

Preliminary Work On Characterizing B Ka in Mg borides

04-06-2011 (Updated 10-31-2011)

I recently attempted to analyze Mg borides using EPMA. I hadn't done much serious work on boron previously and decided to look at the whole ball of wax again. As everyone knows there is a large absorption correction (250 to 300%) for this system and of course the peak shape issues need to be examined.

First I measured the B Ka signal in boron metal, boron nitride and also the unknown samples which were supposedly MgB₄ and MgB₂ at a number of voltages (5 to 20 keV) to get the MAC for B Ka in B, N and Mg. An empirically measured B Ka in Mg MAC had not been published previously interestingly enough.

Here are the values I obtained using Pouchou's Xmac program running from 5 to 20 keV using a Cameca PC25 LDE crystal:

Emitter	Absorber	MAC	Material
B Ka	B	3068	Boron metal
B Ka	B	2750	Boron nitride (assumed 50:50 composition)
B Ka	N	10421	Boron nitride (assumed 50:50 composition)
B Ka	Mg	54251	MgB ₄ (assumed stoichiometric)
B Ka	Mg	54834	MgB ₂ (assumed stoichiometric)

By the way, I don't think my BN is quite stoichiometric. I think there is a binder in it, possibly carbon based (oxygen I measured at ~0.4 wt %) but I can't find anyone that has published actual compositions on BN. If anyone knows of empirical measurements on cubic BN for B, N, C and O please let me know. It polishes as a very fine grained sintered material, appears beam stable and fluoresces bright blue under the beam.

I've assumed a (very) rough average B Ka MAC for Mg borides at 54,500 though there may actually be a systematic difference between the compounds due to the Mg L absorption edge moving around with the different bonding though it's not that close.

I also measured the APF factors to deal with the expected peak shift/shape changes and they are significant, at least for the MgB₂. Fortunately these materials are all binaries so it's easy to extract the APF factors for use as compound APFs. That is one can easily measure the effect of B Ka in the presence of Mg by measuring for example, a wavescan on boron metal (the "std"), and then wavescans on the MgB₄ and MgB₂ (the "unkns"). The APF work was done at 15 keV on an SX100 using a PC25 crystal from Cameca which is optimized for boron.

But note: depending on whether you keep the peak position constant for the different materials (and incorporate the peak shift effect in the APF calculation) or use a different peak position for each material (and minimize the peak shift and essentially only correct for peak shape changes, not to mention also improving your counting statistics by staying on the peak), how you calculate the APF is slightly different for the two cases.

In each case one can use the Probe for EPMA Model Background dialog to get the peak and integrated areas on wavescans acquired with sufficient precision. Though one might have to utilize a deconvolution package if there are other extraneous peaks present in other systems. Note that if one clicks the Integrate button the program calculates the Peak and Integrated intensities and also the P/I and I/P intensities. Which ratio you would choose for a given wavescan depends on whether this was the "std" material or the "unk" material for the APF calculation. That is, use the P/I ratio from your standard material and the I/P ratio from your unknown material.

But let's say you wanted to use the same peak position for both the std and the unk and correct for both peak shift and peak shape. Now in the case of say, sulfur, the change between pyrite and anhydrite is almost all peak shift, so you probably wouldn't want to use the same spectrometer position for both materials. But if the spectral change between the two materials is mostly peak shape then you probably would use the same spectrometer positions and just correct for changes in peak shape.

The point is it works either way, but one should think carefully about it. By default, the Probe for EPMA program uses the peak position recorded for each wavescan sample. Now if you are using the same peak position for all samples (and correcting for both peak shift and peak shape changes) that is fine. Multiply the appropriate P/I and I/P ratio from your two materials, edit your EMPAPF.DAT file and you are done.

But if you want to minimize the peak shift effect and use the optimum peak position for each material (and optimize your counting statistics), and only correct for peak shape changes, you should either make sure that each wavescan has the correct on-peak position specified before acquisition. Or you can click one of the peak fit options in the Model Background dialog (usually Maxima or Highest). Then click the Integrate button again and the program will recalculate the P/I and I/P values based on the newly fitted peak position.

Of course in this case, you must use the same optimized peak positions for your actual quantitative standard and unknown acquisitions. The APF values I found doing this (using different optimized peak positions for each material) are (relative to boron metal):

"b"	"ka"	"n"	1.029	"BN/B/PC25/147.6"
"b"	"ka"	"mg"	1.004	"MgB4/B/PC25/147.6"
"b"	"ka"	"mg"	0.928	"MgB2/B/PC25/147.6"

In the case of utilizing a fixed peak position from the boron metal, the APF values are somewhat larger as would be expected to account for the peak shift effects relative to boron metal in addition to peak shape effects as shown here:

"b"	"ka"	"n"	1.214	"BN/B/PC25/147.6"
"b"	"ka"	"mg"	1.017	"MgB4/B/PC25/147.6"
"b"	"ka"	"mg"	0.937	"MgB2/B/PC25/147.6"

Anyway, doing this I get fairly reasonable quant values for these Mg borides, but the stoichiometries are somewhat variable depending on which matrix correction is used (not to mention the totals).

I used the APFs relative to boron metal at the optimized peak position for each material (1.004 for MgB4 and 0.928 for MgB2). One can simply toggle the APF flag in the Analytical Options dialog to turn the APF calculation on or off for each compound. I'm also using the above empirically measured B Ka MACs to override the FFAST MAC values.

The following data was all measured at 6 keV, 30 nA on a Cameca Sx100 using TAP for Mg Ka, PC12 for O ka and PC25 for B Ka. More experimental conditions are listed at the end.

Summary of All Calculated (averaged) Matrix Corrections:

Un 11 Mg-B dark phase9
FFAST Chantler (NIST v 2.1, 2005)

Elemental Weight Percents:

ELEM:	Mg	B	O	B	TOTAL	
1	34.307	67.090	.449	.000	101.846	Armstrong/Love Scott (default)
2	30.786	68.445	.399	.000	99.630	Conventional Philibert/Duncumb-Reed
3	34.289	67.778	.444	.000	102.511	Heinrich/Duncumb-Reed

4	34.569	65.861	.429	.000	100.860	Love-Scott I
5	34.523	65.603	.425	.000	100.551	Love-Scott II
6	34.959	66.070	.443	.000	101.471	Packwood Phi(pz) (EPQ-91)
7	40.293	67.025	.504	.000	107.821	Bastin (original) Phi(pz)
8	36.085	59.091	.447	.000	95.623	Bastin PROZA Phi(pz) (EPQ-91)
9	36.004	70.083	.456	.000	106.543	Pouchou and Pichoir - Full
10	36.007	69.188	.457	.000	105.652	Pouchou and Pichoir - Simplified
AVER:	35.182	66.623	.445	.000	102.251	
SDEV:	2.358	3.023	.027	.000	3.613	
SERR:	.746	.956	.009	.000		
MIN:	30.786	59.091	.399	.000	95.623	
MAX:	40.293	70.083	.504	.000	107.821	

Atomic Percents:

ELEM:	Mg	B	O	B	TOTAL	
1	18.463	81.170	.367	.000	100.000	Armstrong/Love Scott (default)
2	16.618	83.055	.327	.000	100.000	Conventional Philibert/Duncumb-Reed
3	18.304	81.336	.360	.000	100.000	Heinrich/Duncumb-Reed
4	18.861	80.783	.356	.000	100.000	Love-Scott I
5	18.901	80.745	.353	.000	100.000	Love-Scott II
6	18.983	80.652	.365	.000	100.000	Packwood Phi(pz) (EPQ-91)
7	21.015	78.586	.399	.000	100.000	Bastin (original) Phi(pz)
8	21.277	78.323	.400	.000	100.000	Bastin PROZA Phi(pz) (EPQ-91)
9	18.535	81.108	.357	.000	100.000	Pouchou and Pichoir - Full
10	18.730	80.909	.361	.000	100.000	Pouchou and Pichoir - Simplified
AVER:	18.969	80.667	.365	.000	100.000	
SDEV:	1.333	1.354	.022	.000	.000	
SERR:	.422	.428	.007	.000		
MIN:	16.618	78.323	.327	.000	100.000	
MAX:	21.277	83.055	.400	.000	100.000	

Summary of All Calculated (averaged) Matrix Corrections:

Un 14 Mg-B light phase9
FFAST Chantler (NIST v 2.1, 2005)

Elemental Weight Percents:

ELEM:	Mg	B	O	B	TOTAL	
1	49.046	54.731	.536	.000	104.313	Armstrong/Love Scott (default)
2	44.941	57.511	.505	.000	102.956	Conventional Philibert/Duncumb-Reed
3	48.760	54.988	.547	.000	104.295	Heinrich/Duncumb-Reed
4	49.155	53.045	.531	.000	102.731	Love-Scott I
5	49.107	53.631	.527	.000	103.264	Love-Scott II
6	49.654	53.114	.544	.000	103.312	Packwood Phi(pz) (EPQ-91)
7	55.235	52.819	.602	.000	108.656	Bastin (original) Phi(pz)
8	50.618	44.949	.542	.000	96.110	Bastin PROZA Phi(pz) (EPQ-91)
9	50.630	57.589	.559	.000	108.778	Pouchou and Pichoir - Full
10	50.583	56.167	.558	.000	107.309	Pouchou and Pichoir - Simplified
AVER:	49.773	53.854	.545	.000	104.172	
SDEV:	2.532	3.595	.026	.000	3.671	
SERR:	.801	1.137	.008	.000		
MIN:	44.941	44.949	.505	.000	96.110	
MAX:	55.235	57.589	.602	.000	108.778	

Atomic Percents:

ELEM:	Mg	B	O	B	TOTAL	
1	28.381	71.147	.471	.000	100.000	Armstrong/Love Scott (default)
2	25.690	73.871	.439	.000	100.000	Conventional Philibert/Duncumb-Reed
3	28.169	71.351	.480	.000	100.000	Heinrich/Duncumb-Reed
4	29.068	70.454	.477	.000	100.000	Love-Scott I
5	28.819	70.711	.470	.000	100.000	Love-Scott II
6	29.246	70.267	.486	.000	100.000	Packwood Phi(pz) (EPQ-91)
7	31.602	67.875	.524	.000	100.000	Bastin (original) Phi(pz)
8	33.219	66.241	.540	.000	100.000	Bastin PROZA Phi(pz) (EPQ-91)
9	27.995	71.535	.470	.000	100.000	Pouchou and Pichoir - Full

10	28.482	71.040	.478	.000	100.000	Pouchou and Pichoir - Simplified
AVER:	29.067	70.449	.484	.000	100.000	
SDEV:	2.053	2.081	.029	.000	.000	
SERR:	.649	.658	.009	.000		
MIN:	25.690	66.241	.439	.000	100.000	
MAX:	33.219	73.871	.540	.000	100.000	

So then I decided to try measuring integrated intensities to avoid any APF issues (and the seemingly small but possibly significant crystallographic effect I observed on the APFs when measuring different grains) (though acquisition is slow!). The calculations below are using all 10 matrix corrections (note the new feature which also calculates atomic percents for all 10 matrix corrections if specified by the user- nice for comparing stoichiometry).

Summary of All Calculated (averaged) Matrix Corrections:

Un 2 MgB4-1
FFAST Chantler (NIST v 2.1, 2005)

Elemental Weight Percents:

ELEM:	Mg	B	O	TOTAL	
1	35.413	66.591	.507	102.511	Armstrong/Love Scott (default)
2	31.654	68.225	.468	100.347	Conventional Philibert/Duncumb-Reed
3	35.207	67.344	.520	103.070	Heinrich/Duncumb-Reed
4	35.493	65.323	.503	101.319	Love-Scott I
5	35.445	65.073	.498	101.016	Love-Scott II
6	35.894	65.582	.519	101.995	Packwood Phi(pz) (EPQ-91)
7	41.274	66.220	.589	108.083	Bastin (original) Phi(pz)
8	36.984	58.299	.523	95.807	Bastin PROZA Phi(pz) (EPQ-91)
9	36.942	69.591	.534	107.067	Pouchou and Pichoir - Full
10	36.943	68.686	.535	106.165	Pouchou and Pichoir - Simplified
AVER:	36.125	66.094	.519	102.738	
SDEV:	2.377	3.126	.031	3.629	
SERR:	.752	.989	.010		
MIN:	31.654	58.299	.468	95.807	
MAX:	41.274	69.591	.589	108.083	

Atomic Percents:

ELEM:	Mg	B	O	TOTAL	
1	19.050	80.534	.415	100.000	Armstrong/Love Scott (default)
2	17.042	82.575	.383	100.000	Conventional Philibert/Duncumb-Reed
3	18.788	80.791	.422	100.000	Heinrich/Duncumb-Reed
4	19.383	80.199	.418	100.000	Love-Scott I
5	19.422	80.163	.415	100.000	Love-Scott II
6	19.495	80.077	.428	100.000	Packwood Phi(pz) (EPQ-91)
7	21.605	77.926	.469	100.000	Bastin (original) Phi(pz)
8	21.904	77.624	.471	100.000	Bastin PROZA Phi(pz) (EPQ-91)
9	19.022	80.560	.418	100.000	Pouchou and Pichoir - Full
10	19.224	80.353	.424	100.000	Pouchou and Pichoir - Simplified
AVER:	19.493	80.080	.426	100.000	
SDEV:	1.386	1.411	.026	.000	
SERR:	.438	.446	.008		
MIN:	17.042	77.624	.383	100.000	
MAX:	21.904	82.575	.471	100.000	

Summary of All Calculated (averaged) Matrix Corrections:

Un 3 MgB2-1
FFAST Chantler (NIST v 2.1, 2005)

Elemental Weight Percents:

ELEM:	Mg	B	O	TOTAL	
1	53.042	54.432	.617	108.091	Armstrong/Love Scott (default)
2	48.798	57.240	.585	106.623	Conventional Philibert/Duncumb-Reed
3	52.733	54.645	.629	108.007	Heinrich/Duncumb-Reed

4	53.160	52.692	.612	106.464	Love-Scott I
5	53.110	53.376	.608	107.093	Love-Scott II
6	53.682	52.840	.625	107.147	Packwood Phi(pz) (EPQ-91)
7	59.417	52.282	.689	112.388	Bastin (original) Phi(pz)
8	54.491	44.272	.622	99.385	Bastin PROZA Phi(pz) (EPQ-91)
9	54.663	57.243	.643	112.549	Pouchou and Pichoir - Full
10	54.608	55.845	.642	111.095	Pouchou and Pichoir - Simplified
AVER:	53.770	53.487	.627	107.884	
SDEV:	2.604	3.703	.028	3.792	
SERR:	.824	1.171	.009		
MIN:	48.798	44.272	.585	99.385	
MAX:	59.417	57.243	.689	112.549	

Atomic Percents:

ELEM:	Mg	B	O	TOTAL	
1	30.081	69.387	.532	100.000	Armstrong/Love Scott (default)
2	27.361	72.141	.498	100.000	Conventional Philibert/Duncumb-Reed
3	29.874	69.584	.542	100.000	Heinrich/Duncumb-Reed
4	30.812	68.648	.539	100.000	Love-Scott I
5	30.521	68.948	.531	100.000	Love-Scott II
6	30.958	68.494	.548	100.000	Packwood Phi(pz) (EPQ-91)
7	33.384	66.028	.589	100.000	Bastin (original) Phi(pz)
8	35.167	64.222	.610	100.000	Bastin PROZA Phi(pz) (EPQ-91)
9	29.658	69.811	.530	100.000	Pouchou and Pichoir - Full
10	30.152	69.310	.539	100.000	Pouchou and Pichoir - Simplified
AVER:	30.797	68.657	.546	100.000	
SDEV:	2.129	2.160	.032	.000	
SERR:	.673	.683	.010		
MIN:	27.361	64.222	.498	100.000	
MAX:	35.167	72.141	.610	100.000	

So far it appears the MgB4 is fairly stoichiometric, but the MgB2 seems a little light heavy on the boron side. I then calculated them all just doing boron by difference as a sanity check:

Summary of All Calculated (averaged) Matrix Corrections:

Un 11 Mg-B dark phase9
FFAST Chantler (NIST v 2.1, 2005)

Elemental Weight Percents:

ELEM:	Mg	B	O	B	TOTAL	
1	34.479	.000	.432	65.089	100.000	Armstrong/Love Scott (default)
2	30.782	.000	.399	68.819	100.000	Conventional Philibert/Duncumb-Reed
3	34.275	.000	.443	65.283	100.000	Heinrich/Duncumb-Reed
4	34.564	.000	.429	65.007	100.000	Love-Scott I
5	34.519	.000	.425	65.056	100.000	Love-Scott II
6	34.951	.000	.442	64.607	100.000	Packwood Phi(pz) (EPQ-91)
7	39.959	.000	.497	59.543	100.000	Bastin (original) Phi(pz)
8	36.163	.000	.449	63.387	100.000	Bastin PROZA Phi(pz) (EPQ-91)
9	35.896	.000	.453	63.651	100.000	Pouchou and Pichoir - Full
10	35.911	.000	.454	63.634	100.000	Pouchou and Pichoir - Simplified
AVER:	35.150	.000	.442	64.408	100.000	
SDEV:	2.270	.000	.025	2.294	.000	
SERR:	.718	.000	.008	.726		
MIN:	30.782	.000	.399	59.543	100.000	
MAX:	39.959	.000	.497	68.819	100.000	

Atomic Percents:

ELEM:	Mg	B	O	B	TOTAL	
1	19.002	.000	.362	80.636	100.000	Armstrong/Love Scott (default)
2	16.542	.000	.326	83.132	100.000	Conventional Philibert/Duncumb-Reed
3	18.864	.000	.370	80.766	100.000	Heinrich/Duncumb-Reed
4	19.060	.000	.359	80.581	100.000	Love-Scott I
5	19.029	.000	.356	80.616	100.000	Love-Scott II

6	19.325	.000	.371	80.303	100.000	Packwood Phi(pz) (EPQ-91)
7	22.891	.000	.433	76.676	100.000	Bastin (original) Phi(pz)
8	20.165	.000	.381	79.454	100.000	Bastin PROZA Phi(pz) (EPQ-91)
9	19.980	.000	.383	79.637	100.000	Pouchou and Pichoir - Full
10	19.990	.000	.384	79.626	100.000	Pouchou and Pichoir - Simplified
AVER:	19.485	.000	.372	80.143	100.000	
SDEV:	1.573	.000	.027	1.600	.000	
SERR:	.497	.000	.009	.506		
MIN:	16.542	.000	.326	76.676	100.000	
MAX:	22.891	.000	.433	83.132	100.000	

Summary of All Calculated (averaged) Matrix Corrections:
 Un 14 Mg-B light phase9
 FFAST Chantler (NIST v 2.1, 2005)

Elemental Weight Percents:

ELEM:	Mg	B	O	B	TOTAL	
1	48.995	.000	.533	50.471	100.000	Armstrong/Love Scott (default)
2	45.039	.000	.505	54.456	100.000	Conventional Philibert/Duncumb-Reed
3	48.709	.000	.544	50.746	100.000	Heinrich/Duncumb-Reed
4	49.121	.000	.530	50.349	100.000	Love-Scott I
5	49.069	.000	.526	50.405	100.000	Love-Scott II
6	49.597	.000	.542	49.861	100.000	Packwood Phi(pz) (EPQ-91)
7	54.555	.000	.592	44.853	100.000	Bastin (original) Phi(pz)
8	50.748	.000	.546	48.706	100.000	Bastin PROZA Phi(pz) (EPQ-91)
9	50.356	.000	.553	49.091	100.000	Pouchou and Pichoir - Full
10	50.345	.000	.553	49.102	100.000	Pouchou and Pichoir - Simplified
AVER:	49.654	.000	.542	49.804	100.000	
SDEV:	2.344	.000	.023	2.366	.000	
SERR:	.741	.000	.007	.748		
MIN:	45.039	.000	.505	44.853	100.000	
MAX:	54.555	.000	.592	54.456	100.000	

Atomic Percents:

ELEM:	Mg	B	O	B	TOTAL	
1	30.071	.000	.494	69.434	100.000	Armstrong/Love Scott (default)
2	26.829	.000	.454	72.717	100.000	Conventional Philibert/Duncumb-Reed
3	29.831	.000	.503	69.666	100.000	Heinrich/Duncumb-Reed
4	30.177	.000	.492	69.331	100.000	Love-Scott I
5	30.133	.000	.488	69.380	100.000	Love-Scott II
6	30.583	.000	.504	68.913	100.000	Packwood Phi(pz) (EPQ-91)
7	34.976	.000	.573	64.451	100.000	Bastin (original) Phi(pz)
8	31.571	.000	.513	67.917	100.000	Bastin PROZA Phi(pz) (EPQ-91)
9	31.232	.000	.518	68.250	100.000	Pouchou and Pichoir - Full
10	31.222	.000	.518	68.260	100.000	Pouchou and Pichoir - Simplified
AVER:	30.663	.000	.506	68.832	100.000	
SDEV:	2.009	.000	.030	2.039	.000	
SERR:	.635	.000	.010	.645		
MIN:	26.829	.000	.454	64.451	100.000	
MAX:	34.976	.000	.573	72.717	100.000	

So even just calculating boron by difference from Mg and O (which should be no big deal as the absorption correction for Mg is small, ~7%) we still see that the MgB2 is a little low in Mg and too high in boron (The MgB4 looks excellent). I think this consistent result between all three methods is therefore real, so far as I can tell.

Experimental Conditions (based on MgB2)

Un 26 Mg-B light phase11
 TakeOff = 40.0 KiloVolt = 6.0 Beam Current = 30.0 Beam Size = 2

Column Condition Method Specified (1), Column Condition String =
 C:\UserData\Bohnenstiehl\03-2011\6 keV, 4, 0.pcc
 (Magnification (analytical) = 40000), Beam Mode = Analog Spot
 (Magnification (default) = 400, Magnification (imaging) = 3632)
 Image Shift (X,Y): 0, 0

Compositional analyses were acquired on an electron microprobe (Cameca SX100 (TCP/IP Socket)) equipped with 5 tunable wavelength dispersive spectrometers. Operating conditions were 40 degrees takeoff angle, and a beam energy of 6 keV. The beam current was 30 nA, and the beam diameter was 2 microns.

Elements were acquired using analyzing crystals LTAP for Mg ka, PC1 for O ka, and PC25 for B ka.

The standards were MgO synthetic for Mg ka, O ka, and Boron metal for B ka. The counting time was 60 seconds for all elements. The off peak counting time was 30 seconds for all elements. The off peak correction method was Exponential for all elements.

Unknown and standard intensities were corrected for deadtime. Standard intensities were corrected for standard drift over time.

Empirical Mass Absorption Coefficients were utilized to correct x-ray intensities for matrix corrections.

See Bastin, G.F. and Heijligers, H.J.M (1991) Quantitative electron probe microanalysis of ultra-light elements (boron - oxygen), in Electron Probe Quantitation, ed K.F.J. Heinrich and D.E. Newbury, Plenum Press, NY, 145-161

Also Bastin, G.F. and Heijligers, H.J.M. (1992) Present and future of light element analysis with electron beam instruments, Microbeam Analysis, 1, 61-73.

Current Mass Absorption Coefficients From:
 FFAST Chantler (NIST v 2.1, 2005)

Z-LINE	X-RAY	Z-ABSOR	MAC
Mg	ka	Mg	4.4533e+02
Mg	ka	B	5.7871e+02
Mg	ka	O	2.3871e+03
Mg	ka	B	5.7871e+02
B	ka	Mg	5.4500e+04 *
B	ka	B	3.0680e+03 *
B	ka	O	1.3696e+04
B	ka	B	3.0680e+03 *
O	ka	Mg	4.3851e+03
O	ka	B	6.8087e+03
O	ka	O	1.1204e+03
O	ka	B	6.8087e+03

* indicates empirical MAC

Empirical Mass Absorption Coefficients From:
 C:\Probe Software\Probe for EPMA\EMPMAC.DAT

Z-LINE	X-RAY	Z-ABSOR	MAC
B	ka	Mg	5.4500e+04 Donovan (2011)
B	ka	B	3.0680e+03 Donovan (2011)
B	ka	B	3.0680e+03 Donovan (2011)

Area Peak Factors were utilized to correct x-ray intensities for wavelength peak shift and/or shape changes for compound compositions by summing binary APF values.

See G. F. Bastin and H. J. M. Heijligers, Quantitative Electron Probe Microanalysis of Carbon in Binary Carbides, Parts I and II, X-Ray Spectr. 15: 135-150, 1986

Empirical Area Peak Factors From:
C:\Probe Software\Probe for EPMA\EMPAPF.DAT

Z-LINE	X-RAY	Z-ABSOR	APF
B	ka	Mg	.9280 MgB2/B/WSi/59.8

Results are the average of 10 points and detection limits ranged from .016 weight percent for Mg ka to .040 weight percent for B ka.

Analytical sensitivity (at the 99% confidence level) ranged from .103 percent relative for Mg ka to 3.020 percent relative for O ka.

The exponential or polynomial background fit was utilized.

See John J. Donovan, Heather A. Lowers and Brian G. Rusk, Improved electron probe microanalysis of trace elements in quartz, American Mineralogist, 96, 274-282, 2011

The matrix correction method was ZAF or Phi-Rho-Z calculations and the mass absorption coefficients dataset was FFAST Chantler (NIST v 2.1, 2005).

See J. T. Armstrong, Quantitative analysis of silicates and oxide minerals: Comparison of Monte-Carlo, ZAF and Phi-Rho-Z procedures, Microbeam Analysis--1988, p 239-246