

Sample Preparation and Calibration Packages for High Precision XRF Analysis

Accurate, precise and reproducible results in XRF analysis depend on a number of factors including manual sample handling; tractability of reference materials; the stability and performance of automatic sample processing equipment; the quality of the calibration and the performance of the spectrometer itself. A range of complete application packages is now available, including the preparation, installation and validation of the calibration on your spectrometer.

CemCal™ For Cement and Raw Meal Applications

The CemCal™ package includes

- Sample preparation method and recipes
- Certified Reference Materials for calibration and validation
- Preparation of Calibration Standards
- Drift Correction Samples
- Vulcan MA series fusion machine, accessories and consumables
- Installation of equipment, method and full application training
- Calibration of your XRF spectrometer, method / calibration validation

The application range for CemCal™ includes

- Basic Raw Meal
- Clinker
- Cement
- Raw Materials

Basic and Raw Material calibrations

The calibrations are based on selected Certified Reference Materials (CRM) so that analyses can be included in your appropriate quality management system.

The basic concentration ranges will be from the lower limit of detection (LLD) to the upper levels, shown in table 1. The actual LLD will depend on the type and model of spectrometer you are using. An extended calibration (for raw materials), shown in table 2, can be provided, which, depending on the elements of interest may require a different recipe.

Basic calibration for Raw Material, Clinker, Cement (Table 1)

Oxide	% max	Oxide	%max	Oxide	% max	Oxide	% max
Na ₂ O	1.0	P ₂ O ₅	0.5	TiO ₂	1.0	ZnO	0.1
MgO	6.0	SO ₃	5.0	Cr ₂ O ₃	0.05	SrO	1.0
Al ₂ O ₃	10.0	K ₂ O	2.0	Mn ₂ O ₃	0.2		
SiO ₂	30.0	CaO	70.0	Fe ₂ O ₃	5.0		

Extended calibration for Raw Materials, Silicates, Clay, Gypsum, Iron Ore (Table 2)

Oxide	% max	Oxide	%max	Oxide	% max	Oxide	% max
Na ₂ O	4.0	P ₂ O ₅	3.0	TiO ₂	3.0	ZnO	0.1
MgO	35.0	SO ₃	60.0	Cr ₂ O ₃	0.1	SrO	1.0
Al ₂ O ₃	100.0	K ₂ O	5.0	Mn ₂ O ₃	2.0		
SiO ₂	100.0	CaO	100.0	Fe ₂ O ₃	100.0		

Automatic fusion apparatus. The Vulcan MA series

The new Vulcan MA range of fusion machines uses gas burners with an optimum combination of gas, air and oxygen, controlled by automatic valves, enabling exactly reproducible adjustment of the fusion temperature. Up to ten different fusion programs can be stored permanently in memory, for instant recall.

The Vulcan MA is available for simultaneous fusion of two, four or six samples.

More detailed information on the Vulcan range of fusion machines is available in a separate publication.

Validation

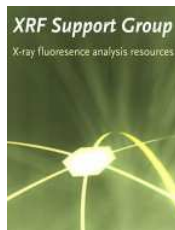
A certified reference material, which is not part of the calibration, is provided with the package as a validation standard. This allows validation of the complete analytical process including the fusion machine and XRF spectrometer. A portion of the standard is fused and analysed at defined periods for quality assessment.

In addition, a set of stable glass based setting up samples is provided for routine monitoring and spectrometer drift correction



The application package and calibration service is provided in partnership with the XRF Support Group, comprising, Fluxana GmbH & Co. KG, H D Elektronik GmbH and Dr Schlotz XRF Consulting and Training.

If you would like a quotation or just to discuss your applications and review the options for sample preparation equipment please contact Analysco, directly or through our web site.



FLUXANA
HD ELEKTRONIK UND
ELEKTROTECHNIK GmbH
www.fluxana.com

Dr.Schlotz
XRF Consulting & Training
www.x-ray-fluorescence.de

Supplementary information to CemCal Performance and validation

Accuracy and reproducibility

BCS 354	Cert	STD 01	STD 02	STD 03	STD 04	STD 05	STD 06	STD 07	RSD
SiO ₂	21.80	21.81	21.81	21.78	21.60	21.66	21.72	21.70	0.08
Al ₂ O ₃	4.85	4.82	4.82	4.89	4.88	4.88	4.91	4.89	0.04
TiO ₂	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.00
CaO	70.00	70.18	70.09	69.88	69.76	69.78	69.95	69.90	0.05
MgO	0.42	0.39	0.42	0.44	0.42	0.38	0.41	0.42	0.02
K ₂ O	0.11	0.10	0.11	0.11	0.12	0.11	0.11	0.11	0.01
Na ₂ O	0.10	0.07	0.10	0.11	0.05	0.10	0.17	0.12	0.04
P ₂ O ₅	0.12	0.121	0.131	0.135	0.13	0.131	0.128	0.13	0.00
Cr ₂ O ₃	0.00	0.001	0.00	0.005	0.003	0.004	0.003	0.002	0.00
SrO	0.11	0.106	0.105	0.106	0.105	0.103	0.105	0.106	0.00
SO ₃	2.25	2.214	2.113	2.154	2.121	2.127	2.142	2.154	0.03
Fe ₂ O ₃	0.30	0.297	0.303	0.31	0.31	0.31	0.30	0.30	0.01
Mn ₂ O ₃	0.06	0.059	0.163	0.058	0.059	0.06	0.06	0.06	0.00
ZnO		-0.003	-0.003	0.002	0.002	0.002	0.003	0.002	0.00
Sum	100.16	00.21	100.10	100.02	99.60	99.69	100.05	99.93	

NIST CRMs measured as unknowns

	Cert unignited	NIST 1880A-1 unignited	NIST 1880A-1 ignited
LOI	1.3	1.3	
SiO ₂	20.31	20.36	20.63
Al ₂ O ₃	5.18	5.11	5.18
TiO ₂	0.25	0.27	0.27
CaO	63.83	63.95	64.79
MgO	1.72	1.74	1.76
K ₂ O	0.92	0.88	0.89
Na ₂ O	0.19	0.20	0.20
P ₂ O ₅	0.22	0.22	0.23
Cr ₂ O ₃	0.007	0.01	0.01
SrO	0.083	0.09	0.09
SO ₃	3.25	3.23	3.27
Fe ₂ O ₃	2.81	2.82	2.85
Mn ₂ O ₃	0.127	0.13	0.13
ZnO	0.005	0.00	0.00
Sum	100.20	100.12	100.12

	Cert unignited	NIST 1881A-1 unignited	NIST 1881A-1 ignited
LOI	1.59	1.59	
SiO ₂	22.26	22.29	22.65
Al ₂ O ₃	7.06	7.06	7.17
TiO ₂	0.3663	0.36	0.37
CaO	57.58	57.57	58.50
MgO	2.981	2.91	2.96
K ₂ O	1.228	1.21	1.23
Na ₂ O	0.199	0.18	0.18
P ₂ O ₅	0.1459	0.15	0.149
Cr ₂ O ₃	0.0588	0.06	0.062
SrO	0.036	0.04	0.039
SO ₃	3.366	3.39	3.448
Fe ₂ O ₃	3.09	3.07	3.119
Mn ₂ O ₃	0.1042	0.11	0.109
ZnO	0.0489	0.05	0.05
Sum	100.11	100.12	99.85

Supplementary information to CemCal Performance and validation

NIST CRMs measured as unknowns

	Cert unignited	NIST 1882A-1 unignited	NIST 1882A-1 ignited
LOI	0.2	0.2	
SiO ₂	4.01	4.16	4.17
Al ₂ O ₃	39.14	39.08	39.16
TiO ₂	1.786	1.81	1.81
CaO	39.29	39.02	39.10
MgO	0.51	0.45	0.45
K ₂ O	0.051	0.06	0.06
Na ₂ O	0.021	<0.1	<0.1
P ₂ O ₅	0.07	0.08	0.08
Cr ₂ O ₃	0.113	0.11	0.115
SrO	0.024	0.02	0.021
SO ₃		0.05	0.049
Fe ₂ O ₃	14.67	14.95	14.975
Mn ₂ O ₃	0.06	0.04	0.043
ZnO	0.004	0.00	0.002
Sum	99.95	100.12	99.97

	Cert unignited	NIST 1883A-1 unignited	NIST 1883A-1 ignited
LOI	0.35	0.35	
SiO ₂	0.24	0.25	0.25
Al ₂ O ₃	70.04	70.02	70.27
TiO ₂	0.02	0.02	0.02
CaO	29.52	29.48	29.58
MgO	0.19	-0.07	-0.07
K ₂ O	0.014	0.02	0.02
Na ₂ O	0.3	0.18	0.18
P ₂ O ₅	0.003	0.00	0.001
Cr ₂ O ₃	0.006	0.01	0.006
SrO	0.019	0.02	0.018
SO ₃		0.00	0.005
Fe ₂ O ₃	0.078	0.07	0.066
Mn ₂ O ₃	0.003	0.00	0.033
ZnO		0.00	-0.033
Sum	100.78	100.12	100.33

	Cert unignited	NIST 1884A-1 unignited	NIST 1884A-1 ignited
LOI	1.06	1.06	
SiO ₂	20.57	20.71	20.93
Al ₂ O ₃	4.264	4.27	4.32
TiO ₂	0.186	0.18	0.18
CaO	62.26	62.44	63.11
MgO	4.475	4.55	4.60
K ₂ O	0.997	0.96	0.97
Na ₂ O	0.2161	0.09	0.09
P ₂ O ₅	0.1278	0.12	0.123
Cr ₂ O ₃	0.0166	0.01	0.015
SrO	0.298	0.29	0.296
SO ₃	2.921	2.91	2.943
Fe ₂ O ₃	2.695	2.65	2.682
Mn ₂ O ₃	0.0853	0.09	0.091
ZnO	0.0101	0.01	0.008
Sum	00.08	100.12	00.36

	Cert unignited	NIST 1886A-1 unignited	NIST 1886A-1 ignited
LOI	1.68	1.68	
SiO ₂	20.909	21.00	21.36
Al ₂ O ₃	4.026	3.97	4.04
TiO ₂	0.195	0.20	0.2
CaO	62.39	62.57	63.64
MgO	4.033	4.05	4.12
K ₂ O	0.206	0.22	0.22
Na ₂ O	1.068	0.89	0.91
P ₂ O ₅	0.122	0.12	0.122
Cr ₂ O ₃	0.195	0.02	0.018
SrO	0.638	0.62	0.632
SO ₃	2.83	2.80	2.849
Fe ₂ O ₃	1.929	1.94	1.974
Mn ₂ O ₃	0.0478	0.05	0.053
ZnO	0.0029	0.00	0
Sum	100.10	100.12	100.36

Supplementary information to CemCal Performance and validation

NIST CRMs measured as unknowns

	Cert Unignited	NIST 1886A-1 unignited	NIST 1886A-1 ignited
LOI	1.56	1.56	
SiO ₂	22.38	22.39	22.75
Al ₂ O ₃	3.875	3.86	3.92
TiO ₂	0.084	0.08	0.08
CaO	67.87	67.96	69.04
MgO	1.932	1.89	1.92
K ₂ O	0.093	0.08	0.08
Na ₂ O	0.021	<0.1	<0.1
P ₂ O ₅	0.022	0.03	0.028
Cr ₂ O ₃	0.0024	0.00	-0.002
SrO	0.018	0.02	0.02
SO ₃	2.086	2.03	2.058
Fe ₂ O ₃	0.152	0.14	0.141
Mn ₂ O ₃	0.0073	0.01	0.007
ZnO	0.001	0.00	-0.003
Sum	100.01	100.12	99.90

	Cert Unignited	NIST 1887A-1 unignited	NIST 1887A-1 ignited
LOI	1.43	1.43	
SiO ₂	18.637	18.71	18.98
Al ₂ O ₃	6.202	6.15	6.24
TiO ₂	0.2658	0.27	0.27
CaO	60.90	61.13	62.02
MgO	2.835	2.89	2.93
K ₂ O	1.1	1.06	1.08
Na ₂ O	0.4778	0.30	0.3
P ₂ O ₅	0.306	0.31	.0315
Cr ₂ O ₃	0.009	0.00	0.005
SrO	0.322	0.32	0.328
SO ₃	4.622	4.56	4.628
Fe ₂ O ₃	2.861	2.85	2.893
Mn ₂ O ₃	0.1186	0.12	0.125
ZnO	0.0667	0.07	0.071
Sum	100.15	100.12	100.18

	Cert Unignited	NIST 1888A-1 unignited	NIST 1888A-1 ignited
LOI	1.75	1.75	
SiO ₂	21.22	21.28	21.66
Al ₂ O ₃	4.265	4.28	4.36
TiO ₂	0.263	0.26	0.26
CaO	63.23	63.43	64.56
MgO	2.982	3.00	3.05
K ₂ O	0.526	0.49	0.5
Na ₂ O	0.1066	0.10	0.1
P ₂ O ₅	0.08	0.08	0.082
Cr ₂ O ₃	0.0186	0.02	0.02
SrO	0.082	0.09	0.087
SO ₃	2.131	2.19	2.233
Fe ₂ O ₃	3.076	3.12	3.174
Mn ₂ O ₃	0.1256	0.13	0.133
ZnO	0.107	0.11	0.117
Sum	99.96	100.12	100.25

	Cert Unignited	NIST 1889A-1 unignited	NIST 1889A-1 ignited
LOI	3.28	3.28	
SiO ₂	20.66	20.64	21.34
Al ₂ O ₃	3.89	3.78	3.91
TiO ₂	0.227	0.23	0.24
CaO	65.34	65.67	67.89
MgO	0.814	0.77	0.8
K ₂ O	0.605	0.57	0.59
Na ₂ O	0.195	0.19	0.2
P ₂ O ₅	0.11	0.11	0.111
Cr ₂ O ₃	0.0072	0.01	0.006
SrO	0.042	0.05	0.047
SO ₃	2.69	2.73	2.825
Fe ₂ O ₃	1.937	1.93	1.999
Mn ₂ O ₃	0.2588	0.27	0.281
ZnO	0.0048	0.00	0.002
Sum	100.06	100.12	100.12

Supplementary information to CemCal Performance and validation

JCA CRMs measured as unknowns

	Cert	FLX NO	Cert	FLX NO	Cert	FLX NO	Cert	FLX NO	Cert	FLX NO
		B0122		B0124		B0126		B0128		B0130
XRF	1	1	2	2	3	3	4	4	5	5
LOI		1.3		2		1.6		0.8		1.3
SiO ₂	22.23	22.11	21.31	21.3	20.67	20.5	20.71	20.65	20.52	20.5
Al ₂ O ₃	5.35	5.3	5.29	5.32	4.57	4.56	4.73	4.76	5.07	5.1
TiO ₂	0.33	0.33	0.31	0.31	0.28	0.27	0.26	0.27	0.25	0.25
CaO	64.14	63.78	65.17	64.96	66.32	65.98	99.17	65.85	65.99	65.8
MgO	1.75	1.76	1.77	1.77	1.53	1.53	1.37	1.36	0.94	0.99
K ₂ O	0.4	0.37	0.5	0.48	0.45	0.43	0.54	0.49	0.46	0.44
Na ₂ O	0.29	0.05	0.38	0.36	0.3	0.14	0.24	0.1	0.32	0.31
P ₂ O ₅	0.06	0.05	0.11	0.115	0.13	0.114	0.4	0.4	0.1	0.107
Cr ₂ O ₃		0.01		0.035		0.007		0.008		0.027
SrO	0.037	0.036	0.045	0.046	0.049	0.05	0.036	0.037	0.027	0.027
SO ₃	2.33	2.272	1.91	1.882	3.18	3.121	2.64	2.57	3.02	2.996
Fe ₂ O ₃	3.05	3.018	2.93	2.914	2.43	2.398	2.8	2.762	2.99	2.971
Mn ₂ O ₃	0.17	0.168	0.23	0.245	0.09	0.087	0.06	0.059	0.31	0.323
ZnO		0.025		0.087		0.063		0.05		0.053
Sum	100.137	99.2658	99.955	99.8148	99.999	99.238	99.956	99.3654	99.997	99.8899

	Cert	FLX NO	Cert	FLX NO	Cert	FLX NO	Cert	FLX NO	Cert	FLX NO
		B0132		B0134		B0136		B0138		B0140
XRF	6	6	7	7	8	8	9	9	10	10
LOI		1.4		2		1.3		1.5		0.8
SiO ₂	20.71	20.74	22.76	22.68	23.23	23.19	23.82	23.78	22.99	23.07
Al ₂ O ₃	5.02	5	4.26	4.25	3.82	3.81	3.4	3.4	6.29	6.31
TiO ₂	0.24	0.25	0.25	0.25	0.27	0.28	0.16	0.16	0.52	0.52
CaO	66.23	66.06	64.27	64.1	64.15	64.11	64.75	64.67	61.67	61.83
MgO	1.81	1.81	1.03	1.02	1.52	1.51	0.78	0.76	2.71	2.71
K ₂ O	0.23	0.2	0.35	0.33	0.54	0.49	0.39	0.37	0.62	0.57
Na ₂ O	0.26	0.05	0.17	0.12	0.1	-0.01	0.24	0.2	0.17	0.08
P ₂ O ₅	0.05	0.055	0.06	0.069	0.19	0.181	0.06	0.061	0.13	0.135
Cr ₂ O ₃		0.014		0.015		0.018		0.013		0.011
SrO	0.035	0.036	0.03	0.03	0.038	0.037	0.024	0.024	0.043	0.043
SO ₃	2.61	2.537	2.42	2.344	1.93	0.9	1.94	1.917	2.25	2.278
Fe ₂ O ₃	2.7	2.683	4.11	4.096	4.02	4.022	4.18	4.172	2.39	2.395
Mn ₂ O ₃	0.21	0.22	0.07	0.071	0.23	0.245	0.12	0.131	0.16	0.161
ZnO		0.064		0.131		0.041		0.147		0.067
Sum	100.105	99.7027	99.78	99.504	100.038	99.8335	99.864	99.8194	99.943	100.1826

Supplementary information to CemCal Performance and validation

JCA CRMs measured as unknowns

	Cert	FLX NO	Cert	FLX NO	Cert	FLX NO	Cert	FLX NO	Cert	FLX NO
		B0142		B0144		B0146		B0148		B0150
XRF	11	11	12	12	13	13	14	14	15	15
LOI		1.2		1		1.5		0.6		0
SiO ₂	24.42	24.52	26.34	26.39	26.62	26.69	25.74	25.85	29.29	29.26
Al ₂ O ₃	7.37	7.45	8.95	8.98	9.22	9.26	8.7	8.79	10.7	10.78
TiO ₂	0.55	0.56	0.73	0.72	0.41	0.42	0.66	0.67	0.64	0.64
CaO	59.15	59.36	54.9	54.91	55.36	55.36	55.15	55.33	49.28	48.25
MgO	2.63	2.66	3.33	3.34	2.98	2.95	3.98	4.02	5.12	5.11
K ₂ O	0.51	0.47	0.44	0.41	0.41	0.37	0.31	0.24	0.42	0.37
Na ₂ O	0.26	0.18	0.23	0.12	0.3	0.08	0.26	0.16	0.25	0.03
P ₂ O ₅	0.23	0.219	0.17	0.178	0.06	0.043	0.04	0.033	0.06	0.051
Cr ₂ O ₃		0.009		0.007		0.02		0.014		0.01
SrO	0.046	0.046	0.051	0.049	0.037	0.037	0.051	0.051	0.071	0.069
SO ₃	2.5	2.51	2.9	2.917	1.94	1.942	2.93	2.951	2.56	2.557
Fe ₂ O ₃	2.26	2.251	1.82	1.8	2.02	2.014	2.03	2.018	1.32	1.299
Mn ₂ O ₃	0.18	0.179	0.2	0.206	0.68	0.704	0.31	0.321	0.53	0.55
ZnO		0.03		0.024		0.035		0.046		0.032
Sum	11.106	100.4463	100.061	100.0635	100.037	99.9319	100.161	100.491	100.241	100.0043

Supplementary information to CemCal Performance and validation

Gypsum CRMs measured as unknowns (FluXana Raw Materials Calibration)

FLX GYP C	631	630	629	628	627	626	625	624	623	622	Mean	RSD	Cert
SiO ₂	4.77	4.81	4.76	4.75	4.84	4.74	4.72	4.74	4.78	4.82	4.77	0.039	4.74
Al ₂ O ₃	1.05	1.04	1.01	1.04	1.03	1.03	1.03	1.03	1.04	1.08	1.04	0.018	1.07
Fe ₂ O ₃	0.532	0.528	0.524	0.523	0.528	0.528	0.532	0.529	0.531	0.536	0.53	0.004	0.54
CaO	41.21	41.14	41.0	41.08	41.08	41.13	41.17	41.16	41.2	41.32	41.16	0.073	41.17
MgO	6.97	6.94	6.96	7.06	6.94	7.02	7.05	6.92	7.08	7.04	7.00	0.058	7.25
SO ₃	44.62	44.58	44.38	44.56	44.4	44.32	44.53	44.76	44.58	44.7	44.54	0.140	44.69
K ₂ O	0.42	0.41	0.42	0.42	0.42	0.42	0.42	0.42	0.44	0.43	0.42	0.008	0.49
P ₂ O ₅	0.025	0.02	0.024	0.031	0.028	0.03	0.034	0.021	0.016	0.031	0.03	0.006	0.02

FLX GYP D	618	615	614	617	616	613	612	611	Mean	RSD	Cert
SiO ₂	11.17	10.96	11.17	11.06	11.07	10.97	10.98	11.06	11.06	0.083	11
Al ₂ O ₃	2.46	2.45	2.44	2.46	2.41	2.41	2.42	2.44	2.44	0.021	2.57
Fe ₂ O ₃	1.333	1.319	1.341	1.346	1.291	1.343	1.336	1.312	1.33	0.019	1.37
CaO	35.73	35.89	35.74	35.82	35.73	35.75	35.83	35.8	35.79	0.058	35.66
MgO	2.06	2.08	1.99	2.09	2.03	1.98	2.08	2.06	2.05	0.042	2.19
SO ₃	46.42	46.67	46.36	46.21	46.19	46.59	46.37	46.69	46.50	0.191	46.41
K ₂ O	0.67	0.67	0.67	0.68	0.67	0.69	0.67	0.66	0.67	0.009	0.68
P ₂ O ₅	0.032	0.031	0.035	0.028	0.035	0.029	0.025	0.036	0.03	0.004	0.03

FLX GYP A	607	606	605	604	603	602	601	Mean	RSD	Cert
SiO ₂	0.69	0.62	0.67	0.68	0.7	0.61	0.6	0.65	0.042	0.56
Al ₂ O ₃	0.11	0.11	0.11	0.11	0.12	0.1	0.1	0.11	0.007	0.13
Fe ₂ O ₃	0.041	0.036	0.037	0.046	0.045	0.038	0.04	0.04	0.004	0.06
CaO	41.24	41.56	41.46	41.34	41.34	41.47	41.27	41.38	0.117	41.17
MgO	0.25	0.2	0.26	0.23	0.24	0.25	0.23	0.24	0.020	0.23
SO ₃	57.74	57.46	57.63	57.56	57.7	57.48	57.45	57.57	0.118	57.81
K ₂ O	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.000	0.03
P ₂ O ₅	0.015	0.005	0.007	0.008	0.01	0.012		0.01	0.004	0.01