ATILH
ASSOCIATION TECHNIQUE DE L'INDUSTRIE DES LIANTS HYDRAULIQUES

Bureau de Normalisation des Liants Hydrauliques

7, place de la Défense - 92974 Paris-la-Défense Cedex - Tél.: +33 (0)1 55 23 01 42 - Fax: +33 (0)1 49 67 10 46
E-mail : a.bonnet@atilh.fr

Certificate of Analysis

Reference Material TL-2BGa
Portland Cement
(CEM I 52,5 N)

Distributed by :

TECHLAB

I – Participation and execution of tests

Each year the "*Association Technique de l'Industrie des Liants Hydrauliques*" (ATILH) organises an interlaboratory test campaign involving in particular the participation of the cement production industry laboratories, the cement end-user laboratories and Research and Inspection Centers within the construction materials sector. This participation is compulsory for laboratories accredited by COFRAC for cement testing. The tests are carried out in accordance with standardised methods where latter exist, otherwise according to everyday traditional methods.

II – Statistical analysis of the results

Outliers are eliminated via the STUDENT's test with a confidence level of 98 %. A reiteration is set at this threshold in order to keep only those values which are related to the "Normal or Gaussian" distribution, the latter being entirely defined by 2 parameters: mean and standard deviation. The coefficient of variation symbolised by "V" is the ratio between the standard deviation "σ" and the mean value $\overline{X}$.

III – Specific surface and particle size analysis

For the calibration of the Blaine permeability apparatus, follow the requirements of the EN 196-6 standard, paying particular attention to the temperature corrections, if any. To determine the volume of the compacted layer, it is not essential to use the Reference Material (but ensure that a sufficient quantity is taken so that the mass of the mercury does not modify the compaction of the powder layer). Reference Material should be used systematically:

a) after 1000 tests ;
b) when using another type of manometric liquid, another type of filter paper, a new manometer tube or a new perforated disc;
c) If discrepancies are systematic with the secondary reference cement.

Table 1

	Mean value	Dispersion characteristics Reproducibility	
	$\overline{X}$	σ	V (%)
Particle density (g/cm^3) with picnometer method	3,15	0,02	0,66
Blaine Specific Area (cm^2/g) with EN 196-6	4206	74	1,75

Table 2

Particle size analysis by laser diffraction (ISO 13320-1)			Air-jet sieving - Alpine test (NF X11-640)	
Equivalent size aperture a (µm)	Mean (% of < a)*	σ (%)* (reproducibility)	Mean (% of < a)	σ (%) (reproducibility)
2,0	**13,4**	**3.7**		
3,15	**20,2**	**4.1**		
4	**24,1**	**4,1**		
5	**28,0**	**3,9**		
6,3	**32.7**	**4.0**		
8	**38,1**	**4,2**		
12,5	**51,2**	**4,8**		
16	**58,9**	**5,3**		
25	**75,0**	**4,9**	**86,0**	**5,2**
31,5	**83,0**	**3,8**	**89,1**	**3,4**
40	**90,1**	**3,3**	**93,5**	**1,4**
50	**94,7**	**2,7**		
63	**97,9**	**1,4**		
80	**99,1**	**1,0**	**99,5**	**0,2**
100	**99,6**	**1,0**		
125	**99,8**	**0,7**		
160	**100**	**0,1**		

**Number of values from 51 to 58*

III – Sample conditioning

The sample of this reference material is packaged in sold in batch of 2 phials of 20 g. Physico-chemical properties of the sample are stable until the phials is open. After opening the phials the local conditions of storage of the sample (courtroom with low humidity, maintaining in a drier, close the bottle immediately after use) will allow its potential reuse.

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Certificate of Analysis

Reference Material TL-2Ca
Portland Cement
(CEM I 52,5 N)

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I – Participation and execution of tests

Each year the "*Association Technique de l'Industrie des Liants Hydrauliques*" (ATILH) organises an interlaboratory test campaign involving in particular the participation of the cement production industry laboratories, the cement end-user laboratories and Research and Inspection Centers within the construction materials sector. This participation is compulsory for laboratories accredited by COFRAC for cement testing. The tests are carried out in accordance with standardised methods where latter exist, otherwise according to everyday traditional methods.

II – Statistical analysis of the results

Outliers are eliminated via the STUDENT's test with a confidence level of 98 %. A reiteration is set at this threshold in order to keep only those values which are related to the "Normal or Gaussian" distribution, the latter being entirely defined by 2 parameters: mean and standard deviation. The coefficient of variation symbolised by "V" is the ratio between the standard deviation "σ" and the mean value $\overline{X}$.

III – Chemical composition

X-ray fluorescence spectrometry, fused bead (ISO 29581-2)				Chemical Analysis (EN 196-2)		
Elements	Mean $\overline{X}$ (%)	Standard deviation σ (%) reproducibility	Coefficient of variation V (%)	Mean $\overline{X}$ (%)	Standard deviation σ (%) reproducibility	Coefficient of variation V (%)
Loss on ignition	-	-	-	1,31	0,06	4,51
SiO_2	20,01	0,21	1,04			
Al_2O_3	4,89	0,06	1,26			
Fe_2O_3	3,42	0,04	1,15			
CaO	64,51	0,31	0,48			
MgO	1,34	0,06	4,19			
$SO3$[3]	3,00	0,04	1,50	2,90	0,10	3,38
Free CaO[2]	1,06	0,17	15,85			
Insoluble[3]				0,49	0,08	16,79
Na_2O[1]	0,10	0,03	29,36	0,11	0,04	32,35
K_2O[1]	0,77	0,03	3,34	0,74	nc	nc
TiO_2	0,20	0,01	3,56			
P_2O_5	0,07*					
SrO	0,20*					

[1] photometric method [2] all methods combined [3] gravimetric method * P_2O_5 ±0,003 % - SrO ±0,003 %

TECHLAB Matériaux de Référence Certifiés & Equipements de Laboratoire
4C la Tannerie – BP 30 055 – 57072 METZ CEDEX 3 (France)
Tél : +33 (0)3 87 75 54 29 - Fax : +33 (0)3 87 36 23 90 - Email : techlab@techlab.fr

III – Sample conditioning

The sample of this reference material is packaged in sold in batch of 2 phials of 20 g. Physico-chemical properties of the sample are stable until the phials is open. After opening the phials the local conditions of storage of the sample (courtroom with low humidity, maintaining in a drier, close the bottle immediately after use) will allow its potential reuse.

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Reference Material TL-203BGa

Portland Composite Cement
(CEM II/B-M 32,5 R)

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I – Participation and execution of tests

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II – Statistical analysis of the results

Outliers are eliminated via the STUDENT's test with a confidence level of 98 %. A reiteration is set at this threshold in order to keep only those values which are related to the "Normal or Gaussian" distribution, the latter being entirely defined by 2 parameters: mean and standard deviation. The coefficient of variation symbolised by "V" is the ratio between the standard deviation "σ*"* and the mean value $\overline{X}$.

III – Specific surface and particle size analysis

For the calibration of the Blaine permeability apparatus, follow the requirements of the EN 196-6 standard, paying particular attention to the temperature corrections, if any. To determine the volume of the compacted layer, it is not essential to use the Reference Material (but ensure that a sufficient quantity is taken so that the mass of the mercury does not modify the compaction of the powder layer). Reference Material should be used systematically:

a) after 1000 tests ;
b) when using another type of manometric liquid, another type of filter paper, a new manometer tube or a new perforated disc;
c) If discrepancies are systematic with the secondary reference cement..

Table 1

	Mean value $\overline{X}$	Dispersion characteristics Reproducibility	
		σ	V (%)
Particle density (g/cm3) with picnometer method)	**3.05**	**0,02**	**0,68**
Blaine Specific Area (cm2/g) with EN 196-6	**4329**	**112**	**2.58**

Table 2

Particle size analysis by laser diffraction (ISO 13320-1)			Granulométrie Alpine (NF X11-640)	
Equivalent size aperture **a** (µm)	Mean (% of < **a***)	σ (%) (reproducibility)	Mean (% of < **a****)	σ (%) (reproducibility)
1,25	**9,0**	**2,7**		
1,6	**11,0**	**3,2**		
2,0	**13,5**	**3,1**		
2,5	**16,5**	**3,4**		
3,15	**19,8**	**4,0**		
4,0	**23,6**	**4,1**		
5,0	**27,7**	**4,2**		
6,3	**32,1**	**4,1**		
8,0	**37,2**	**4,1**		
10,0	**42,7**	**4,2**		
12,5	**48,7**	**4,5**		
16,0	**55,9**	**4,5**		
20,0	**63,3**	**4,4**		
25,0	**70,7**	**4,1**		
31,5	**78,6**	**3,8**	**83,2**	**3,0**
40,0	**85,9**	**3,7**	**89,9**	**1,2**
50,0	**91,8**	**3,5**	**93,4**	**1,2**
63,0	**96,2**	**2,2**	**96,7**	**1,2**
80,0	**98,4**	**1,2**	**98,9**	**0,6**
100,0	**99,4**	**0,7**	**99,3**	**0,7**
125,0	**99,8**	**0,3**	**99,7**	**0,4**
160,0	**100,0**	**0,2**	**100,0**	**0,04**

**number of values from 75 to 84*
*** number of values from 26 to 54*

III – Sample conditioning

The sample of this reference material is packaged in 40 g glass bottle, sealed with a secure screw cap. Physicochemical properties of the sample are stable until the bottle is closed and the cap untouched. After opening the bottle the local conditions of storage of the sample (courtroom with low humidity, maintaining in a drier, close the bottle immediately after use) will allow its potential reuse.

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III – Chemical composition

X-ray fluorescence spectrometry, fused bead (ISO 29581-2)				Chemical Analysis (EN 196-2)		
Elements	Mean *X* (%)	Standard deviation σ (%) reproducibility	Coefficient of variation V (%)	Mean *X* (%)	Standard deviation σ (%) reproducibility	Coefficient of variation V (%)
Loss on ignition	-	-	-	**7,24**	**0,10**	**1,35**
SiO_2	**18,75**	**0,31**	**1,65**	**18,85**	**0,21**	**1,10**
Al_2O_3	**4,78**	**0,07**	**1,48**	**4,93**	**0,18**	**3,63**
Fe_2O_3	**2,34**	**0,04**	**1,78**	**2,31**	**0,05**	**2,05**
CaO	**57,09**	**0,45**	**0,79**	**56,96**	**0,46**	**0,81**
MgO	**4,80**	**0,11**	**2,26**	**4,78**	**0,18**	**3,84**
SO3 (**3**)	**3,28**	**0,12**	**3,54**	**3,09**	**0,07**	**2,27**
Free CaO (2)	-	-	-	**1,23**	**0,19**	**15,67**
Insoluble (3)				**0,58**	**0,12**	**20,08**
Na_2O (1)	**0,21**	**0,03**	**15,85**	**0,26**	**0,03**	**9,59**
K_2O (1)	**0,93**	**0,03**	**3,44**	**0,92**	**0,08**	**8,80**
TiO_2	**0,24**	**0,01**	**3,74**			
P_2O_5	**0,13***					
SrO	**0,06***					

(1) photometric method (2) all methods combined (3) gravimetric method * P_2O_5 ±0,003 % - SrO ±0,003 %