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# Soil Reference Material Data Sheets: BGS110 to BGS119

Environmental Change, Adaptation and Resilience

Open Report OR/20/014





BRITISH GEOLOGICAL SURVEY

ENVIRONMENTAL CHANGE, ADAPTATION AND RESILIENCE

OPEN REPORT OR/20/014

# Soil Reference Material Data Sheets: BGS110 to BGS119

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# 1 Introduction

The British Geological Survey (BGS) has produced a suite of 10 new soil Reference Materials, BGS110 to BGS119. They are intended for use as quality control samples for the determination of total elemental concentrations in soils.

The Reference Materials contain a wide range of concentrations to cater for different analytical needs, interests and industries, e.g. agriculture, geochemical exploration, contaminated land.

Data sheets for each of these materials are available on the BGS website

<https://www.bgs.ac.uk/sciencefacilities/laboratories/geochemistry/igf/Services/referenceMaterials.html>.

For **purchasing and more information** on Reference Materials, please contact

[inorganicgeochemistry@bgs.ac.uk](mailto:inorganicgeochemistry@bgs.ac.uk)

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## 2 Data Sheets

The following pages contain the Reference Material Data Sheets for BGS110 to BGS119.

The Data Sheet for each material consists of four pages detailing analytical methods used, Reference Values and Information Values.



## Reference Soil BGS110

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### **Silty sand soil overlying till**

#### Sample information

Silty sand soil overlying till, with Lower Palaeozoic and granitic clasts, from Vallemount, Co. Wicklow, Ireland.

#### Sample handling

The bulk sample was dried at 30°C, disaggregated, and sieved to <2 mm, giving a final mass of 33 kg.

This was batch milled in 500 mL agate milling vessels with agate balls. A portion of the milled sample was checked by sieving to ensure it met the quality threshold (>99% at <53 µm and >95% at <32 µm).

The milled material was then combined and homogenised by rotation in a 125 L barrel.

A riffle splitter was repeatedly used to separate the homogenised material to achieve 8 portions of approximately 4.13 kg, each of which was divided using a rotary splitter into interim portions of 0.52 kg.

To create the final reference material portions, sets of five of the 0.52 kg bags were re-combined and subsequently twice divided using a rotary splitter. The final portions of at least 40 g were stored in labelled plastic bottles.

#### Homogeneity testing

Sample homogeneity testing was carried out at the interim portion stage of sample handling. Sixteen bags were randomly selected for homogeneity testing. From each randomly selected bag, three subsamples were used to make pressed powder pellets for XRF analysis. Samples were each analysed in a single analytical run, in a fully randomised sequence.

Statistical analysis (mean, standard deviation and relative standard deviation (RSD)) was undertaken for each quantifiable analyte. Data were also assessed for outliers using Grubb's Test (Lister, 1982), at >3 standard deviations of the mean. No data were excluded on the criteria of outliers.

Homogeneity statistics were calculated for analytes where the mean was greater than the detection limit (DL). Where standard deviation was  $\leq$ DL, the homogeneity was deemed acceptable. Where standard deviation >DL, and the RSD  $\leq$ 5%, the homogeneity was deemed acceptable; for the small number of analytes which did not pass this test, analysis of variance (ANOVA) was used. Where the ANOVA F-statistic was below the critical value, the variation between bags was not significantly greater ( $P \leq 0.05$ ) than the variation within bags, thus the homogeneity was deemed acceptable. No tested data failed the homogeneity testing criteria.

#### Intended use

This reference material is intended to be used as a quality control sample for the determination of total elemental concentrations in soils.

#### Storage

This material should be stored in a cool, dark, dry environment, with the lid securely sealed.

# Reference Soil BGS110

## Instructions for use

The material should be well mixed, by shaking multiple times with the lid still on, to ensure that subsequent sub-sampling is representative.

## Safety

Usual safety precautions apply for handling; material may contain elements or other substances at concentrations that are potentially harmful to health.

## Methods of data analysis for Reference Values

Participating laboratories were requested to undertake three independent analyses of the candidate reference material using their standard procedures. Reported data were collated, and methods intended to define total elemental concentrations were selected (see Table 1). Outlying data were identified where they exceeded two standard deviations from the mean, and excluded from calculation of the Reference Values. Data, for any analyte reported by a given laboratory, which consistently lay at the upper or lower extreme of the data populations for BGS110 to BGS119 were also excluded.

The Reference Value (the mean concentration) and the expanded uncertainty, which is calculated as twice the standard deviation of the analysis, are shown in Table 2.

Where fewer than 15 results were remaining after outlier exclusion, the data are provided as Information Values with only the mean concentration given in Table 3.

Analytical method codes are defined in Table 1 for the methods used to determine concentrations of elements listed in Tables 2 and 3.

*Table 1: Analytical methods used*

Method code	Preparation	Measurement
a	HF, HClO <sub>4</sub> , HCl digestion	AAS
d	HF, HClO <sub>4</sub> , HCl digestion	ICP-MS
f	HF, HClO <sub>4</sub> , HNO <sub>3</sub> digestion	ICP-MS
g	Sodium peroxide fusion	ICP-MS
h	H <sub>2</sub> O <sub>2</sub> ; HCl, HNO <sub>3</sub> , HF digestion	ICP-OES
i	Lithium borate fusion	ICP-OES
j	Lithium borate fusion bead	WD-XRFS
k	Pressed powder pellet	WD-XRFS
l	Pressed powder pellet	ED-XRFS





# Reference Soil BGS110

## Reference and Information Values

In Tables 2 and 3, the mean is reported to three significant figures. In Table 2 the expanded uncertainty is reported to the same order of magnitude as the mean for the analyte.

Table 2. Reference Values

Analyte	Mean concentration $\pm$ expanded uncertainty (mg kg <sup>-1</sup> )	Method code <sup>§</sup>	Number of data points
Al	39200 $\pm$ 6400	g, i, j, k, l	18
As	9.20 $\pm$ 0.94	d, f, h, k, l	22
Ba	194 $\pm$ 27	d, f, g, k, l	24
Ca	2310 $\pm$ 850	a, f, i, j, k, l	25
Co	7.53 $\pm$ 1.52	d, f, i, k	19
Cr	39.5 $\pm$ 15.8	d, f, g, i, k, l	22
Cs	4.76 $\pm$ 1.60	d, f, g, k, l	19
Cu	11.9 $\pm$ 1.9	d, f, h, k	21
Fe	15300 $\pm$ 2100	a, f, i, j, k, l	26
Ga	8.45 $\pm$ 1.17	d, g, k, l	18
K	11800 $\pm$ 1200	a, f, g, i, j, k, l	24
La	15.2 $\pm$ 4.0	d, g, k	15
Mg	2580 $\pm$ 2150	a, f, g, i, j, k, l	23
Mn	823 $\pm$ 170	a, f, i, j, k, l	27
Mo	1.21 $\pm$ 0.86	d, f, h, k, l	22
Na	8180 $\pm$ 1260	a, f, i, j, k, l	20
Nb	7.43 $\pm$ 1.53	d, g, k, l	18
Ni	20.1 $\pm$ 6.7	d, f, i, k, l	26
P	860 $\pm$ 251	f, j, k, l	18
Pb	37.4 $\pm$ 4.8	d, f, h, k, l	24
Rb	72.3 $\pm$ 7.7	d, f, g, i, k, l	25
Si	367000 $\pm$ 48000	g, i, j, k, l	18
Sr	70.3 $\pm$ 11.6	d, f, g, i, k, l	25
Th	4.60 $\pm$ 2.49	d, g, k	15
Ti	2190 $\pm$ 720	d, g, i, j, k, l	24
U	1.71 $\pm$ 0.69	d, f, g, k	15
V	45.8 $\pm$ 7.2	d, f, i, k, l	23
Y	10.6 $\pm$ 2.4	d, g, k, l	17
Zn	79.4 $\pm$ 14.1	d, f, h, k, l	23
Zr	141 $\pm$ 18	g, i, k, l	18

<sup>§</sup> Defined in Table 1.

# Reference Soil BGS110

Table 3. Information Values

Analyte	Mean concentration (mg kg <sup>-1</sup> )	Method code <sup>§</sup>	Number of data points
Ag	0.405	d, f, l	12
B	26.9	g	3
Be	2.79	d, f, g	9
Bi	0.192	d, f	6
Br	18.1	k, l	8
Cd	0.430	d, f, l	12
Ce	29.7	d, g, k	14
Cl	97.4	k, l	5
Dy	1.67	d, g	6
Er	0.964	d, g	6
Eu	0.556	d, g	6
Gd	2.10	d, g	6
Ge	2.80	d, k	6
Hf	3.56	d, g, k	11
Ho	0.321	d, g	6
I	5.78	k, l	6
In	0.029	d	3
Li	38.8	d, f, h	12
Lu	0.136	d, g	6
Nd	12.0	d, g, k	14
Pr	3.33	d, g	6
S	399	f	3
Sb	0.671	d, f, l	7
Sc	3.42	d, k	11
Se	0.644	d, f, k, l	13
Sm	2.59	d, g, k	8
Sn	2.03	d, g, k, l	14
Ta	1.70	d, g, k	11
Tb	0.286	d, g	6
Te	0.100	d	3
Tl	0.564	d, f	9
Tm	0.140	d, g	6
W	1.14	d, g, k	12
Yb	0.942	d, g	6

<sup>§</sup> Defined in Table 1.

## References

Lister, B. (1982). *Evaluation of Analytical Data: A Practical Guide for Geoanalysts*. Geostandards Newsletter, 6: 175-205.

## Reference Soil BGS111

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### Silty clay soil overlying serpentinite

#### Sample information

Silty clay soil overlying serpentinite (chromite-bearing, with minor Ni sulphides), from Cummer, Co. Wexford, Ireland.

#### Sample handling

The bulk sample was dried at 30°C, disaggregated, and sieved to <2 mm, giving a final mass of 43 kg.

This was batch milled in 500 mL agate milling vessels with agate balls. A portion of the milled sample was checked by sieving to ensure it met the quality threshold (>99% at <53 µm and >95% at <32 µm).

The milled material was then combined and homogenised by rotation in a 125 L barrel.

A riffle splitter was repeatedly used to separate the homogenised material to achieve 8 portions of approximately 5.38 kg, each of which was divided using a rotary splitter into interim portions of 0.67 kg.

To create the final reference material portions, sets of four of the 0.67 kg bags were re-combined and subsequently twice divided using a rotary splitter. The final portions of at least 38 g were stored in labelled plastic bottles.

#### Homogeneity testing

Sample homogeneity testing was carried out at the interim portion stage of sample handling. Twelve bags were randomly selected for homogeneity testing. From each randomly selected bag, two subsamples were used to make pressed powder pellets for XRF analysis. Samples were each analysed in a single analytical run, in a fully randomised sequence.

Statistical analysis (mean, standard deviation and relative standard deviation (RSD)) was undertaken for each quantifiable analyte. Data were also assessed for outliers using Grubb's Test (Lister, 1982), at >3 standard deviations of the mean. No data were excluded on the criteria of outliers.

Homogeneity statistics were calculated for analytes where the mean was greater than the detection limit (DL). Where standard deviation was  $\leq$ DL, the homogeneity was deemed acceptable. Where standard deviation >DL, and the RSD  $\leq$ 5%, the homogeneity was deemed acceptable; for the small number of analytes which did not pass this test, analysis of variance (ANOVA) was used. Where the ANOVA F-statistic was below the critical value, the variation between bags was not significantly greater ( $P \leq 0.05$ ) than the variation within bags, thus the homogeneity was deemed acceptable. No tested data failed the homogeneity testing criteria.

#### Intended use

This reference material is intended to be used as a quality control sample for the determination of total elemental concentrations in soils.

#### Storage

This material should be stored in a cool, dark, dry environment, with the lid securely sealed.

# Reference Soil BGS111

## Instructions for use

The material should be well mixed, by shaking multiple times with the lid still on, to ensure that subsequent sub-sampling is representative.

## Safety

Usual safety precautions apply for handling; material may contain elements or other substances at concentrations that are potentially harmful to health.

## Methods of data analysis for Reference Values

Participating laboratories were requested to undertake three independent analyses of the candidate reference material using their standard procedures. Reported data were collated, and methods intended to define total elemental concentrations were selected (see Table 1). Outlying data were identified where they exceeded two standard deviations from the mean, and excluded from calculation of the Reference Values. Data, for any analyte reported by a given laboratory, which consistently lay at the upper or lower extreme of the data populations for BGS110 to BGS119 were also excluded.

The Reference Value (the mean concentration) and the expanded uncertainty, which is calculated as twice the standard deviation of the analysis, are shown in Table 2.

Where fewer than 15 results were remaining after outlier exclusion, the data are provided as Information Values with only the mean concentration given in Table 3.

Analytical method codes are defined in Table 1 for the methods used to determine concentrations of elements listed in Tables 2 and 3.

*Table 1: Analytical methods used*

Method code	Preparation	Measurement
b	Ashed; HF, HClO <sub>4</sub> , HCl digestion	AAS
d	HF, HClO <sub>4</sub> , HCl digestion	ICP-MS
e	Ashed; HF, HClO <sub>4</sub> , HCl digestion	ICP-MS
f	HF, HClO <sub>4</sub> , HNO <sub>3</sub> digestion	ICP-MS
g	Sodium peroxide fusion	ICP-MS
h	H <sub>2</sub> O <sub>2</sub> ; HCl, HNO <sub>3</sub> , HF digestion	ICP-OES
i	Lithium borate fusion	ICP-OES
j	Lithium borate fusion bead	WD-XRFS
k	Pressed powder pellet	WD-XRFS
l	Pressed powder pellet	ED-XRFS



# Reference Soil BGS111

## Reference and Information Values

In Tables 2 and 3, the mean is reported to three significant figures. In Table 2 the expanded uncertainty is reported to the same order of magnitude as the mean for the analyte.

Table 2. Reference Values

Analyte	Mean concentration $\pm$ expanded uncertainty (mg kg <sup>-1</sup> )	Method code <sup>§</sup>	Number of data points
Al	69900 $\pm$ 8800	g, i, j, k, l	18
As	44.0 $\pm$ 5.2	e, f, h, k, l	26
Ba	306 $\pm$ 36	e, f, g, k, l	24
Ca	6080 $\pm$ 1110	f, i, j, k, l	24
Co	33.4 $\pm$ 5.5	d, f, i, k	20
Cr	1490 $\pm$ 210	g, i, k, l	18
Cs	5.09 $\pm$ 2.15	e, f, g, k, l	17
Cu	27.9 $\pm$ 3.3	d, f, h, k	21
Fe	53300 $\pm$ 6500	b, f, i, j, k, l	25
Ga	17.8 $\pm$ 2.4	e, g, k, l	18
K	16900 $\pm$ 1500	b, f, g, i, j, k, l	24
La	29.9 $\pm$ 3.1	e, g, k	15
Mg	19600 $\pm$ 8800	b, f, g, i, j, l	21
Mn	1850 $\pm$ 330	b, f, i, j, k, l	26
Mo	1.88 $\pm$ 1.25	e, f, h, k, l	22
Na	6130 $\pm$ 1760	b, f, i, j, k, l	21
Nb	13.7 $\pm$ 3.5	e, g, k, l	20
Nd	22.6 $\pm$ 5.0	e, g, k	15
Ni	227 $\pm$ 31	e, f, i, k, l	27
P	1780 $\pm$ 160	f, j, l	15
Pb	24.7 $\pm$ 3.1	d, f, h, k, l	24
Rb	117 $\pm$ 21	e, f, g, i, k, l	25
Si	235000 $\pm$ 33000	g, i, j, k, l	18
Sr	65.0 $\pm$ 11.0	e, f, g, i, k, l	26
Ti	5640 $\pm$ 1570	g, i, j, k, l	21
U	2.77 $\pm$ 0.98	e, f, g, k	15
V	140 $\pm$ 23	e, f, i, k, l	22
Y	20.7 $\pm$ 2.7	g, k, l	15
Zn	82.6 $\pm$ 14.7	e, f, h, k, l	24
Zr	185 $\pm$ 14.0	g, i, k, l	18

<sup>§</sup> Defined in Table 1.

# Reference Soil BGS111

Table 3. Information Values

Analyte	Mean concentration (mg kg <sup>-1</sup> )	Method code <sup>§</sup>	Number of data points
Ag	0.443	e, f, l	12
B	66.8	g	3
Be	2.01	e, f, g	8
Bi	0.318	d, f	6
Br	35.0	k, l	9
Cd	0.334	e, f	9
Ce	56.5	e, g, k	14
Cl	87.8	k, l	6
Dy	3.29	e, g	6
Er	1.87	e, g	6
Eu	0.997	e, g	6
Gd	4.09	e, g	6
Ge	4.83	e, k	9
Hf	4.57	e, g, k	12
Ho	0.617	e, g	6
I	13.8	k, l	6
In	0.069	e	3
Li	28.9	e, f, h	12
Lu	0.295	e, g	6
Pr	6.38	e, g	6
S	897	f	3
Sb	1.80	d, f, l	9
Sc	15.3	e, k	12
Se	1.01	e, f, g, k, l	14
Sm	4.36	e, g, k	10
Sn	3.03	e, g, k, l	14
Ta	1.77	e, g, k	11
Tb	0.560	e, g	6
Te	0.138	e	3
Th	7.30	e, g, k	14
Tl	0.492	e, f	9
Tm	0.264	e, g	6
W	2.68	e, g, k	14
Yb	1.87	e, g	6

<sup>§</sup> Defined in Table 1.

## References

Lister, B. (1982). *Evaluation of Analytical Data: A Practical Guide for Geoanalysts*. Geostandards Newsletter, 6: 175-205.

## Reference Soil BGS112

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### **Silty sand soil overlying granite**

#### Sample information

Silty sand soil overlying granitic till, from Brittas, Co. Wicklow, Ireland.

#### Sample handling

The bulk sample was dried at 30°C, disaggregated, and sieved to <2 mm, giving a final mass of 65 kg.

This was batch milled in 500 mL agate milling vessels with agate balls. A portion of the milled sample was checked by sieving to ensure it met the quality threshold (>99% at <53 µm and >95% at <32 µm).

The milled material was then combined and homogenised by rotation in a 290 L barrel.

A riffle splitter was repeatedly used to separate the homogenised material to achieve 16 portions of approximately 4.06 kg, each of which was divided using a rotary splitter into interim portions of 0.51 kg.

To create the final reference material portions, sets of six of the 0.51 kg bags were re-combined and subsequently twice divided using a rotary splitter. The final portions of at least 43 g were stored in labelled plastic bottles.

#### Homogeneity testing

Sample homogeneity testing was carried out at the interim portion stage of sample handling. Twelve bags were randomly selected for homogeneity testing. From each randomly selected bag, two subsamples were used to make pressed powder pellets for XRF analysis. Samples were each analysed in a single analytical run, in a fully randomised sequence.

Statistical analysis (mean, standard deviation and relative standard deviation (RSD)) was undertaken for each quantifiable analyte. Data were also assessed for outliers using Grubb's Test (Lister, 1982), at >3 standard deviations of the mean. No data were excluded on the criteria of outliers.

Homogeneity statistics were calculated for analytes where the mean was greater than the detection limit (DL). Where standard deviation was  $\leq$ DL, the homogeneity was deemed acceptable. Where standard deviation >DL, and the RSD  $\leq$ 5%, the homogeneity was deemed acceptable; for the small number of analytes which did not pass this test, analysis of variance (ANOVA) was used. Where the ANOVA F-statistic was below the critical value, the variation between bags was not significantly greater ( $P \leq 0.05$ ) than the variation within bags, thus the homogeneity was deemed acceptable. No tested data failed the homogeneity testing criteria.

#### Intended use

This reference material is intended to be used as a quality control sample for the determination of total elemental concentrations in soils.

#### Storage

This material should be stored in a cool, dark, dry environment, with the lid securely sealed.

# Reference Soil BGS112

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## Instructions for use

The material should be well mixed, by shaking multiple times with the lid still on, to ensure that subsequent sub-sampling is representative.

## Safety

Usual safety precautions apply for handling; material may contain elements or other substances at concentrations that are potentially harmful to health.

## Methods of data analysis for Reference Values

Participating laboratories were requested to undertake three independent analyses of the candidate reference material using their standard procedures. Reported data were collated, and methods intended to define total elemental concentrations were selected (see Table 1). Outlying data were identified where they exceeded two standard deviations from the mean, and excluded from calculation of the Reference Values. Data, for any analyte reported by a given laboratory, which consistently lay at the upper or lower extreme of the data populations for BGS110 to BGS119 were also excluded.

The Reference Value (the mean concentration) and the expanded uncertainty, which is calculated as twice the standard deviation of the analysis, are shown in Table 2.

Where fewer than 15 results were remaining after outlier exclusion, the data are provided as Information Values with only the mean concentration given in Table 3.

Analytical method codes are defined in Table 1 for the methods used to determine concentrations of elements listed in Tables 2 and 3.

*Table 1: Analytical methods used*

Method code	Preparation	Measurement
a	HF, HClO <sub>4</sub> , HCl digestion	AAS
d	HF, HClO <sub>4</sub> , HCl digestion	ICP-MS
f	HF, HClO <sub>4</sub> , HNO <sub>3</sub> digestion	ICP-MS
g	Sodium peroxide fusion	ICP-MS
h	H <sub>2</sub> O <sub>2</sub> ; HCl, HNO <sub>3</sub> , HF digestion	ICP-OES
i	Lithium borate fusion	ICP-OES
j	Lithium borate fusion bead	WD-XRFS
k	Pressed powder pellet	WD-XRFS
l	Pressed powder pellet	ED-XRFS



## Reference Soil BGS112

## Reference and Information Values

In Tables 2 and 3, the mean is reported to three significant figures. In Table 2 the expanded uncertainty is reported to the same order of magnitude as the mean for the analyte.

Table 2. Reference Values

Analyte	Mean concentration $\pm$ expanded uncertainty (mg kg <sup>-1</sup> )	Method code <sup>§</sup>	Number of data points
Al	79700 $\pm$ 13800	g, i, j, k, l	18
As	38.1 $\pm$ 12.2	d, f, h, k, l	25
Ba	402 $\pm$ 38	d, f, g, k, l	23
Ca	3050 $\pm$ 820	a, f, i, j, k, l	26
Co	8.71 $\pm$ 1.20	d, f, i, k	19
Cr	32.8 $\pm$ 13.3	d, f, g, i, k, l	23
Cs	9.36 $\pm$ 1.76	d, f, g, k, l	20
Cu	16.5 $\pm$ 2.3	d, f, h, k	21
Fe	23000 $\pm$ 3900	a, f, i, j, k, l	26
Ga	18.2 $\pm$ 2.8	d, g, k, l	18
K	24100 $\pm$ 2400	a, f, g, i, j, k, l	23
La	30.1 $\pm$ 3.2	d, g, k	15
Mg	4960 $\pm$ 3680	a, f, g, i, j, k, l	24
Mn	800 $\pm$ 147	a, f, i, j, k, l	25
Mo	0.999 $\pm$ 1.05	d, f, h, k, l	22
Na	12200 $\pm$ 1600	a, f, i, j, k, l	21
Nb	9.50 $\pm$ 1.41	d, g, k, l	18
Ni	14.0 $\pm$ 4.5	d, f, i, k, l	25
P	885 $\pm$ 221	f, j, k, l	18
Pb	29.4 $\pm$ 3.7	d, f, h, k, l	24
Rb	140 $\pm$ 16	d, f, g, k, l	24
Si	313000 $\pm$ 25000	g, i, j, k, l	18
Sn	8.79 $\pm$ 3.48	d, g, k, l	15
Sr	102 $\pm$ 15	d, f, g, i, k, l	27
Ti	3170 $\pm$ 890	d, g, i, j, k, l	24
U	2.84 $\pm$ 0.81	d, f, g, k	15
V	58.4 $\pm$ 11.0	d, f, i, k, l	22
Y	14.5 $\pm$ 3.2	d, g, k, l	18
Zn	83.2 $\pm$ 11.8	d, f, h, k, l	26
Zr	172 $\pm$ 31	g, i, k, l	18

<sup>§</sup> Defined in Table 1.

## Reference Soil BGS112

Table 3. Information Values

Analyte	Mean concentration (mg kg <sup>-1</sup> )	Method code <sup>§</sup>	Number of data points
Ag	0.331	d, f, l	12
B	15.2	g	3
Be	3.43	d, f, g	9
Bi	0.403	d, f	6
Br	29.7	k, l	9
Cd	0.218	f	6
Ce	52.9	d, g, k	14
Cl	111	k, l	6
Dy	2.62	d, g	6
Er	1.33	d, g	6
Eu	1.12	d, g	6
Gd	4.08	d, g	6
Ge	3.26	d, k	8
Hf	4.29	d, g, k	12
Ho	0.455	d, g	6
I	15.4	k, l	6
In	0.066	d	3
Li	117	d, f, h	12
Lu	0.183	d, g	6
Nd	25.4	d, g, k	14
Pr	7.24	d, g	6
S	343	f	3
Sb	0.231	d, f	6
Sc	5.61	d, k	11
Se	0.734	d, f, k, l	13
Sm	4.98	d, g, k	9
Ta	1.93	d, g, k	10
Tb	0.494	d, g	6
Te	0.131	d	3
Th	9.31	d, g, k	14
Tl	0.722	d, f	9
Tm	0.184	d, g	6
W	1.70	d, g, k, l	12
Yb	1.20	d, g	6

<sup>§</sup> Defined in Table 1.

## References

Lister, B. (1982). *Evaluation of Analytical Data: A Practical Guide for Geoanalysts*. Geostandards Newsletter, 6: 175-205.

## Reference Soil BGS113

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### **Silty clay soil overlying clastic metasediments**

#### Sample information

Silty clay soil overlying Silurian clastic metasediments, collected in an area of metalliferous mineralisation, (primarily Pb-Zn), from Castleblayney, Co. Monaghan, Ireland.

#### Sample handling

The bulk sample was dried at 30°C, disaggregated, and sieved to <2 mm, giving a final mass of 33 kg.

This was batch milled in 500 mL agate milling vessels with agate balls. A portion of the milled sample was checked by sieving to ensure it met the quality threshold (>99% at <53 µm and >95% at <32 µm).

The milled material was then combined and homogenised by rotation in a 125 L barrel.

A riffle splitter was repeatedly used to separate the homogenised material to achieve 8 portions of approximately 4.13 kg, each of which was divided using a rotary splitter into interim portions of 0.52 kg.

To create the final reference material portions, sets of five of the 0.52 kg bags were re-combined and subsequently twice divided using a rotary splitter. The final portions of at least 38 g were stored in labelled plastic bottles.

#### Homogeneity testing

Sample homogeneity testing was carried out at the interim portion stage of sample handling. Twelve bags were randomly selected for homogeneity testing. From each randomly selected bag, two subsamples were used to make pressed powder pellets for XRF analysis. Samples were each analysed in a single analytical run, in a fully randomised sequence.

Statistical analysis (mean, standard deviation and relative standard deviation (RSD)) was undertaken for each quantifiable analyte. Data were also assessed for outliers using Grubb's Test (Lister, 1982), at >3 standard deviations of the mean. No data were excluded on the criteria of outliers.

Homogeneity statistics were calculated for analytes where the mean was greater than the detection limit (DL). Where standard deviation was  $\leq$ DL, the homogeneity was deemed acceptable. Where standard deviation >DL, and the RSD  $\leq$ 5%, the homogeneity was deemed acceptable; for the small number of analytes which did not pass this test, analysis of variance (ANOVA) was used. Where the ANOVA F-statistic was below the critical value, the variation between bags was not significantly greater ( $P \leq 0.05$ ) than the variation within bags, thus the homogeneity was deemed acceptable. No tested data failed the homogeneity testing criteria.

#### Intended use

This reference material is intended to be used as a quality control sample for the determination of total elemental concentrations in soils.

#### Storage

This material should be stored in a cool, dark, dry environment, with the lid securely sealed.

# Reference Soil BGS113

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## Instructions for use

The material should be well mixed, by shaking multiple times with the lid still on, to ensure that subsequent sub-sampling is representative.

## Safety

Usual safety precautions apply for handling; material may contain elements or other substances at concentrations that are potentially harmful to health.

## Methods of data analysis for Reference Values

Participating laboratories were requested to undertake three independent analyses of the candidate reference material using their standard procedures. Reported data were collated, and methods intended to define total elemental concentrations were selected (see Table 1). Outlying data were identified where they exceeded two standard deviations from the mean, and excluded from calculation of the Reference Values. Data, for any analyte reported by a given laboratory, which consistently lay at the upper or lower extreme of the data populations for BGS110 to BGS119 were also excluded.

The Reference Value (the mean concentration) and the expanded uncertainty, which is calculated as twice the standard deviation of the analysis, are shown in Table 2.

Where fewer than 15 results were remaining after outlier exclusion, the data are provided as Information Values with only the mean concentration given in Table 3.

Analytical method codes are defined in Table 1 for the methods used to determine concentrations of elements listed in Tables 2 and 3.

*Table 1: Analytical methods used*

Method code	Preparation	Measurement
b	Ashed; HF, HClO <sub>4</sub> , HCl digestion	AAS
e	Ashed; HF, HClO <sub>4</sub> , HCl digestion	ICP-MS
f	HF, HClO <sub>4</sub> , HNO <sub>3</sub> digestion	ICP-MS
g	Sodium peroxide fusion	ICP-MS
h	H <sub>2</sub> O <sub>2</sub> ; HCl, HNO <sub>3</sub> , HF digestion	ICP-OES
i	Lithium borate fusion	ICP-OES
j	Lithium borate fusion bead	WD-XRFS
k	Pressed powder pellet	WD-XRFS
l	Pressed powder pellet	ED-XRFS

# Reference Soil BGS113

## Reference and Information Values

In Tables 2 and 3, the mean is reported to three significant figures. In Table 2 the expanded uncertainty is reported to the same order of magnitude as the mean for the analyte.

Table 2. Reference Values

Analyte	Mean concentration $\pm$ expanded uncertainty (mg kg <sup>-1</sup> )	Method code <sup>§</sup>	Number of data points
Al	80600 $\pm$ 9600	g, i, j, k, l	18
As	40.9 $\pm$ 3.8	e, f, h, k, l	23
Ba	537 $\pm$ 61	e, f, g, k, l	24
Ca	4360 $\pm$ 690	f, i, j, k, l	24
Cd	64.2 $\pm$ 11.2	e, f, k, l	21
Ce	92.5 $\pm$ 15.0	e, g, k	15
Co	46.0 $\pm$ 6.7	e, f, i, k	21
Cr	130 $\pm$ 21	e, f, g, i, k, l	22
Cs	3.51 $\pm$ 2.56	e, f, g, k, l	19
Cu	91.6 $\pm$ 9.8	e, f, h, k	21
Fe	46100 $\pm$ 6600	b, f, i, j, k, l	26
Ga	19.4 $\pm$ 3.1	e, g, k, l	18
K	17700 $\pm$ 1200	b, f, g, i, j, k, l	24
Mg	9350 $\pm$ 4690	b, f, g, i, j, l	21
Mn	1390 $\pm$ 260	b, f, g, i, j, k, l	27
Mo	3.64 $\pm$ 0.88	e, f, g, h, k	21
Na	7730 $\pm$ 2320	b, f, i, j, k, l	21
Nb	11.8 $\pm$ 3.7	e, g, k, l	19
Nd	63.7 $\pm$ 5.1	e, g, k	15
Ni	147 $\pm$ 20	e, f, i, k, l	25
P	1290 $\pm$ 140	f, j, k, l	18
Pb	481 $\pm$ 102	e, f, h, k, l	27
Rb	84.5 $\pm$ 16.1	e, f, g, i, k, l	25
Se	3.10 $\pm$ 1.61	e, f, k, l	18
Si	240000 $\pm$ 29000	g, i, j, k, l	18
Sr	95.0 $\pm$ 15.1	e, f, g, i, k, l	27
Th	11.9 $\pm$ 4.7	e, g, k	15
Ti	4160 $\pm$ 1330	e, g, i, j, k, l	24
U	4.85 $\pm$ 1.13	e, f, g, k	15
V	139 $\pm$ 31	e, f, i, k, l	23
Y	49.6 $\pm$ 7.9	e, g, k, l	18
Zn	2410 $\pm$ 290	e, f, k, l	24
Zr	180 $\pm$ 16	g, i, k, l	18

<sup>§</sup> Defined in Table 1.

# Reference Soil BGS113

Table 3. Information Values

Analyte	Mean concentration (mg kg <sup>-1</sup> )	Method code <sup>§</sup>	Number of data points
Ag	1.50	e, f, l	12
B	30.3	g	3
Be	1.96	e, f, g	9
Bi	0.312	e, f	6
Br	23.4	k, l	9
Cl	60.8	k, l	6
Dy	8.65	e, g	6
Er	4.53	e, g	6
Eu	2.99	e, g	6
Gd	12.3	e, g	6
Ge	4.48	e, k	9
Hf	5.91	e, g, k	12
Ho	1.62	e, g	6
I	13.2	k, l	6
In	0.084	e	3
La	60.7	e, g, k	14
Li	47.5	e, f, h	12
Lu	0.624	e, g	6
Pr	15.1	e, g	6
S	902	f	3
Sb	1.05	e, f, l	9
Sc	20.0	e, k	12
Sm	12.5	e, g, k	11
Sn	1.87	e, g, k, l	11
Ta	2.56	e, g, k	12
Tb	1.57	e, g	6
Te	0.245	e	3
Tl	1.09	e, f	9
Tm	0.612	e, g	6
W	2.66	e, g, k, l	11
Yb	3.85	e, g, k	8

<sup>§</sup> Defined in Table 1.

## References

Lister, B. (1982). *Evaluation of Analytical Data: A Practical Guide for Geoanalysts*. Geostandards Newsletter, 6: 175-205.



## Reference Soil BGS114

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### **Silty soil overlying clastic metasediments**

#### Sample information

Silty soil overlying Silurian clastic metasediments, from Carrickmacross, Co. Monaghan, Ireland.

#### Sample handling

The bulk sample was dried at 30°C, disaggregated, and sieved to <2 mm, giving a final mass of 59 kg.

This was batch milled in 500 mL agate milling vessels with agate balls. A portion of the milled sample was checked by sieving to ensure it met the quality threshold (>99% at <53 µm and >95% at <32 µm).

The milled material was then combined and homogenised by rotation in a 290 L barrel.

A riffle splitter was repeatedly used to separate the homogenised material to achieve 16 portions of approximately 3.69 kg, each of which was divided using a rotary splitter into interim portions of 0.46 kg.

To create the final reference material portions, sets of six of the 0.46 kg bags were re-combined and subsequently twice divided using a rotary splitter. The final portions of at least 41 g were stored in labelled plastic bottles.

#### Homogeneity testing

Sample homogeneity testing was carried out at the interim portion stage of sample handling. Twelve bags were randomly selected for homogeneity testing. From each randomly selected bag, two subsamples were used to make pressed powder pellets for XRF analysis. Samples were each analysed in a single analytical run, in a fully randomised sequence.

Statistical analysis (mean, standard deviation and relative standard deviation (RSD)) was undertaken for each quantifiable analyte. Data were also assessed for outliers using Grubb's Test (Lister, 1982), at >3 standard deviations of the mean. No data were excluded on the criteria of outliers.

Homogeneity statistics were calculated for analytes where the mean was greater than the detection limit (DL). Where standard deviation was  $\leq$ DL, the homogeneity was deemed acceptable. Where standard deviation >DL, and the RSD  $\leq$ 5%, the homogeneity was deemed acceptable; for the small number of analytes which did not pass this test, analysis of variance (ANOVA) was used. Where the ANOVA F-statistic was below the critical value, the variation between bags was not significantly greater ( $P \leq 0.05$ ) than the variation within bags, thus the homogeneity was deemed acceptable. No tested data failed the homogeneity testing criteria.

#### Intended use

This reference material is intended to be used as a quality control sample for the determination of total elemental concentrations in soils.

#### Storage

This material should be stored in a cool, dark, dry environment, with the lid securely sealed.

# Reference Soil BGS114

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## Instructions for use

The material should be well mixed, by shaking multiple times with the lid still on, to ensure that subsequent sub-sampling is representative.

## Safety

Usual safety precautions apply for handling; material may contain elements or other substances at concentrations that are potentially harmful to health.

## Methods of data analysis for Reference Values

Participating laboratories were requested to undertake three independent analyses of the candidate reference material using their standard procedures. Reported data were collated, and methods intended to define total elemental concentrations were selected (see Table 1). Outlying data were identified where they exceeded two standard deviations from the mean, and excluded from calculation of the Reference Values. Data, for any analyte reported by a given laboratory, which consistently lay at the upper or lower extreme of the data populations for BGS110 to BGS119 were also excluded.

The Reference Value (the mean concentration) and the expanded uncertainty, which is calculated as twice the standard deviation of the analysis, are shown in Table 2.

Where fewer than 15 results were remaining after outlier exclusion, the data are provided as Information Values with only the mean concentration given in Table 3.

Analytical method codes are defined in Table 1 for the methods used to determine concentrations of elements listed in Tables 2 and 3.

*Table 1: Analytical methods used*

Method code	Preparation	Measurement
a	HF, HClO <sub>4</sub> , HCl digestion	AAS
d	HF, HClO <sub>4</sub> , HCl digestion	ICP-MS
f	HF, HClO <sub>4</sub> , HNO <sub>3</sub> digestion	ICP-MS
g	Sodium peroxide fusion	ICP-MS
h	H <sub>2</sub> O <sub>2</sub> ; HCl, HNO <sub>3</sub> , HF digestion	ICP-OES
i	Lithium borate fusion	ICP-OES
j	Lithium borate fusion bead	WD-XRFS
k	Pressed powder pellet	WD-XRFS
l	Pressed powder pellet	ED-XRFS



# Reference Soil BGS114

## Reference and Information Values

In Tables 2 and 3, the mean is reported to three significant figures. In Table 2 the expanded uncertainty is reported to the same order of magnitude as the mean for the analyte.

Table 2. Reference Values

Analyte	Mean concentration $\pm$ expanded uncertainty (mg kg <sup>-1</sup> )	Method code <sup>§</sup>	Number of data points
Al	78500 $\pm$ 16100	g, i, j, k, l	18
As	11.8 $\pm$ 3.3	d, f, k, l	21
Ba	427 $\pm$ 50	d, f, g, k, l	24
Ca	2750 $\pm$ 620	f, i, j, k, l	24
Co	19.6 $\pm$ 3.0	d, f, i, k	19
Cr	118 $\pm$ 29	d, f, g, i, k, l	23
Cs	5.68 $\pm$ 2.97	d, f, g, k, l	20
Cu	38.8 $\pm$ 4.2	d, f, h, k	21
Fe	45200 $\pm$ 5500	a, f, i, j, k, l	25
Ga	16.6 $\pm$ 2.5	d, g, k, l	18
K	22100 $\pm$ 2600	a, f, g, i, j, k, l	24
La	36.3 $\pm$ 4.1	d, g, k	15
Mg	12500 $\pm$ 6600	a, f, g, i, j, l	21
Mn	1180 $\pm$ 220	a, f, i, j, k, l	27
Mo	1.14 $\pm$ 1.02	d, f, h, k, l	22
Na	7790 $\pm$ 1470	a, f, i, j, k, l	20
Nb	12.9 $\pm$ 2.8	d, g, k, l	19
Nd	33.5 $\pm$ 4.2	d, g, k	15
Ni	66.7 $\pm$ 12.9	d, f, i, k, l	26
P	657 $\pm$ 185	f, j, k, l	18
Pb	38.1 $\pm$ 4.3	d, f, h, k, l	24
Rb	100 $\pm$ 15	d, g, i, k, l	24
Si	301000 $\pm$ 29000	g, i, j, k, l	18
Sr	71.9 $\pm$ 11.8	d, f, g, i, k, l	27
Th	9.96 $\pm$ 3.08	d, g, k	15
Ti	4500 $\pm$ 1360	d, g, i, j, k, l	24
U	2.61 $\pm$ 0.82	d, f, g, k	15
V	118 $\pm$ 22	d, f, i, k, l	22
Y	31.1 $\pm$ 4.9	d, g, k, l	18
Zn	106 $\pm$ 13	d, f, h, k, l	26
Zr	239 $\pm$ 20	g, i, k, l	18

<sup>§</sup> Defined in Table 1.

# Reference Soil BGS114

Table 3. Information Values

Analyte	Mean concentration (mg kg <sup>-1</sup> )	Method code <sup>§</sup>	Number of data points
Ag	0.519	d, f, l	12
B	55.4	g	3
Be	2.30	d, f, g	9
Bi	0.191	d, f	6
Br	10.5	k, l	6
Cd	0.309	d, f, l	10
Ce	72.6	d, g, k	14
Cl	54.9	k, l	6
Dy	5.57	d, g	6
Er	3.11	d, g	6
Eu	1.63	d, g	6
Gd	6.63	d, g	6
Ge	4.01	d, k	9
Hf	5.89	d, g, k	12
Ho	1.05	d, g	6
I	7.01	k, l	6
In	0.065	d	3
Li	39.2	d, f, h	12
Lu	0.454	d, g	6
Pr	8.99	d, g	6
S	217	f	3
Sb	1.27	d, f, l	9
Sc	16.0	d, k	12
Se	0.593	d, f, l	12
Sm	6.23	d, g, k	12
Sn	1.60	d, g, k, l	14
Ta	1.92	d, g, k	12
Tb	0.948	d, g	6
Te	0.183	d	3
Tl	0.647	d, f	8
Tm	0.436	d, g	6
W	1.58	d, g, k	12
Yb	2.95	d, g	6

<sup>§</sup> Defined in Table 1.

## References

Lister, B. (1982). *Evaluation of Analytical Data: A Practical Guide for Geoanalysts*. Geostandards Newsletter, 6: 175-205.

## Reference Soil BGS115

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### **Silty/peaty soil overlying limestone**

#### Sample information

Organic-rich silty/peaty soil overlying Carboniferous Limestone, from Belturbet, Co. Cavan, Ireland.

#### Sample handling

The bulk sample was dried at 30°C, disaggregated, and sieved to <2 mm, giving a final mass of 14 kg.

This was batch milled in 500 mL agate milling vessels with agate balls. A portion of the milled sample was checked by sieving to ensure it met the quality threshold (>99% at <53 µm and >95% at <32 µm).

The milled material was then combined and homogenised by rotation in a 125 L barrel.

A riffle splitter was repeatedly used to separate the homogenised material to achieve 4 portions of approximately 3.50 kg, each of which was divided using a rotary splitter into interim portions of 0.44 kg.

To create the final reference material portions, sets of four of the 0.44 kg bags were re-combined and subsequently twice divided using a rotary splitter. The final portions of at least 24 g were stored in labelled plastic bottles.

#### Homogeneity testing

Sample homogeneity testing was carried out at the interim portion stage of sample handling. Ten bags were randomly selected for homogeneity testing. From each randomly selected bag, two subsamples were used to make pressed powder pellets for XRF analysis. Samples were each analysed in a single analytical run, in a fully randomised sequence.

Statistical analysis (mean, standard deviation and relative standard deviation (RSD)) was undertaken for each quantifiable analyte. Data were also assessed for outliers using Grubb's Test (Lister, 1982), at >3 standard deviations of the mean. No data were excluded on the criteria of outliers.

Homogeneity statistics were calculated for analytes where the mean was greater than the detection limit (DL). Where standard deviation was  $\leq$ DL, the homogeneity was deemed acceptable. Where standard deviation >DL, and the RSD  $\leq$ 5%, the homogeneity was deemed acceptable; for the small number of analytes which did not pass this test, analysis of variance (ANOVA) was used. Where the ANOVA F-statistic was below the critical value, the variation between bags was not significantly greater ( $P \leq 0.05$ ) than the variation within bags, thus the homogeneity was deemed acceptable. No tested data failed the homogeneity testing criteria.

#### Intended use

This reference material is intended to be used as a quality control sample for the determination of total elemental concentrations in soils.

#### Storage

This material should be stored in a cool, dark, dry environment, with the lid securely sealed.

# Reference Soil BGS115

## Instructions for use

The material should be well mixed, by shaking multiple times with the lid still on, to ensure that subsequent sub-sampling is representative.

## Safety

Usual safety precautions apply for handling; material may contain elements or other substances at concentrations that are potentially harmful to health.

## Methods of data analysis for Reference Values

Participating laboratories were requested to undertake three independent analyses of the candidate reference material using their standard procedures. Reported data were collated, and methods intended to define total elemental concentrations were selected (see Table 1). Outlying data were identified where they exceeded two standard deviations from the mean, and excluded from calculation of the Reference Values. Data, for any analyte reported by a given laboratory, which consistently lay at the upper or lower extreme of the data populations for BGS110 to BGS119 were also excluded.

The Reference Value (the mean concentration) and the expanded uncertainty, which is calculated as twice the standard deviation of the analysis, are shown in Table 2.

Where fewer than 15 results were remaining after outlier exclusion, the data are provided as Information Values with only the mean concentration given in Table 3.

Analytical method codes are defined in Table 1 for the methods used to determine concentrations of elements listed in Tables 2 and 3.

*Table 1: Analytical methods used*

Method code	Preparation	Measurement
b	Ashed; HF, HClO <sub>4</sub> , HCl digestion	AAS
e	Ashed; HF, HClO <sub>4</sub> , HCl digestion	ICP-MS
f	HF, HClO <sub>4</sub> , HNO <sub>3</sub> digestion	ICP-MS
g	Sodium peroxide fusion	ICP-MS
h	H <sub>2</sub> O <sub>2</sub> ; HCl, HNO <sub>3</sub> , HF digestion	ICP-OES
i	Lithium borate fusion	ICP-OES
j	Lithium borate fusion bead	WD-XRFS
k	Pressed powder pellet	WD-XRFS
l	Pressed powder pellet	ED-XRFS

# Reference Soil BGS115

## Reference and Information Values

In Tables 2 and 3, the mean is reported to three significant figures. In Table 2 the expanded uncertainty is reported to the same order of magnitude as the mean for the analyte.

Table 2. Reference Values

Analyte	Mean concentration $\pm$ expanded uncertainty (mg kg <sup>-1</sup> )	Method code <sup>§</sup>	Number of data points
Al	21900 $\pm$ 3600	g, i, j, k, l	16
As	5.15 $\pm$ 1.88	e, f, k, l	21
Ba	168 $\pm$ 30	e, f, g, k, l	21
Ca	12200 $\pm$ 1800	b, f, i, j, k, l	24
Ce	36.4 $\pm$ 8.0	e, g, k	15
Co	4.56 $\pm$ 2.82	e, f, i, k	21
Cr	30.0 $\pm$ 6.2	e, f, g, i, k, l	22
Cu	22.8 $\pm$ 10.6	e, f, h, k	21
Fe	18300 $\pm$ 3400	b, f, i, j, k, l	27
Ga	4.74 $\pm$ 0.74	e, f, k, l	18
K	2370 $\pm$ 260	b, f, g, i, j, k, l	22
La	22.9 $\pm$ 6.4	e, g, k	15
Mg	1530 $\pm$ 940	b, f, g, i, j, k, l	23
Mn	95.0 $\pm$ 24.5	b, f, i, j, k, l	26
Mo	0.984 $\pm$ 0.410	e, f, h, k, l	16
Na	1630 $\pm$ 530	b, f, i, j, k, l	21
Nb	4.09 $\pm$ 1.45	e, g, k, l	18
Nd	21.1 $\pm$ 4.2	e, g, k	15
Ni	23.4 $\pm$ 3.9	e, f, i, k, l	25
P	788 $\pm$ 64	f, j, k, l	18
Pb	29.9 $\pm$ 8.2	e, f, h, k, l	27
Rb	12.2 $\pm$ 3.1	e, f, g, i, k, l	24
Se	4.04 $\pm$ 2.33	e, f, k, l	17
Si	103000 $\pm$ 21000	g, i, j, k, l	18
Sr	73.1 $\pm$ 12.2	e, f, g, i, k, l	27
Th	3.23 $\pm$ 2.71	e, g, k	15
Ti	1310 $\pm$ 450	e, g, i, j, k, l	24
U	6.75 $\pm$ 1.68	e, f, g, k	15
V	36.1 $\pm$ 5.6	e, f, i, k, l	21
Y	19.2 $\pm$ 2.3	e, g, k, l	18
Zn	24.6 $\pm$ 2.7	e, f, k, l	24
Zr	84.1 $\pm$ 17.7	g, i, k, l	18

<sup>§</sup> Defined in Table 1.

# Reference Soil BGS115

Table 3. Information Values

Analyte	Concentration (mg kg <sup>-1</sup> )	Analytical method code	Number of data points
Ag	0.090	e, f	8
B	12.2	g	3
Be	0.864	e, f, g	8
Bi	0.093	e, f	6
Br	37.1	k, l	9
Cd	0.737	e, f, l	12
Cl	244	k, l	6
Cs	1.59	e, f, g, k	14
Dy	3.27	e, g	6
Er	1.74	e, g	6
Eu	0.983	e, g	6
Gd	3.99	e, f	6
Ge	2.54	e, k	6
Hf	2.33	e, g, k	11
Ho	0.617	e, g	6
I	5.97	k, l	6
In	0.027	e	3
Li	20.7	e, f, h	12
Lu	0.222	e, g	6
Pr	4.94	e, g	6
S	6700	f	3
Sb	0.403	e, f	6
Sc	5.46	e, k	11
Sm	4.29	e, g, k	12
Sn	1.30	e, g, k	11
Ta	0.777	e, g, k	9
Tb	0.567	e, g	6
Te	0.065	e	3
Tl	0.129	e, f	9
Tm	0.226	e, g	6
W	0.617	e, g, k	9
Yb	1.48	e, g	6

<sup>§</sup> Defined in Table 1.

## References

Lister, B. (1982). *Evaluation of Analytical Data: A Practical Guide for Geoanalysts*. Geostandards Newsletter, 6: 175-205.



## Reference Soil BGS116

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### **Silty soil overlying limestone**

#### Sample information

Silty soil overlying Carboniferous Limestone, from Carlingford, Co. Louth, Ireland.

#### Sample handling

The bulk sample was dried at 30°C, disaggregated, and sieved to <2 mm, giving a final mass of 62 kg.

This was batch milled in 500 mL agate milling vessels with agate balls. A portion of the milled sample was checked by sieving to ensure it met the quality threshold (>99% at <53 µm and >95% at <32 µm).

The milled material was then combined and homogenised by rotation in a 290 L barrel.

A riffle splitter was repeatedly used to separate the homogenised material to achieve 16 portions of approximately 3.88 kg, each of which was divided using a rotary splitter into interim portions of 0.48 kg.

To create the final reference material portions, sets of six of the 0.48 kg bags were re-combined and subsequently twice divided using a rotary splitter. The final portions of at least 43 g were stored in labelled plastic bottles.

#### Homogeneity testing

Sample homogeneity testing was carried out at the interim portion stage of sample handling. Twelve bags were randomly selected for homogeneity testing. From each randomly selected bag, two subsamples were used to make pressed powder pellets for XRF analysis. Samples were each analysed in a single analytical run, in a fully randomised sequence.

Statistical analysis (mean, standard deviation and relative standard deviation (RSD)) was undertaken for each quantifiable analyte. Data were also assessed for outliers using Grubb's Test (Lister, 1982), at >3 standard deviations of the mean. No data were excluded on the criteria of outliers.

Homogeneity statistics were calculated for analytes where the mean was greater than the detection limit (DL). Where standard deviation was  $\leq$ DL, the homogeneity was deemed acceptable. Where standard deviation >DL, and the RSD  $\leq$ 5%, the homogeneity was deemed acceptable; for the small number of analytes which did not pass this test, analysis of variance (ANOVA) was used. Where the ANOVA F-statistic was below the critical value, the variation between bags was not significantly greater ( $P \leq 0.05$ ) than the variation within bags, thus the homogeneity was deemed acceptable. No tested data failed the homogeneity testing criteria.

#### Intended use

This reference material is intended to be used as a quality control sample for the determination of total elemental concentrations in soils.

#### Storage

This material should be stored in a cool, dark, dry environment, with the lid securely sealed.

# Reference Soil BGS116

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## Instructions for use

The material should be well mixed, by shaking multiple times with the lid still on, to ensure that subsequent sub-sampling is representative.

## Safety

Usual safety precautions apply for handling; material may contain elements or other substances at concentrations that are potentially harmful to health.

## Methods of data analysis for Reference Values

Participating laboratories were requested to undertake three independent analyses of the candidate reference material using their standard procedures. Reported data were collated, and methods intended to define total elemental concentrations were selected (see Table 1). Outlying data were identified where they exceeded two standard deviations from the mean, and excluded from calculation of the Reference Values. Data, for any analyte reported by a given laboratory, which consistently lay at the upper or lower extreme of the data populations for BGS110 to BGS119 were also excluded.

The Reference Value (the mean concentration) and the expanded uncertainty, which is calculated as twice the standard deviation of the analysis, are shown in Table 2.

Where fewer than 15 results were remaining after outlier exclusion, the data are provided as Information Values with only the mean concentration given in Table 3.

Analytical method codes are defined in Table 1 for the methods used to determine concentrations of elements listed in Tables 2 and 3.

*Table 1: Analytical methods used*

Method code	Preparation	Measurement
a	HF, HClO <sub>4</sub> , HCl digestion	AAS
d	HF, HClO <sub>4</sub> , HCl digestion	ICP-MS
f	HF, HClO <sub>4</sub> , HNO <sub>3</sub> digestion	ICP-MS
g	Sodium peroxide fusion	ICP-MS
h	H <sub>2</sub> O <sub>2</sub> ; HCl, HNO <sub>3</sub> , HF digestion	ICP-OES
i	Lithium borate fusion	ICP-OES
j	Lithium borate fusion bead	WD-XRFS
k	Pressed powder pellet	WD-XRFS
l	Pressed powder pellet	ED-XRFS



# Reference Soil BGS116

## Reference and Information Values

In Tables 2 and 3, the mean is reported to three significant figures. In Table 2 the expanded uncertainty is reported to the same order of magnitude as the mean for the analyte.

Table 2. Reference Values

Analyte	Mean concentration $\pm$ expanded uncertainty (mg kg <sup>-1</sup> )	Method code <sup>§</sup>	Number of data points
Al	66700 $\pm$ 11900	g, i, j, k, l	18
As	14.5 $\pm$ 3.2	f, h, k, l	21
Ba	330 $\pm$ 24	d, f, g, k, l	23
Ca	8090 $\pm$ 1670	f, i, j, k, l	24
Ce	54.7 $\pm$ 5.8	d, g, k	15
Co	13.1 $\pm$ 1.5	d, f, i, k	19
Cr	90.3 $\pm$ 26.2	d, f, g, i, k, l	23
Cu	44.1 $\pm$ 4.6	d, f, h, k	21
Fe	34000 $\pm$ 5400	a, f, i, j, k, l	26
Ga	13.2 $\pm$ 2.0	d, g, k, l	18
K	15700 $\pm$ 1900	a, f, g, i, j, k, l	24
La	24.6 $\pm$ 2.7	d, g, k	15
Mg	11400 $\pm$ 5800	a, f, g, i, j, l	21
Mn	838 $\pm$ 173	a, f, i, j, k, l	27
Mo	1.37 $\pm$ 1.07	d, f, h, k, l	22
Na	13800 $\pm$ 2000	a, f, i, j, k, l	21
Nb	10.7 $\pm$ 2.1	d, g, k, l	19
Ni	42.5 $\pm$ 8.6	d, f, i, k, l	25
P	1420 $\pm$ 310	f, j, k, l	18
Pb	31.9 $\pm$ 3.4	d, f, h, k, l	24
Rb	84.2 $\pm$ 19.7	d, f, g, i, k, l	26
Si	313000 $\pm$ 40000	g, i, j, k, l	18
Sr	129 $\pm$ 17	d, f, g, i, k, l	26
Ti	4020.0 $\pm$ 1270	d, g, i, j, k, l	24
U	3.17 $\pm$ 1.24	d, f, g, k	15
V	90.7 $\pm$ 18.0	d, f, i, k, l	22
Y	19.2 $\pm$ 3.5	d, g, k, l	18
Zn	105 $\pm$ 12	d, f, k, l	24
Zr	212 $\pm$ 22	g, i, k, l	18

<sup>§</sup> Defined in Table 1.

## Reference Soil BGS116

Table 3. Information Values

Analyte	Mean concentration (mg kg <sup>-1</sup> )	Method code <sup>§</sup>	Number of data points
Ag	0.455	d, f, l	12
B	33.5	g	3
Be	1.69	d, f, g	9
Bi	0.176	d, f	6
Br	16.0	k, l	9
Cd	0.442	d, f, l	12
Cl	75.1	k, l	6
Cs	3.38	d, f, g, k, l	14
Dy	3.38	d, g	6
Er	1.94	d, g	6
Eu	1.01	d, g	6
Gd	4.06	d, g	6
Ge	3.83	d, k	8
Hf	5.34	d, g, k	12
Ho	0.648	d, g	6
I	85.6	k, l	9
In	0.050	d	3
Li	26.9	d, f, h	12
Lu	0.277	d, g	6
Nd	21.5	d, g, k	14
Pr	5.78	d, g	6
S	387	f	3
Sb	0.684	d, f, l	9
Sc	9.99	d, k	11
Se	0.56	d, f, l	11
Sm	4.28	d, g, k	11
Sn	2.34	d, g, k, l	14
Ta	1.51	d, g, k	10
Tb	0.576	d, g	6
Te	0.14	d	3
Th	7.16	d, g, k	14
Tl	0.509	d, f	9
Tm	0.275	d, g	6
W	1.52	d, g, k	11
Yb	1.87	d, g	6

<sup>§</sup> Defined in Table 1.

## References

Lister, B. (1982). *Evaluation of Analytical Data: A Practical Guide for Geoanalysts*. Geostandards Newsletter, 6: 175-205.

## Reference Soil BGS117

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### Silty soil overlying sandstone

#### Sample information

Silty soil overlying Devonian Old Red Sandstone, from Moorpark, Co. Cork, Ireland.

#### Sample handling

The bulk sample was dried at 30°C, disaggregated, and sieved to <2 mm, giving a final mass of 74 kg.

This was batch milled in 500 mL agate milling vessels with agate balls. A portion of the milled sample was checked by sieving to ensure it met the quality threshold (>99% at <53 µm and >95% at <32 µm).

The milled material was then combined and homogenised by rotation in a 290 L barrel.

A riffle splitter was repeatedly used to separate the homogenised material to achieve 16 portions of approximately 4.63 kg, each of which was divided using a rotary splitter into interim portions of 0.58 kg.

To create the final reference material portions, sets of five of the 0.58 kg bags were re-combined and subsequently twice divided using a rotary splitter. The final portions of at least 41 g were stored in labelled plastic bottles.

#### Homogeneity testing

Sample homogeneity testing was carried out at the interim portion stage of sample handling. Twelve bags were randomly selected for homogeneity testing. From each randomly selected bag, two subsamples were used to make pressed powder pellets for XRF analysis. Samples were each analysed in a single analytical run, in a fully randomised sequence.

Statistical analysis (mean, standard deviation and relative standard deviation (RSD)) was undertaken for each quantifiable analyte. Data were also assessed for outliers using Grubb's Test (Lister, 1982), at >3 standard deviations of the mean. No data were excluded on the criteria of outliers.

Homogeneity statistics were calculated for analytes where the mean was greater than the detection limit (DL). Where standard deviation was  $\leq$ DL, the homogeneity was deemed acceptable. Where standard deviation >DL, and the RSD  $\leq$ 5%, the homogeneity was deemed acceptable; for the small number of analytes which did not pass this test, analysis of variance (ANOVA) was used. Where the ANOVA F-statistic was below the critical value, the variation between bags was not significantly greater ( $P \leq 0.05$ ) than the variation within bags, thus the homogeneity was deemed acceptable. No tested data failed the homogeneity testing criteria.

#### Intended use

This reference material is intended to be used as a quality control sample for the determination of total elemental concentrations in soils.

#### Storage

This material should be stored in a cool, dark, dry environment, with the lid securely sealed.

# Reference Soil BGS117

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## Instructions for use

The material should be well mixed, by shaking multiple times with the lid still on, to ensure that subsequent sub-sampling is representative.

## Safety

Usual safety precautions apply for handling; material may contain elements or other substances at concentrations that are potentially harmful to health.

## Methods of data analysis for Reference Values

Participating laboratories were requested to undertake three independent analyses of the candidate reference material using their standard procedures. Reported data were collated, and methods intended to define total elemental concentrations were selected (see Table 1). Outlying data were identified where they exceeded two standard deviations from the mean, and excluded from calculation of the Reference Values. Data, for any analyte reported by a given laboratory, which consistently lay at the upper or lower extreme of the data populations for BGS110 to BGS119 were also excluded.

The Reference Value (the mean concentration) and the expanded uncertainty, which is calculated as twice the standard deviation of the analysis, are shown in Table 2.

Where fewer than 15 results were remaining after outlier exclusion, the data are provided as Information Values with only the mean concentration given in Table 3.

Analytical method codes are defined in Table 1 for the methods used to determine concentrations of elements listed in Tables 2 and 3.

*Table 1: Analytical methods used*

Method code	Preparation	Measurement
a	HF, HClO <sub>4</sub> , HCl digestion	AAS
d	HF, HClO <sub>4</sub> , HCl digestion	ICP-MS
f	HF, HClO <sub>4</sub> , HNO <sub>3</sub> digestion	ICP-MS
g	Sodium peroxide fusion	ICP-MS
h	H <sub>2</sub> O <sub>2</sub> ; HCl, HNO <sub>3</sub> , HF digestion	ICP-OES
i	Lithium borate fusion	ICP-OES
j	Lithium borate fusion bead	WD-XRFS
k	Pressed powder pellet	WD-XRFS
l	Pressed powder pellet	ED-XRFS

# Reference Soil BGS117

## Reference and Information Values

In Tables 2 and 3, the mean is reported to three significant figures. In Table 2 the expanded uncertainty is reported to the same order of magnitude as the mean for the analyte.

Table 2. Reference Values

Analyte	Mean concentration $\pm$ expanded uncertainty (mg kg <sup>-1</sup> )	Method code <sup>§</sup>	Number of data points
Al	34100 $\pm$ 9400	g, j, k, l	15
As	10.0 $\pm$ 2.1	d, f, h, k, l	24
Ba	158 $\pm$ 21	d, f, g, k, l	23
Ca	2250 $\pm$ 640	f, i, j, k, l	24
Ce	43.8 $\pm$ 5.9	d, g, k	15
Co	8.21 $\pm$ 1.39	d, f, i, k	20
Cr	53.7 $\pm$ 19.9	d, f, g, i, k, l	23
Cs	3.37 $\pm$ 2.60	d, f, g, k, l	18
Cu	16.5 $\pm$ 2.3	d, f, h, k	21
Fe	21400 $\pm$ 3000	a, f, i, j, k, l	24
Ga	7.36 $\pm$ 1.10	d, g, k, l	18
K	8630 $\pm$ 1300	a, f, g, i, j, k, l	23
La	21.4 $\pm$ 4.9	d, g, k	15
Mg	3140 $\pm$ 2560	a, f, g, i, j, k, l	22
Mn	704 $\pm$ 177	a, f, i, j, k, l	26
Mo	0.92 $\pm$ 1.12	d, f, h, k, l	21
Na	2190 $\pm$ 640	a, f, i, j, k, l	20
Nb	14.1 $\pm$ 3.5	d, g, k, l	20
Ni	18.0 $\pm$ 4.8	d, f, i, k, l	24
P	1160 $\pm$ 330	f, j, k, l	18
Pb	23.1 $\pm$ 3.2	d, f, h, k, l	24
Rb	54.2 $\pm$ 6.1	d, f, g, k, l	24
Si	372000 $\pm$ 51000	g, i, j, k, l	17
Sr	27.9 $\pm$ 5.1	d, f, g, i, k, l	27
Th	5.78 $\pm$ 2.98	d, g, k	15
Ti	3820 $\pm$ 1600	d, g, i, j, k, l	24
U	1.90 $\pm$ 0.64	d, f, g, k	15
V	49.5 $\pm$ 11.5	d, f, i, k, l	23
Y	17.6 $\pm$ 1.4	g, k, l	15
Zn	63.8 $\pm$ 10.4	d, f, h, k, l	25
Zr	316 $\pm$ 45	g, i, k, l	18

<sup>§</sup> Defined in Table 1.

## Reference Soil BGS117

Table 3. Information Values

Analyte	Mean concentration (mg kg <sup>-1</sup> )	Method code <sup>§</sup>	Number of data points
Ag	0.429	d, f, l	12
B	35.1	g	3
Be	0.712	d, f, g	8
Bi	0.131	d, f	6
Br	17.4	k, l	9
Cd	0.194	d, f, l	10
Cl	90.4	k, l	6
Dy	2.45	d, g	6
Er	1.49	d, g	6
Eu	0.658	d, g	6
Gd	2.93	d, g	6
Ge	2.88	d, k	8
Hf	6.92	d, g, k	12
Ho	0.477	d, g	6
I	6.71	k, l	6
In	0.034	d,	3
Li	20.7	d, f, h	12
Lu	0.234	d, g	6
Nd	17.8	d, g, k	14
Pr	4.82	d, g	6
S	449	f	3
Sb	0.988	d, f, l	9
Sc	4.65	d, k	11
Se	0.623	d, f, k, l	13
Sm	3.14	d, g, k	9
Sn	1.66	d, g, k, l	14
Ta	1.580	d, g, k	11
Tb	0.403	d, g	6
Te	0.110	d	3
Tl	0.285	d, f	9
Tm	0.216	d, g	6
W	1.10	d, g, k	13
Yb	1.49	d, g	6

<sup>§</sup> Defined in Table 1.

## References

Lister, B. (1982). *Evaluation of Analytical Data: A Practical Guide for Geoanalysts*. Geostandards Newsletter, 6: 175-205.

## Reference Soil BGS118

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### Silty soil overlying shale

#### Sample information

Silty soil overlying Carboniferous shales with high metal (Mo, U) contents, from Glangevlin, Co. Cavan, Ireland.

#### Sample handling

The bulk sample was dried at 30°C, disaggregated, and sieved to <2 mm, giving a final mass of 41 kg.

This was batch milled in 500 mL agate milling vessels with agate balls. A portion of the milled sample was checked by sieving to ensure it met the quality threshold (>99% at <53 µm and >95% at <32 µm).

The milled material was then combined and homogenised by rotation in a 125 L barrel.

A riffle splitter was repeatedly used to separate the homogenised material to achieve 8 portions of approximately 5.13 kg, each of which was divided using a rotary splitter into interim portions of 0.64 kg.

To create the final reference material portions, sets of four of the 0.64 kg bags were re-combined and subsequently twice divided using a rotary splitter. The final portions of at least 38 g were stored in labelled plastic bottles.

#### Homogeneity testing

Sample homogeneity testing was carried out at the interim portion stage of sample handling. Twelve bags were randomly selected for homogeneity testing. From each randomly selected bag, two subsamples were used to make pressed powder pellets for XRF analysis. Samples were each analysed in a single analytical run, in a fully randomised sequence.

Statistical analysis (mean, standard deviation and relative standard deviation (RSD)) was undertaken for each quantifiable analyte. Data were also assessed for outliers using Grubb's Test (Lister, 1982), at >3 standard deviations of the mean. No data were excluded on the criteria of outliers.

Homogeneity statistics were calculated for analytes where the mean was greater than the detection limit (DL). Where standard deviation was  $\leq$ DL, the homogeneity was deemed acceptable. Where standard deviation >DL, and the RSD  $\leq$ 5%, the homogeneity was deemed acceptable; for the small number of analytes which did not pass this test, analysis of variance (ANOVA) was used. Where the ANOVA F-statistic was below the critical value, the variation between bags was not significantly greater ( $P \leq 0.05$ ) than the variation within bags, thus the homogeneity was deemed acceptable. No tested data failed the homogeneity testing criteria.

#### Intended use

This reference material is intended to be used as a quality control sample for the determination of total elemental concentrations in soils.

#### Storage

This material should be stored in a cool, dark, dry environment, with the lid securely sealed.

# Reference Soil BGS118

## Instructions for use

The material should be well mixed, by shaking multiple times with the lid still on, to ensure that subsequent sub-sampling is representative.

## Safety

Usual safety precautions apply for handling; material may contain elements or other substances at concentrations that are potentially harmful to health.

## Methods of data analysis for Reference Values

Participating laboratories were requested to undertake three independent analyses of the candidate reference material using their standard procedures. Reported data were collated, and methods intended to define total elemental concentrations were selected (see Table 1). Outlying data were identified where they exceeded two standard deviations from the mean, and excluded from calculation of the Reference Values. Data, for any analyte reported by a given laboratory, which consistently lay at the upper or lower extreme of the data populations for BGS110 to BGS119 were also excluded.

The Reference Value (the mean concentration) and the expanded uncertainty, which is calculated as twice the standard deviation of the analysis, are shown in Table 2.

Where fewer than 15 results were remaining after outlier exclusion, the data are provided as Information Values with only the mean concentration given in Table 3.

Analytical method codes are defined in Table 1 for the methods used to determine concentrations of elements listed in Tables 2 and 3.

*Table 1: Analytical methods used*

Method code	Preparation	Measurement
a	HF, HClO <sub>4</sub> , HCl digestion	AAS
d	HF, HClO <sub>4</sub> , HCl digestion	ICP-MS
f	HF, HClO <sub>4</sub> , HNO <sub>3</sub> digestion	ICP-MS
g	Sodium peroxide fusion	ICP-MS
h	H <sub>2</sub> O <sub>2</sub> ; HCl, HNO <sub>3</sub> , HF digestion	ICP-OES
i	Lithium borate fusion	ICP-OES
j	Lithium borate fusion bead	WD-XRFS
k	Pressed powder pellet	WD-XRFS
l	Pressed powder pellet	ED-XRFS





# Reference Soil BGS118

## Reference and Information Values

In Tables 2 and 3, the mean is reported to three significant figures. In Table 2 the expanded uncertainty is reported to the same order of magnitude as the mean for the analyte.

Table 2. Reference Values

Analyte	Mean concentration ± expanded uncertainty (mg kg <sup>-1</sup> )	Method code <sup>§</sup>	Number of data points
Al	88100 ± 6900	g, i, j, k, l	18
As	17.1 ± 6.1	d, f, h, k, l	24
Ba	224 ± 21	d, f, g, k, l	22
Ca	445 ± 362	f, i, j, k, l	23
Ce	86.4 ± 12.7	d, g, k	15
Co	10.4 ± 2.3	d, f, i, k	19
Cr	94.5 ± 17.9	d, f, g, i, k, l	23
Cs	4.88 ± 2.26	d, f, g, k, l	20
Cu	113 ± 13	d, f, h, k	21
Fe	35500 ± 2800	a, f, i, j, k, l	24
Ga	20.4 ± 3.0	d, g, k, l	18
K	8760 ± 530	a, f, g, i, j, k, l	23
La	43.4 ± 7.4	d, g, k	15
Mg	4440 ± 3510	a, f, g, i, j, k, l	24
Mn	334 ± 119	a, f, i, j, k, l	25
Mo	20.4 ± 3.1	d, f, h, k, l	23
Na	2010 ± 530	a, f, i, j, k, l	21
Nb	17.5 ± 2.9	d, g, k, l	19
Nd	35.9 ± 5.0	d, g, k	15
Ni	28.0 ± 4.2	d, f, i, k, l	25
P	1480 ± 200	f, j, k, l	18
Pb	31.1 ± 4.0	d, f, h, k, l	24
Rb	68.2 ± 15.5	d, f, g, i, k, l	27
Se	4.35 ± 1.41	d, f, k, l	18
Si	243000 ± 27000	g, i, j, k, l	18
Sn	4.38 ± 2.82	d, g, k, l	15
Sr	77.2 ± 15.9	d, f, g, i, k, l	27
Ti	4590 ± 1000	d, g, i, j, k, l	24
U	9.54 ± 1.99	d, f, g, k	15
V	177 ± 35	d, f, i, k, l	23
Y	24.7 ± 3.1	d, g, k, l	18
Zn	60.3 ± 8.3	d, f, h, k, l	25
Zr	130 ± 18	g, i, k, l	18

<sup>§</sup> Defined in Table 1.

# Reference Soil BGS118

Table 3. Information Values

Analyte	Mean concentration (mg kg <sup>-1</sup> )	Method code <sup>§</sup>	Number of data points
Ag	0.457	d, f, l	12
B	51.9	g	3
Be	2.01	d, f, g	8
Bi	0.264	d, f	6
Br	23.1	k, l	9
Cd	1.25	d, f, l	12
Cl	117	k, l	6
Dy	4.65	d, g	6
Er	2.48	d, g	6
Eu	1.42	d, g	6
Gd	6.00	d, g	6
Ge	3.59	d, k	9
Hf	3.81	d, g, k	12
Ho	0.872	d, g	6
I	7.69	k, l	6
In	0.0763	d	3
Li	76.4	d, f, h	12
Lu	0.334	d, g	6
Pr	9.68	d, g	6
S	938	f	3
Sb	2.04	d, f, l	9
Sc	13.5	d, k	12
Sm	6.42	d, g, k	12
Ta	2.42	d, g, k	12
Tb	0.811	d, g	6
Te	0.142	d	3
Th	9.45	d, g, k	14
Tl	1.16	d, f	9
Tm	0.338	d, g	6
W	2.00	d, g, k	13
Yb	2.23	d, g	6

<sup>§</sup> Defined in Table 1.

## References

Lister, B. (1982). *Evaluation of Analytical Data: A Practical Guide for Geoanalysts*. Geostandards Newsletter, 6: 175-205.

## Reference Soil BGS119

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### Soil with industrial contamination (<250 µm)

#### Sample information

A combination of soils with industrial contamination, collected from sites across the UK.

#### Sample handling

Individual samples were received, having been previously dried at <40°C and passed through a 250 µm sieve. These were then combined into a 16 kg bulk material which was homogenised by rotation in a 42 L barrel.

A riffle splitter was used to separate the homogenised material to achieve 2 portions of approximately 8 kg, each of which was divided using a rotary splitter into interim portions of approximately 1 kg.

To create the final reference material portions, nine of the 1 kg bags were re-combined and subsequently separated using a riffle splitter and a rotary divider. The final portions of at least 26 g were stored in labelled plastic bottles.

#### Homogeneity testing

Sample homogeneity testing was carried out at the interim portion stage of sample handling. From each interim portion, three subsamples were used to make pressed powder pellets for XRF analysis. Samples were each analysed in a single analytical run, in a fully randomised sequence.

Statistical analysis (mean, standard deviation and relative standard deviation (RSD)) was undertaken for each quantifiable analyte. Data were also assessed for outliers using Grubb's Test (Lister, 1982), at >3 standard deviations of the mean. No data were excluded on the criteria of outliers.

Homogeneity statistics were calculated for analytes where the mean was greater than the detection limit (DL). Where standard deviation was  $\leq$ DL, the homogeneity was deemed acceptable. Where standard deviation >DL, and the RSD  $\leq$ 5%, the homogeneity was deemed acceptable; for the small number of analytes which did not pass this test, analysis of variance (ANOVA) was used. Where the ANOVA F-statistic was below the critical value, the variation between bags was not significantly greater ( $P \leq 0.05$ ) than the variation within bags (except for Ba,  $p=0.04$ ), thus the homogeneity was deemed acceptable. No tested data failed the homogeneity testing criteria.

#### Intended use

This reference material is intended to be used as a quality control sample for the determination of total elemental concentrations in soils.

#### Storage

This material should be stored in a cool, dark, dry environment, with the lid securely sealed.

# Reference Soil BGS119

## Instructions for use

The material should be well mixed, by shaking multiple times with the lid still on, to ensure that subsequent sub-sampling is representative.

## Safety

Usual safety precautions apply for handling, particularly as this is industrially contaminated soil; material may contain elements or other substances at concentrations that are potentially harmful to health.

## Methods of data analysis for Reference Values

Participating laboratories were requested to undertake three independent analyses of the candidate reference material using their standard procedures. Reported data were collated, and methods intended to define total elemental concentrations were selected (see Table 1). Outlying data were identified where they exceeded two standard deviations from the mean, and excluded from calculation of the Reference Values. Data, for any analyte reported by a given laboratory, which consistently lay at the upper or lower extreme of the data populations for BGS110 to BGS119 were also excluded.

The Reference Value (the mean concentration) and the expanded uncertainty, which is calculated as twice the standard deviation of the analysis, are shown in Table 2.

Where fewer than 15 results were remaining after outlier exclusion, the data are provided as Information Values with only the mean concentration given in Table 3.

Analytical method codes are defined in Table 1 for the methods used to determine concentrations of elements listed in Tables 2 and 3.

*Table 1: Analytical methods used*

Method code	Preparation	Measurement
b	Ashed; HF, HClO <sub>4</sub> , HCl digestion	AAS
e	Ashed; HF, HClO <sub>4</sub> , HCl digestion	ICP-MS
f	HF, HClO <sub>4</sub> , HNO <sub>3</sub> digestion	ICP-MS
g	Sodium peroxide fusion	ICP-MS
h	H <sub>2</sub> O <sub>2</sub> ; HCl, HNO <sub>3</sub> , HF digestion	ICP-OES
i	Lithium borate fusion	ICP-OES
j	Lithium borate fusion bead	WD-XRFS
k	Pressed powder pellet	WD-XRFS
l	Pressed powder pellet	ED-XRFS

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## Reference and Information Values

In Tables 2 and 3, the mean is reported to three significant figures. In Table 2 the expanded uncertainty is reported to the same order of magnitude as the mean for the analyte.

Table 2. Reference Values

Analyte	Mean concentration $\pm$ expanded uncertainty (mg kg <sup>-1</sup> )	Method code <sup>§</sup>	Number of data points
Al	75000 $\pm$ 17300	g, i, j, k, l	18
As	350 $\pm$ 34	e, f, h, k, l	24
Ba	500 $\pm$ 62	e, f, g, k, l	24
Ca	17200 $\pm$ 2800	b, f, i, j, k, l	24
Ce	84.5 $\pm$ 22.4	e, g, k,	15
Co	26.1 $\pm$ 5.6	e, f, i, k,	20
Cr	127 $\pm$ 84	e, f, g, i, k, l	22
Cs	7.40 $\pm$ 3.81	e, f, g, k, l	21
Cu	77.1 $\pm$ 11.1	e, f, h, k,	21
Fe	76100 $\pm$ 13100	b, f, i, j, k, l	27
Ga	16.4 $\pm$ 2.2	e, g, k, l	18
K	16200 $\pm$ 1600	b, f, g, i, j, l	21
La	40.1 $\pm$ 8.4	e, g, k,	15
Mg	4630 $\pm$ 2750	b, f, g, i, j, l	21
Mn	1180 $\pm$ 230	b, f, i, j, k, l	26
Mo	5.94 $\pm$ 1.50	e, f, h, k, l	22
Na	3210 $\pm$ 730	b, f, i, j, k, l	20
Nb	12.3 $\pm$ 2.9	e, g, k, l	19
Ni	67.0 $\pm$ 9.6	e, f, k, l	24
P	1670 $\pm$ 340	f, j, k, l	18
Pb	870 $\pm$ 204	e, f, h, k, l	27
Rb	94.8 $\pm$ 17.6	e, f, g, i, k, l	25
Si	231000 $\pm$ 46000	g, i, j, k, l	17
Sn	12.5 $\pm$ 9.3	e, g, k, l	15
Sr	145 $\pm$ 25	e, f, g, i, k, l	26
Th	13.7 $\pm$ 6.9	e, g, k,	15
Ti	3770 $\pm$ 1390	e, g, i, j, k, l	24
U	2.59 $\pm$ 0.97	e, f, g, k,	15
V	177 $\pm$ 23	e, f, k, l	21
W	2.91 $\pm$ 2.9	e, g, k,	15
Y	30.4 $\pm$ 8.5	e, g, k, l	18
Zn	338 $\pm$ 48	e, f, k, l	24
Zr	234 $\pm$ 29	g, i, k, l	18

<sup>§</sup> Defined in Table 1.

# Reference Soil BGS119

Table 3. Information Values

Analyte	Mean concentration (mg kg <sup>-1</sup> )	Method code <sup>§</sup>	Number of data points
Ag	1.55	e, f, l	12
B	58.5	g	3
Be	3.10	e, f, g	9
Bi	0.818	e, f	6
Br	14.0	k, l	6
Cd	2.82	e, f, l	12
Cl	259	k, l	5
Dy	5.62	e, g	6
Er	3.09	e, g	6
Eu	1.59	e, g	6
Gd	6.73	e, g	6
Ge	7.26	e, k	9
Hf	5.94	e, g, k	12
Ho	1.04	e, g	6
I	7.83	k, l	6
In	0.624	e, l	6
Li	81.9	e, f, h	12
Lu	0.446	e, g	6
Nd	36.0	e, g, k	14
Pr	9.05	e, g	6
S	6260	f	3
Sb	22.5	e, f, k, l	12
Sc	14.5	e, k	12
Se	2.80	e, f, k, l	14
Sm	6.21	e, g, k	11
Ta	2.01	e, g, k	11
Tb	0.950	e, g	6
Te	0.440	e	3
Tl	2.01	e, f	9
Tm	0.443	e, g	6
Yb	2.94	e, g	6

<sup>§</sup> Defined in Table 1.

## References

Lister, B. (1982). *Evaluation of Analytical Data: A Practical Guide for Geoanalysts*. Geostandards Newsletter, 6: 175-205.