



## **Chapter T**

# **Major Element Analysis by Wavelength Dispersive X-ray Fluorescence Spectrometry**

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# Major element analysis by wavelength dispersive X-ray fluorescence spectrometry

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## Principle

Ten major elements are determined in rocks and minerals by wavelength dispersive X-ray fluorescence spectrometry (WDXRF). The sample is first ignited, then fused with lithium tetraborate and the resultant glass disc is introduced into a wavelength dispersive X-ray spectrometer. The disc is irradiated with X-rays from an X-ray tube. X-ray photons emitted by the elements in the sample are counted and concentrations determined using previously prepared calibration standards. In addition to 10 major elements, the method provides a gravimetric loss-on-ignition.

## Interferences

Interferences, with analysis by WDXRF, may result from mineralogical or other structural effects, line overlaps, and matrix effects. The structure of the sample, mineralogical or otherwise, is eliminated through fusion with a suitable flux. Fusion of the sample diminishes matrix effects and produces a stable, flat, homogenous sample for presentation to the spectrometer. Selecting certain types of crystal monochromators eliminates many of the line overlaps and multi-order line interferences. A mathematical correction procedure (deJongh, 1973) is used to correct for the absorption and enhancement matrix effects.

## Scope

Concentrations of the elements in rocks and minerals are determined independent of the oxidation state and are reported in the oxidation state in which they most commonly occur in the earth's crust. The reporting limits (calibration range) for 10 elements by WDXRF are as follows.

Element	Concentration range (percent)	
SiO <sub>2</sub>	0.10	99.0
Al <sub>2</sub> O <sub>3</sub>	0.10	58.0
Fe <sub>2</sub> O <sub>3</sub>	0.04	28.0
MgO	0.10	60.0
CaO	0.02	60.0
Na <sub>2</sub> O	0.15	30.0
K <sub>2</sub> O	0.02	30.0
TiO <sub>2</sub>	0.02	10.0
P <sub>2</sub> O <sub>5</sub>	0.05	50.0
MnO	0.01	15.0
LOI (925°C)	0.01	100.0

Under normal circumstances of staffing and instrument maintenance, 250 samples per analyst month can be analyzed with this method.

## Apparatus

- Philips PW1606 simultaneous X-ray spectrometer
- Pt-Au alloy crucibles and molds (Taggart and Wahlberg, 1980a)
- Fluxer (Taggart and Wahlberg, 1980b)
- Two muffle furnaces with rocker attachments
- Hot plate and muffle furnace

## Reagents

The samples are digested in Johnson Matthey Spectroflux 100 or equivalent brand (lithium tetraborate). The flux is ordered in homogenized 200 Kg batches. The minus 60-mesh flux is dried for 2 days at 300°C and kept in sealed Mason jars. After drying a loss-on-fusion is performed for each lot of flux from the manufacturer so that an appropriate amount of flux can be weighed out to yield 8.0000 g of lithium tetraborate after fusion. The charges of flux are weighed by a Zymark robot to  $\pm .0035\text{g}$  ( $\pm 0.04\%$  precision). The platinum ware is cleaned in 50 percent reagent grade HCl, rinsed in deionized water and dried at 140°C. The LiBr used as a nonwetting agent is prepared by neutralizing reagent grade concentrated HBr (48%) with  $\text{LiCO}_3$ . This solution is filtered, and diluted 1:1 with deionized water.

## Safety precautions

Fusions and ignitions of samples in a muffle furnace must be performed under a high-velocity canopy hood. Boiling of the HCl cleaning solution is performed in a chemical fume hood with a safety sash. Safety glasses and special nonflammable, nonasbestos, heat-resistant gloves must be worn when removing the fluxer from the muffle furnace. Glass discs are sharp on the rear edge and should be handled with care. Dust from the flux must not be inhaled, so pouring of the powdered flux must be done in a chemical hood. Preparation of the LiBr solution must be done by slowly adding  $\text{LiCO}_3$  to the HBr so the generation of  $\text{CO}_2$  does not cause the acid to spill over the edge of the beaker. See the *CHP* and *MSDS* for further information concerning first-aid treatment and disposal procedures for chemical products used in this method.

## Procedure

A 0.8000 g portion of minus 80-mesh sample is ignited in a tared 95 percent Pt/ 5 percent Au crucible at 925°C for 40 min. The weight loss is reported as percent loss on ignition (LOI). A charge of lithium tetraborate that will contribute 8.0000 g after fusion is added to the sample and the powders are thoroughly mixed. The combined weights of the sample and the flux are calculated to present an “infinitely thick” sample disc to the instrument. A 250 :L aliquot of the 1:1 LiBr solution is added as a nonwetting agent. Seven crucibles containing samples and seven empty molds are loaded onto the automatic fluxer and the loaded apparatus placed in the muffle furnace at 1,120°C. The samples are allowed to come to temperature for 10 min, and are then homogenized in the furnace with an electric motor mechanism that rocks the crucibles side to side 54 times per minute for 35 min. The fluxer is removed from the furnace, the molten mixtures are poured from the seven crucibles into their respective molds, and cooled to near room temperature. An essential feature of this method is the mold design (Taggart and Wahlberg, 1980a). Samples with high concentrations of Cu, Cr, Ni, Fe, Mn and high organic content require various special sample preparation techniques, and in some cases, cannot be prepared at all. Samples with arsenic or lead with concentrations in excess of 2,000 ppm, or with combined As/Pb concentration in excess of 3,000 ppm, cannot be prepared because of risk of damage of the Pt/ Au crucibles. Using the wavelength dispersive X-ray spectrometer, the major element concentrations are determined by comparing the intensities obtained from standards with those obtained from the sample. (Taggart and others, 1981; Taggart and others, 1987).

The following instrumental conditions are for the Phillips PW1606 spectrometer:

Tube..... Rhodium, end window  
 Power ..... 35Kv and 60ma  
 Time..... 100 s  
 Atmosphere ..... Vacuum

See table 1 for the parameters for each of the channels and detectors in the instrument. Sixty-two well characterized and available international standards are used for the calibration. The 15 standards used for the recalibration program were prepared and run in triplicate, while the remaining 47 standards were prepared and run in duplicate. Additionally, four spiked bromine standards, six spiked sulfur standards, 10 blanks with LiBr, and five blanks without LiBr were used in the calibration.

**Table 1.—Operating conditions for determination of elements by WDXRF**

[PX-1=Tungsten Carbide layered, TLAP=thallium hydrogen phtalate, PET=pentaerythritol tetrakis (hydroxymethyl) methane, InSb=Indium Antimonide, Ge=Germanium 111, LiF 200=lithium fluoride (200 lattice orientation), P-10 gas=90 percent argon + 10 percent methane]

<i>Element</i>	<i>Line</i>	<i>Crystal</i>	<i>Detector-gas</i>	<i>Window</i>
Na	K $\alpha$	PX-1	Flow, P-10	2 $\mu$ m, polypropylene
Mg	K $\alpha$	TLAP	Flow, P-10	2 $\mu$ m, polypropylene
Al	K $\alpha$	PET	sealed neon	25 $\mu$ m, beryllium
Si	K $\alpha$	InSb	sealed neon	25 $\mu$ m, beryllium
P	K $\alpha$	Ge	sealed neon	50 $\mu$ m, beryllium
K	K $\alpha$	LiF 200	sealed krypton	100 $\mu$ m, beryllium

Ca	K $\alpha$	LIF 200	sealed krypton	100 $\mu$ m, beryllium
Ti	K $\alpha$	LIF 200	sealed krypton	100 $\mu$ m, beryllium
Mn	K $\alpha$	LIF 200	sealed krypton	100 $\mu$ m, beryllium
Fe	K $\alpha$	LIF 200	sealed krypton	100 $\mu$ m, beryllium

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The Philips PW1606 spectrometer is recalibrated every 2 weeks. The computerized recalibration is performed using discs from the original calibration and are used to set the slope of the calibration curve. The standards used include: AGV-1, DTS-1, BHVO-1, STM-1, NOD-P-1, MRG-1, BX-N, FK-N, GS-N, MICA-FE, NIM-D, NIM-P, GSR-4, GFS-401, and NBS-120C. Six blanks, prepared from the current batch of flux and LiBr are used for recalibration of the curve's intercept. This allows the original calibration to be maintained while compensating for minor changes in the reagents, P-10 gas, or instrument parameters due to equipment maintenance. Following a recalibration, a new disc of the quality control check standard TB-1 is prepared and counted to verify the calibration.

Long-term instrument drift is corrected using drift monitor analyses. Monitor intensity values obtained during the analyses are compared with monitor intensity values from the original calibration. Corrections are calculated by the spectrometer's software. Long-term drift monitoring cannot correct for short-term effects or significant changes in the operating parameters.

In order to keep track of instrumental short-term drift, at least every twelfth disc is an instrument check standard: AGV-1, DTS-1, BCS 381, or BX-N. These standards represent the average, high and low for the 10 analyzed elements. If the analyzed disc exceeds three times the standard deviation of the counting statistics, analysis is halted and the instrument is checked using other discs. If the disc is corrupt, it is removed and another is made. If the instrument shows signs of drift, then a recalibration is performed, as previously described.

In addition to the instrument standards, a sample preparation check standard, TB-1 disc is prepared for every 20 samples produced and analyzed along with the samples. If this disc shows a deviation of 3 standard deviations or more, and the instrument standards show no deviation, then another sample of TB-1 is prepared. If it again shows deviation, then sample preparation is halted and the problem is located. If both the sample preparation standard and the instrument standard exceed control limits, then the instrument recalibration is performed.

## Assignment of uncertainty

The WDXRF method for major element analysis is unique among analytical method packages in that it takes advantage of the summation of the determined elements. This summation acts as a measure of quality control. If an analysis includes the principal elements in a sample, then the total of their determinations should approach 100 percent. This check is the main reason that a LOI was initially incorporated in the package. If an analysis yields a total major element oxide determination of less than 97 percent or greater than 101 percent, then it is automatically repeated. Precision in the WDXRF method depends on the stability of the instrument, the orientation of the sample disc as it is presented to the instrument, and the homogeneity of the sample preparation. Table 2 is the analytical results of 10 major elements for selected reference materials, duplicate samples, and method blanks. Some *pv* data are calculated from element-to-stoichiometric oxide conversion factors (see appendix A, table A1)

**Table 2.—Analytical performance summary for elements (percent) by WDXRF**

[A=Bureau of Analysed Samples Ltd., 1973; B=National Institute of Standards and Technology, 1992; 88GLA2= Gladney and Roelandts, 1988, USGS=USGS certificate of analysis, GPT 2,3,5,6,7,&8 =Proficiency Tests organized by the International Association of Geoanalysts (IAG), remaining *pv* from Potts and others, 1992; LOI=loss on ignition; calc=value calculated as  $\{H_2O^+ + H_2O^- + CO_2(+C) - (FeO \cdot 0.1113)\}$ ]. See page ix of the introduction to this Methods Manual for an explanation of the abbreviations used in the analytical performance summary tables.

<i>Reference</i>	<i>Description</i>	<i>n</i>	<i>Mean</i>	<i>s</i>	<i>pv</i>	<i>% RSD</i>	<i>% R</i>	
<b>SiO<sub>2</sub></b>								
BCS 381	slag	27	8.83	0.04	8.78	A	0.5	101
BCR-2	basalt	20	54.17	0.13	54.1	USGS	0.23	100
JA-2	andesite	30	56.5	0.2	56.18		0.3	101
OU-1	volcanic tuff	2	57.93	0.06	58.247	GPT2	0.10	100
AGV-2	andesite	20	59.21	0.18	59.3	USGS	0.30	100
AMH-1	andesite	3	59.84	0.18	60.337	GPT5	0.30	99
GSD-6	stream sediment	30	60.67	0.07	61.23		0.1	99
OU-4	microdiorite	3	63.22	0.09	63.34	GPT8	0.14	100
SRM 2711	soil	30	63.67	0.06	65.12	B <i>cv</i>	0.1	98
GBPG-1	garn-biot-plag-gneiss	3	65.14	0.16	65.12	GPT7	0.25	100
RGM-1	rhyolite	20	72.89	0.19	73.45	88GLA2	0.27	99
YG-1	granite	3	73.22	0.14	73.363	GPT3	0.19	100
OU-3	microgranite	3	74.17	0.11	74.090	GPT6	0.15	100
GSD-12	stream sediment	30	76.5	0.1	77.29		0.2	99
<b>Al<sub>2</sub>O<sub>3</sub></b>								
BCS 381	slag	27	0.71	0.01	0.67	A	1	106
GSD-12	stream sediment	30	9.30	0.03	9.30		0.3	100
OU-3	microgranite	3	11.13	0.04	11.108	GPT6	0.37	100
SRM 2711	soil	30	12.18	0.03	12.34	B <i>cv</i>	0.2	99
YG-1	granite	3	13.03	0.03	13.056	GPT3	0.20	100
BCR-2	basalt	20	13.48	0.02	13.5	USGS	0.17	100
RGM-1	rhyolite	20	13.68	0.04	13.72	88GLA2		100
GSD-6	stream sediment	30	14.14	0.04	14.16		0.3	100
OU-4	microdiorite	3	14.84	0.01	14.83	GPT8	0.08	100
OU-1	volcanic tuff	2	15.11	0.01	15.136	GPT2	0.09	100
JA-2	andesite	30	15.78	0.05	15.32		0.3	103
GBPG-1	garn-biot-plag-gneiss	3	15.89	0.04	15.75	GPT7	0.23	101
AGV-2	andesite	20	16.88	0.06	16.91		0.21	100
AMH-1	andesite	3	17.47	0.01	17.530	GPT5	0.08	100
<b>Fe<sub>2</sub>O<sub>3</sub></b>								
RGM-1	rhyolite	20	1.84	0.02	1.86	88GLA2		99
YG-1	granite	3	2.77	0.02	2.806	GPT3	0.77	99
OU-2	microgranite	3	3.85	0.02	3.834	GPT6	0.65	100
SRM 2711	soil	30	4.12	0.01	4.13	B <i>cv</i>	0.2	100
GSD-12	stream sediment	30	4.86	0.01	4.88		0.3	100
OU-4	microdiorite	3	5.78	0.01	5.82	GPT8	0.09	99
GSD-6	stream sediment	30	5.88	0.02	5.88		0.3	100
GBPG-1	garn-biot-plag-gneiss	3	5.95	0.01	5.92	GPT7	0.17	101
AMH-1	andesite	3	6.02	0.02	6.098	GPT5	0.31	99
JA-2	andesite	30	6.17	0.02	6.95		0.3	89
AGV-2	andesite	20	6.65	0.03	6.69	USGS	0.46	99
OU-1	volcanic tuff	2	8.92	0.01	8.987	GPT2	0.09	99
BCR-2	basalt	20	13.74	0.05	13.8	USGS	0.34	100
BCS 381	slag	24*	18.12	0.08	19.02	A	0.4	95

\*Missing Fe<sub>2</sub>O<sub>3</sub> values rejected due to Fe contamination

**Table 2.—Analytical performance summary for elements (percent) by WDXRF—  
Continued**

<i>Reference</i>	<i>Description</i>	<i>n</i>	<i>Mean</i>	<i>s</i>	<i>pv</i>		<i>% RSD</i>	<i>% R</i>
<b>MgO</b>								
OU-3	microgranite	3	<0.10		--	GPT6	--	--
YG-1	granite	3	0.12	0.00	0.126	GPT3	2.91	96
RGM-1	rhyolite	20	0.26	0.02	0.275	88GLA2	0.07	93
GSD-12	stream sediment	30	0.44	0.01	0.47		2	94
BCS 381	slag	27	0.82	0.01	1.03	A	1	80
SRM 2711	soil	30	1.72	0.01	1.74	B <i>cv</i>	0.8	99
AGV-2	andesite	20	1.78	0.02	1.79	USGS	1.27	99
GBPG-1	garn-biot-plag-gneiss	3	2.59	0.01	2.57	GPT7	0.46	101
OU-4	microdiorite	3	2.29	0.02	2.30	GPT8	0.80	99
GSD-6	stream sediment	30	2.98	0.01	3.00		0.5	99
AMH-1	andesite	3	3.14	0.02	3.156	GPT5	0.69	100
BCR-2	basalt	20	3.60	0.02	3.59	USGS	0.54	100
OU-1	volcanic tuff	2	4.68	0.02	4.727	GPT2	0.53	99
JA-2	andesite	30	7.28	0.03	7.68		0.4	95
<b>CaO</b>								
OU-3	microgranite	3	0.21	0.00	0.2	GPT6	0.27	106
YG-1	granite	3	1.09	0.00	1.096	GPT3	0.05	100
GSD-12	stream sediment	30	1.16	0.01	1.16		0.8	100
RGM-1	rhyolite	20	1.19	0.00	1.15	88GLA2	0.00	103
GBPG-1	garn-biot-plag-gneiss	3	2.84	0.00	2.90	GPT7	0.05	98
GSD-6	stream sediment	30	3.91	0.01	3.87		0.3	101
SRM 2711	soil	30	3.97	0.01	4.03	B <i>cv</i>	0.3	98
OU-4	microdiorite	3	4.47	0.00	4.48	GPT8	0.08	100
AGV-2	andesite	20	5.16	0.01	5.20	USGS	0.25	99
AMH-1	andesite	3	5.99	0.01	6.064	GPT5	0.19	99
JA-2	andesite	30	6.20	0.02	6.48		0.3	96
OU-1	volcanic tuff	2	6.46	0.00	6.488	GPT2	0.04	100
BCR-2	basalt	20	7.09	0.01	7.12	USGS	0.18	100
BCS 381	slag	27	48.1	0.1	49.0	A	0.2	98
<b>Na<sub>2</sub>O</b>								
BCS 381	slag	27	0.21	0.02	---		10	---
GSD-12	stream sediment	30	0.33	0.01	0.44		3	75
SRM 2711	soil	30	1.47	0.01	1.54	B <i>cv</i>	0.9	95
GSD-6	stream sediment	30	2.18	0.02	2.31		1	94
OU-1	volcanic tuff	2	2.46	0.01	2.463	GPT2	0.43	100
JA-2	andesite	30	3.08	0.02	3.08		0.6	100
BCR-2	basalt	20	3.08	0.05	3.08	USGS	1.48	98
GBPG-1	garn-biot-plag-gneiss	3	3.40	0.02	3.57	GPT7	0.60	95
OU-4	microdioite	3	3.53	0.01	3.61	GPT8	0.30	98
YG-1	granite	3	3.57	0.04	3.603	GPT3	1.06	99
OU-3	microgranite	3	3.57	0.03	3.678	GPT6	0.75	97
RGM-1	rhyolite	20	3.97	0.03	4.07	88GLA2	0.01	98
AGV-2	andesite	20	4.08	0.04	4.19	USGS	0.91	97
AMH-1	andesite	3	4.10	0.04	4.208	GPT5	0.97	97



**Table 2.—Analytical performance summary for elements (percent) by WDXRF—  
Continued**

<i>Reference</i>	<i>Description</i>	<i>n</i>	<i>Mean</i>	<i>s</i>	<i>pv</i>		<i>% RSD</i>	<i>% R</i>
<b>K<sub>2</sub>O</b>								
BCS 381	slag	27	0.039	0.003	---		8	---
OU-1	volcanic tuff	2	0.22	0.00	0.22	GPT2	1.29	102
AMH-1	andesite	3	1.22	0.01	1.227	GPT5	1.227	100
BCR-2	basalt	20	1.77	0.01	1.79	USGS	0.45	99
JA-2	andesite	30	1.83	0.01	1.80		0.5	101
GBPG-1	garn-biot-plag-gneiss	3	2.23	0.00	2.26	GPT-7	0.12	99
GSD-6	stream sediment	30	2.43	0.01	2.44		0.4	99
OU-4	microdiorite	3	2.70	0.01	2.70	GPT8	0.34	100
AGV-2	andesite	20	2.87	0.02	2.88	USGS	0.54	100
GSD-12	stream sediment	30	2.92	0.01	2.91		0.3	100
SRM 2711	soil	30	2.93	0.01	2.95	B <i>cv</i>	0.3	99
RGM-1	rhyolite	20	4.31	0.02	4.30	88GLA2	0.01	100
OU-3	microgranite	3	4.58	0.01	4.55	GPT6	0.29	101
YG-1	granite	3	5.09	0.01	5.0599	GPT3	0.29	101
<b>TiO<sub>2</sub></b>								
YG-1	granite	3	0.21	0.00	0.2012	GPT3	1.27	104
OU-3	microgranite	3	0.23	0.00	0.224	GPT6	0.89	104
GSD-12	stream sediment	30	0.26	0.003	0.25		1	104
RGM-1	rhyolite	20	0.28	0.00	0.267	99GLA2	0.01	104
BCS 381	slag	27	0.33	0.004	0.35	A	1	94
OU-1	volcanic tuff	2	0.45	0.00	0.44	GPT2	0.79	101
SRM 2711	soil	30	0.51	0.004	0.51	B <i>cv</i>	0.8	100
JA-2	andesite	30	0.67	0.006	0.67		0.9	100
GBPG-1	garn-biot-plag-gneiss	3	0.69	0.00	0.699	GPT7	0.38	99
OU-4	microdiorite	3	0.77	0.00	0.77	GPT8	0.61	100
GSD-6	stream sediment	30	0.77	0.004	0.78		0.5	98
AMH-1	andesite	3	0.85	0.00	0.8457	GPT5	0.47	101
AGV-2	andesite	20	1.04	0.00	1.05	USGS	0.42	99
BCR-2	basalt	20	2.27	0.01	2.26	USGS	0.35	100
<b>P<sub>2</sub>O<sub>5</sub></b>								
GSD-12	stream sediment	30	0.085	0.004	0.055		5	155
JA-2	andesite	30	0.187	0.004	0.15		2	127
SRM 2711	soil	30	0.214	0.003	<b>0.197</b>	B <i>cv</i>	1	109
GSD-6	stream sediment	30	0.260	0.004	<b>0.23</b>		2	113
BCS 381	slag	27	15.4	0.04	15.7	A	0.3	98
<b>MnO</b>								
SRM 2711	soil	30	0.083	0.001	<b>0.0823</b>	B <i>cv</i>	1	101
JA-2	andesite	30	0.107	0.001	0.11		0.9	100
GSD-6	stream sediment	30	0.126	0.001	<b>0.13</b>		0.8	100
GSD-12	stream sediment	30	0.184	0.001	<b>0.18</b>		0.5	100
BCS 381	slag	27	3.01	0.01	3.16	A	0.3	95
<b>LOI (925°C)</b>								
BCS 381	slag	27	0.24	0.07	---		29	---
JA-2	andesite	30	1.76	0.10	2.12	calc	5	83
GSD-12	stream sediment	30	2.72	0.02	2.50	?	0.8	109
GSD-6	stream sediment	30	5.70	0.06	5.83	calc	1	98
SRM 2711	soil	30	7.92	0.08	---		1	---

**Table 2.—Continued—Duplicate samples results**

<i>Duplicate samples</i>	<i>k</i>	<i>n</i>	<i>Mean</i>	<i>s</i>	<i>% RSD</i>	<i>Concentration range</i>	<i>No of &lt; (total)</i>	<i>No of &lt; (pairs)</i>
SiO <sub>2</sub>	56	2	60.25	0.07	0.1	30.89 to 96.13	0	0
Al <sub>2</sub> O <sub>3</sub>	56	2	14.81	0.02	0.1	0.79 to 27.29	0	0
Fe <sub>2</sub> O <sub>3</sub>	56	2	6.83	0.02	0.3	0.53 to 36.6	0	0
MgO	53	2	3.28	0.01	0.3	0.19 to 11.32	6	3
CaO	56	2	4.71	0.01	0.2	0.07 to 13.18	0	0
Na <sub>2</sub> O	53	2	2.98	0.01	0.3	0.37 to 5.97	6	3
K <sub>2</sub> O	56	2	2.43	0.01	0.4	0.08 to 9.37	0	0
TiO <sub>2</sub>	56	2	0.809	0.002	0.2	0.073 to 2.85	0	0
P <sub>2</sub> O <sub>5</sub>	56	2	0.217	0.002	0.9	0.053 to 1.05	0	0
MnO	53	2	0.1007	0.0004	0.4	0.01 to 0.307	4	2
LOI	54	2	2.91	0.03	1	0.10 to 21.82	4	2
Total	56	2	99.4	0.1	0.1	96.48 to 100.34	0	0

**Table 2.—Continued—Method blank results** 3s values are considered the lower limit of detection (LOD), and 5s values are considered the lower limit of determination (LLD)

<i>Method blank</i>	<i>n</i>	<i>Mean</i>	<i>s</i>	<i>3s</i>	<i>5s</i>
SiO <sub>2</sub>	60	-0.01	0.01	0.04	0.07
Al <sub>2</sub> O <sub>3</sub>	60	0.03	0.01	0.04	0.07
Fe <sub>2</sub> O <sub>3</sub>	60	0.003	0.004	0.01	0.02
MgO	60	-0.01	0.009	0.03	0.05
CaO	60	0.008	0.001	0.003	0.005
Na <sub>2</sub> O	60	-0.04	0.02	0.06	0.1
K <sub>2</sub> O	60	-0.01	0.002	0.006	0.01
TiO <sub>2</sub>	60	-0.02	0.001	0.003	0.005
P <sub>2</sub> O <sub>5</sub>	60	-0.02	0.002	0.006	0.01
MnO	60	-0.01	0.0004	0.001	0.002

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