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**Certificate of analysis**  
**Reference Material TL-202C**  
**Composite Cement**  
**(CEM V/A 32,5 N)**

**I – General data**

The table below shows the overall results obtained in the 11 laboratories which were involved in the inter-laboratory tests. Each value (mass content in %) is the average of 2 trials :

Laboratory	Loss of Ignition	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	Chloride	Sulphide	Na <sub>2</sub> O	K <sub>2</sub> O	TiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>
1	1,3	29,63	10,15	3,27	45,35	4,40	3,38	0,01	0,226	0,38	1,06	0,51	0,25
2		29,57	9,96	3,29	45,08	4,55	3,17	0,012	0,175	0,23	1,08	0,53	0,27
3	1,42	30,02	10,24	3,43	45,18	4,54	3,17	0,01		0,24	1,07	0,52	0,26
4	1,53	29,36	10,05	3,33	45,09	4,60	2,95	0,02	0,2	0,27	1,11		
5	1,39	29,27	9,96	3,29	45,25	4,48	3,26	0,01	0,2	0,22	1,04		
6	1,83	29,56	10,12	3,29	44,73	4,48	3,54	0,011	0,225	0,23	0,89		
7	1,36	29,70	10,05	3,21	45,13	4,45	3,01	0,01	0,19	0,30	1,10		
8	1,71	29,26	10,28	2,91	45,55	4,13	2,98	0,005	0,175	0,77	1,10		
9	1,40	30,53	10,65	3,31	44,50	4,60	3,52	0,03		0,27	1,03		
10	1,40	29,68*	10,09*	3,26*	45,11*	4,39*	2,97		0,225	0,35*	1,01*		
11	1,77*	29,21*	10,04*	3,36*	45,40*	4,46*	2,98*		0,2*	0,26*	1,04*	0,49*	0,27*
Mean M	<b>1,51</b>	<b>29,61</b>	<b>10,14</b>	<b>3,27</b>	<b>45,12</b>	<b>4,46</b>	<b>3,17</b>	<b>0,01</b>	<b>0,20</b>	<b>0,32</b>	<b>1,05</b>	<b>0,51</b>	<b>0,26</b>
Standard deviation s	<b>0,19</b>	<b>0,39</b>	<b>0,20</b>	<b>0,13</b>	<b>0,30</b>	<b>0,13</b>	<b>0,22</b>	<b>0,01</b>	<b>0,02</b>	<b>0,16</b>	<b>0,06</b>	<b>0,02</b>	<b>0,01</b>

\* value corresponding to only 1 trial

Table I – individual data for each participating laboratory

**II – Certified values**

	Los of Ignition	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	Chloride	Sulphide	Na <sub>2</sub> O	K <sub>2</sub> O
Value <sup>1</sup>	<b>1,51</b>	<b>29,61</b>	<b>10,14</b>	<b>3,27</b>	<b>45,12</b>	<b>4,46</b>	<b>3,17</b>	<b>0,01</b>	<b>0,20</b>	<b>0,32</b>	<b>1,05</b>
Uncertainty <sup>2</sup>	<b>0,14</b>	<b>0,26</b>	<b>0,13</b>	<b>0,09</b>	<b>0,20</b>	<b>0,09</b>	<b>0,15</b>	<b>0,01</b>	<b>0,02</b>	<b>0,11</b>	<b>0,04</b>

<sup>1</sup> best estimate from the average inter-laboratory test results

<sup>2</sup> values coming from the half width confidence interval C(95%). It is equal to  $C(95\%) = (t \cdot s) / \sqrt{n}$  where **t** is the appropriate Student's value, **n** the number of acceptable mean values et **s** the standard deviation.

Statistical analysis of these Inter Laboratory trials was conducted with the assistance of ATILH. Elimination of outliers is performed at 98 by % using Student test. A reiteration is set at this threshold to keep only the values associated with the "Normal or Gaussian" distribution and completely defined by two parameters: mean and standard deviation.

### III – Participating Laboratories - Traceability

An Inter-laboratory test campaign has been organized, laboratories of the Cement Industry in France and Europe as well as laboratories user of cement participated to this round robin test.

The 11 laboratories having participated are:

- CTG Italcementi rue des Technodes, 78931 Guerville Cedex France
- Lafarge Centre Recherche, 95 rue du Montmurier, 38291 St Quentin Fallavier Cedex France
- LERM, 23 rue de la Madeleine, BP 60136, 13631 Arles Cedex France
- CTG Italcementi , via G Camozzi 124, 24121 Bergamo, Italie
- Ceprochim, 6 blvd Preciziei code 062203, 6 Bucharest, Roumanie
- EKET , K Pateli 19, 141 23 Lycovyssi, Attica, Grèce
- Fluxana , sommerdeich 22, 47533 Kleve, Allemagne
- Oddzial MineralnychMaterialow,Budowlanych w Krakowie, 31983 Krakow, ul Cementowa 1, Pologne
- TFB, 10 lidenstrabe, 5103 Wildeg, Suisse
- Université Paul Verlaine, LEM UMR CNRS 7555, 1 bd Arago Metz technopole CP 87811, 57078 Metz Cedex 3 France
- Vicat, Centre Technique LC, BP 36, 38081 L'Isle d'Abeau Cedex France

### IV – Notes on Methods used

The test method most used is the X-ray fluorescence (ISO 29581-2) using fused bead. Other methods have been used by some laboratories as follows:

- Measure of silica, alumina, iron oxide, calcium oxide and magnesia; laboratories 1, 2, 3, 4, 5, 6, 9 and 11 have used X-ray fluorescence; laboratories 7 and 8 have used chemical method EN 196-2; laboratory 10 has used spectrometric ICP method.
- Measure of sulphuric anhydride (SO<sub>3</sub>); laboratories 1, 3, 5, 6, 9 and 11 have used X-ray fluorescence; laboratories 4, 7, 8 and 10 have used chemical method EN 196-2 ; laboratory 2 has used carbon and sulfur analyzer.
- Measure of sodium and potassium oxides; laboratories 1, 2, 3, 4, 5, 6, 9 and 11 have used X-ray fluorescence; laboratories 7, 8 and 10 have used chemical method EN 196-2.
- Loss of ignition has been measured for the 11 laboratories following chemical method EN 196-2.
- Measure of titania and phosphorus pentoxide ; laboratories 1, 2, 3 and 11 have used X-ray fluorescence.
- Measure of chloride; laboratories 2, 5 and 9 have used X-ray fluorescence using pellets; laboratories 4, 6, 7, 8, 10 and 11 have used chemical method EN 196-2; laboratory 1 has used chemical method EN 196-21.
- Measure of sulphide ; laboratories 1, 2, 5, 7, 8, 10 and 11 have used chemical method EN 196-2; laboratory 9 has used X-ray fluorescence using pellets; laboratory 4 has used a method derived from EN 196-21 using a double determination on the sample as it is and on a sample after calcination.

### V – Homogeneity

The batch of cement used for this campaign comes from the current production of cement. It was homogenized (Lödige mixer) and then distributed in 10 sealed drums and each containing a desiccant. Statistical analysis of laser particle size and uncorrected sulphide loss of ignition (EN 196-2), performed on samples taken from each of 10 drums, confirms its homogeneity (standard deviation of 0,018 on the loss of ignition - standard deviation of 0,49 on the mesh size of 8 micrometers).

### VI – Packaging – Intended used

The sample of this reference material is packaged in 40 g glass bottle, sealed with a secure screw cap. The physico-chemical 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. These samples are intended to validate results of chemical analysis of cement by the method of X-ray fluorescence.