat apparatus and position under the Vicat needle. All needle penetrations should be at 10 mm from the rim of the mould and 10 mm from each other.

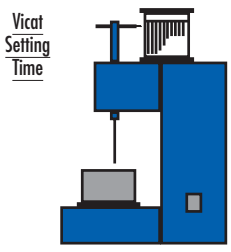
Start the automatic Vicat apparatus 10 min after start of mixing. Record the depths of penetration against time. Time intervals are every 10 min for CA-14 and CA-270 cements, every 2 min for CA-25 R and every 5 min for CA-25 C and CA-25 M.

The time measured from the start of mixing to the time at which the distance between the needle and the base plate is 10 ± 1 mm, followed by successive measurements at which this distance is equal or exceeded, is recorded as the **Initial Setting time (IS)**.

The time measured from the start of mixing to the time at which the distance between the needle and the base plate is 30 ± 1 mm, followed by successive measurements at which this distance is equal or exceeded, is recorded as the **Final Setting time (FS)**.

Vicat Setting Time: Measuring curve with Initial and Final Setting points (Example CA-14 M)





Calcium Aluminate Cements

Cement Test Methods

Exothermic Reaction (EXO)

1. Objective

This procedure describes the Almatis method of determining the exothermic properties of Calcium Aluminate Cement mortar. Conditions are as nearly as possible based on the European Standard EN-196 part 3, May 2005.

2. Principal features of the method

This method describes the determination of the exothermic heat development during cement hydration of a cast mortar test specimen. The cast sample has a weight of 1.5 kg.
The mortar contains 20 wt % of Calcium Aluminate Cement and 80 wt % of a standard Tabular Alumina T60/T64 grog (NORTAB). It has a water/cement ratio of 0.5 for CA-14 cements, for CA-25 R and CA-25 M, 0.45 for CA-270 and CA-25 C (see table below).
The mortar is prepared by mechanical mixing and cast in the moulds under light vibration. A thermocouple (type J) is put into the mortar and connected to a measurement device. The mortar in the moulds is covered. The temperature development of the mortar until complete hydration is measured as function of the time after mixing.

3. Laboratory and equipment

Laboratory conditions complying with 4.1 of EN-196 part 1, May 1987.
Test sieves complying with 4.3 of EN-196 part 1, May 2005.
Hobart-Mixer (~5 litre bowl volume) complying with 4.4 of EN-196 part 1, May 2005.
Vibration table complying with DIN EN-196 part 1, March 1990.
Test apparatus for recording exothermic temperature increase.
Demineralized Water.

4. Mortar

NORTAB grog (made of pure Tabular Alumina T60/T64) is used to determine Vicat setting time of Calcium Aluminate Cement according to this procedure. For NORTAB grog sieve analysis and mortar composition see table opposite. NORTAB grog is applied as a premix of 2800 ± 2 g.

Particle Size Distribution (PSD) of NORTAB grog and Composition of mortar

NORTAB PSD	
Square mesh size [mm]	Sieve Residue [%]
+ 2.0	3 ± 2
+ 1.4	13 ± 3
+ 1.0	14 ± 5
+ 0.5	35 ± 5
+ 0.125	29 ± 5
+ 0.063	4 ± 3
– 0.063	2 ± 2
Raw Material: Tabular Alumina T60/T64	

NORTAB Mortar Composition:
80 % NORTAB and 20 % cement plus

- 10 % H_2O for CA-14 cements
- 10 % H_2O for CA-25 R and CA-25M
- 9 % H_2O for CA-270
- 9 % H_2O for CA-25 C

Calcium Aluminate Cements

Cement Test Methods

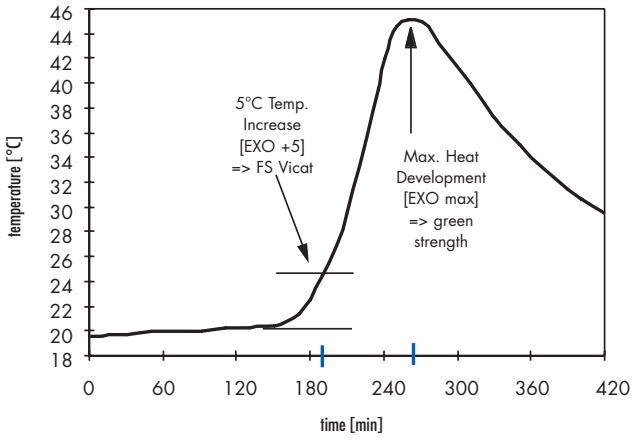
5. Procedure

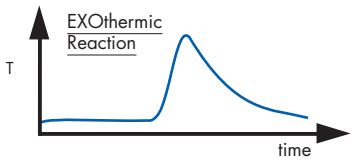
Composition of the mortar	for CA-14 cements, CA-25 R and CA-25 M	for CA-270, CA-25 C
	2800 ± 2 g NORTAB	2800 ± 2 g NORTAB
	700 ± 1 g cement	700 ± 1 g cement
	350 ± 1 g water	315 ± 1 g water

Fill the NORTAB grog in the mixing bowl and add the cement. Start the mixer at low speed, note the starting timer as zero. After 1 min. add the demineralized water. Stop mixer after total mixing time of 5 min.

Transfer the mortar immediately to a box, using 1500 g mortar. Compact the mortar by vibrating for 10 sec. Put a thermocouple (type J) into the mortar and connect to a data recorder (PC logger). Cover the mortar in the mould. Measure the temperature of the mortar as function of time after start of mixing (zero) until complete hydration.

- The time measured from the start of mixing until the exothermic reaction shows a temperature increase of +5 °C is recorded as **EXO+5**.
- The time measured from the start of mixing until maximum temperature of the exothermic reaction is reached is recorded as **EXO max**. It corresponds to the time when there is sufficient green strength for demoulding.





Calcium Aluminate Cements

Cement Test Methods

Vibration Flow

1. Objective

This procedure describes the Almatis method of determining the flow properties of Calcium Aluminate Cement mortar. Conditions are as nearly as possible based on the European Standard EN-196 part 1, May 2005.

2. Principal features of the method

This method involves the determination of the flow properties of cement mortar filled in Vicat moulds. The Vicat moulds are of truncated conical form, 40 mm high, internal diameter at top of 70 mm and bottom of 80 mm.

The mortar contains 20 wt % of Calcium Aluminate Cement and 80 wt % of a standard Tabular Alumina T60/T64 grog (NORTAB). It has a water/cement ratio of 0.5 for CA-14 cements, for CA-25 R and CA-25 M, 0.45 for CA-270 and CA-25 C (see table below).

Sufficient mortar to fill three Vicat moulds is prepared by mechanical mixing. The three Vicat moulds are casted without vibration. The filled moulds are covered. After defined time intervals, a filled Vicat mould is placed on a vibration table, the mould is lifted off, and the mortar sample is vibrated. The diameter after vibration indicates the flow property as a function of time.

3. Laboratory and equipment

Laboratory conditions complying with 4.1 of EN-196 part 1, May 2005.
Test sieves complying with 4.3 of EN-196 part 1, May 2005.
Hobart Mixer (~ 5 litre bowl volume) complying with 4.4 of EN-196 part 1, May 2005. Vibration table complying with DIN EN 196 part 1, March 1990. Vicat moulds complying with 5.1 of EN-196 part 3, May 2005*.
Demineralized water.
Plexi-glass plates.

**Dimensions of Vicat mould for determination of flow properties in use is: 40 mm hight, 70 mm upper diameter, 80 mm lower diameter.*

Particle Size Distribution (PSD) of NORTAB grog and Composition of mortar

NORTAB PSD	
Square mesh size [mm]	Sieve Residue [%]
+ 2.0	3 ± 2
+ 1.4	13 ± 3
+ 1.0	14 ± 5
+ 0.5	35 ± 5
+ 0.125	29 ± 5
+ 0.063	4 ± 3
– 0.063	2 ± 2
Raw Material: Tabular Alumina T60/T64	
NORTAB Mortar Composition: 80 % NORTAB and 20 % cement plus – 10 % H_2O for CA-14 cements – 10 % H_2O for CA-25 R and CA-25M – 9 % H_2O for CA-270 – 9 % H_2O for CA-25 C	

4. Mortar

NORTAB grog (made of pure Tabular Alumina T60/T64) is used to determine Vicat setting time of Calcium Aluminate Cement according to this procedure. For NORTAB grog sieve analysis and mortar composition see table opposite. NORTAB grog is applied as a premix of 2800 ± 2 g.

Calcium Aluminate Cements

Cement Test Methods

5. Procedure

Composition of the mortar	for CA-14 cements, CA-25 R and CA-25 M	for CA-270, CA-25 C
	2800 ± 2 g NORTAB 700 ± 1 g cement 350 ± 1 g water	2800 ± 2 g NORTAB 700 ± 1 g cement 315 ± 1 g water

Fill the NORTAB grog in the mixing bowl and add the cement. Start the mixer at low speed, note the starting timer as zero. After 1 min. add the demineralized water. Stop mixer after total mixing time of 5 min.

After mixing, immediately fill the Vicat moulds with mortar. Use 450 g castable per mould to fill them.
Cover the moulds with a plexi glass plate.
9 min after the mixing started, place the first mould on the vibration table with the large end facing down. Remove the plates and the mould.

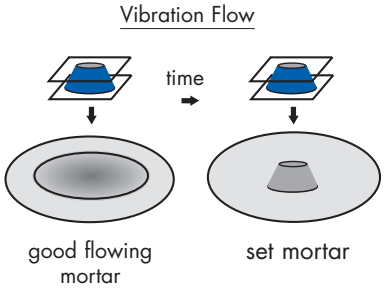
10 min after the mixing started, vibrate the mortar sample for 30 sec at amplitude 0.5 mm, frequency 50 Hz, measure the diameter in four directions, average the value and record it as **Vibration Flow F10** (i.e.: F10=16 cm stands for a vibration diameter of 16 cm tested 10 min after begin of mixing).

Repeat the above procedure for the other two mortar samples at defined intervals, see table below.

The sample is set when the flow diameter equals the large inner diameter of the mould.

Flow measurement intervals

Test Interval	Recorded Value
10 min	F10
30 min	F30
60 min	F60



Calcium Aluminate Cements

Cement Test Methods

Strength

1. Objective

This procedure describes the Almatis method of determining the cold modulus of rupture (CMOR) and cold crushing strength (CCS) of Calcium Aluminate Cement mortar. Conditions are as nearly as possible based on the European Standard EN-196 part 1, May 2005.

2. Principal features of the method

This method involves the determination of the cold crushing strength, and optional the flexural strength of test bars of 40 x 40 x 160 mm size.

The mortar contains 20 wt % of Calcium Aluminate Cement and 80 wt % of a standard Tabular Alumina T60 grog (NORTAB). It has a water/cement ratio of 0.5 for CA-14 cements, for CA-25 R and CA-25M, 0.45 for CA-270 and CA-25 C (see table below).

The mortar is prepared by mechanical mixing and is compacted in a mould using a vibration table. The bars are cured for 24 hours in a moist atmosphere and then demoulded. The bars will either (1) be directly used for strength testing, (2) be dried for 24 hours at 105°C, or will (3) be fired for 5 hours at 1000°C before strength testing. The bars will be broken under bending conditions into 2 halves, indicating the cold modulus of rupture. Each half will be tested for cold crushing strength.

3. Laboratory and equipment

Laboratory, test sieves, mixer, moulds for three bars, flexural and cold crushing strength testing machines are according to the EN-196 part 1, May 2005.
Vibration table according the DIN-EN-196 part 1, March 1990.
Metal spatula (strong material).

4. Mortar

NORTAB grog (made of pure Tabular Alumina T60/T64) is used to determine Vicat setting time of Calcium Aluminate Cement according to this procedure. For NORTAB grog sieve analysis and mortar composition see table opposite. NORTAB grog is applied as a premix of 2800 ± 2 g.

Particle Size Distribution (PSD) of NORTAB grog and Composition of mortar

NORTAB PSD	
Square mesh size [mm]	Sieve Residue [%]
+ 2.0	3 ± 2
+ 1.4	13 ± 3
+ 1.0	14 ± 5
+ 0.5	35 ± 5
+ 0.125	29 ± 5
+ 0.063	4 ± 3
– 0.063	2 ± 2
Raw Material: Tabular Alumina T60/T64	

NORTAB Mortar Composition:
80 % NORTAB and 20 % cement plus

- 10 % H_2O for CA-14 cements
- 10 % H_2O for CA-25 R and CA-25M
- 9 % H_2O for CA-270
- 9 % H_2O for CA-25 C

Calcium Aluminate Cements

Cement Test Methods

5. Procedure

Composition of the mortar	for CA-14 cements, CA-25 R and CA-25 M	for CA-270, CA-25 C
	2800 ± 2 g NORTAB	2800 ± 2 g NORTAB
	700 ± 1 g cement	700 ± 1 g cement
	350 ± 1 g water	315 ± 1 g water

Fill the NORTAB grog in the mixing bowl and add the cement. Start the mixer at low speed, note the starting timer as zero. After 1 min. add the demineralized water. Stop mixer after total mixing time of 5 min.

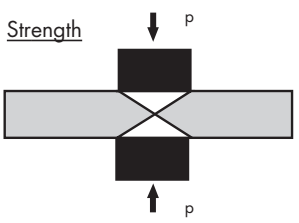
Run the vibration table at an amplitude of 0.50 mm. Cast the bars immediately after the preparation of the mortar by filling in two layers:
Introduce the first layer into each mould with the help of a spreader into the three compartments within 20 sec, vibrate the first layer for the next 20 sec. Then introduce the second layer of mortar within 20 sec and vibrate the layer for 60 sec.

Lift the mould from the vibration table and remove any excess with a strong spatula. Do this in each direction. Then level the surface with the spatula held almost flat.

Cure the bars in the mould covered with a plexi-glass plate for 24 hours at 20 ± 1°C. Remove the mould (if needed a rubber hammer can be used).

- Bar 1: Determine within 20 minutes after demoulding the **cured** strength according to EN-196 part 1, May 2005.
- Bar 2: Place cured bars directly after demoulding for 24 hours in a preheated drying chamber at 105 °C. Take the dried bar out and let cool to 20 °C. - Determine the **dried** strength according to EN-196 part 1, May 2005.
- Bar 3: Place cured bars directly after demoulding in a cold firing furnace. Heat the furnace during 5 hours up to 1000 °C and hold for 5 hours, then shut down. Take the fired bars out and let cool to 20 °C. - Determine the **fired** strength according to EN-196 part 1, May 2005.

Remark: Strengths are recorded in MPa. 1 MPa = 145 psi



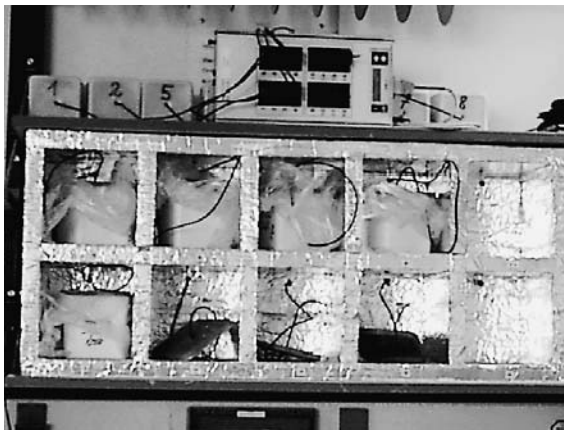
Calcium Aluminate Cements

Cement Test Methods

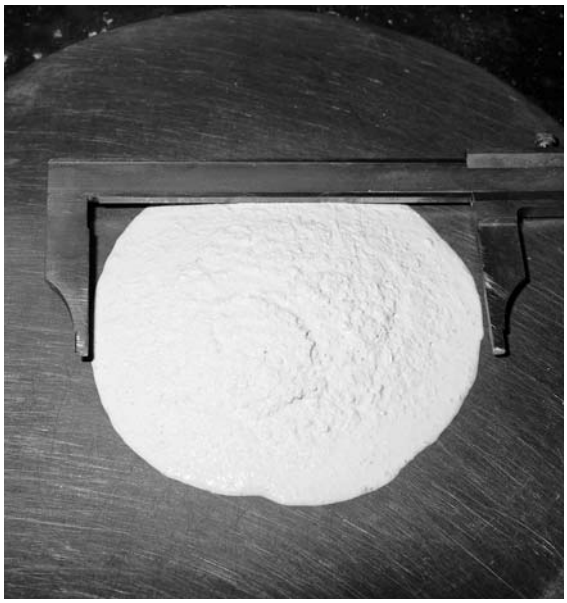
Page for personal notes

Calcium Aluminate Cements

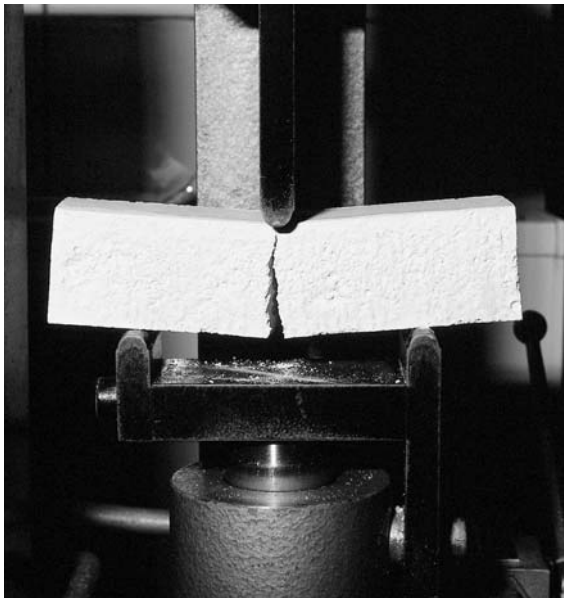
Cement Test Methods



Exothermic Reaction set-up



NORTAB mortar after vibration table test



Strength Test

Calcium Aluminate Cements

Cement Test Methods

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