

Soil Reference Material Data Sheets: BGS120 to BGS126

Environmental Change, Adaptation and Resilience Programme
Internal Report OR/23/055



BRITISH GEOLOGICAL SURVEY

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Soil Reference Material Data Sheets: BGS120 to BGS126

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1 Introduction

There is a paucity of appropriately characterised Reference Materials to inform quality assessments for agronomic chemical analysis of soil. These analyses are used to provide data for systematic regional geochemical/agricultural soil surveys and underpin on-farm decision making for soil fertility and crop yield management. This partially unmet need for reference materials is further exacerbated in low-income/resource settings and limits the frequency with which quality control samples can be routinely analysed: this gap has been specifically identified by the FAO Global Soil Laboratory Network (GLOSOLAN <https://www.fao.org/global-soil-partnership/glosolan/en/>) capacity strengthening activities. The insufficient supply of appropriately characterised reference materials, particularly soils from tropical sources, has been identified as a limiting factor in the global adoption of effective, harmonised analytical methods.

The customer base for new reference materials is potentially broad. In addition to any of the ~1000-strong GLOSOLAN global community of laboratories, other commercial and research laboratories providing agricultural soil sample analyses, in the UK and internationally, as well as academic researchers and PhD students in environmental geochemistry.

Existing soil reference materials on the market may present a matrix-matching problem as they are generally milled to <75 µm, as required for total elemental concentration methods. Routine/survey agricultural soil testing is usually undertaken on a more coarse, un-milled sieved fraction, such as <2 mm or <4 mm. This difference in size fraction (especially the changes to particle surfaces caused by additional mineral breakdown during milling) may cause unintended changes to soil texture and, together with an unrepresentative reduction in sample heterogeneity, lead to systematically biased analysis in relation to conventional soil analysis. The use of un-milled soil material for these Reference Materials has avoided such adverse effects that might influence parameter measurement (e.g., pH) or provide enhanced nutrient availability that is itself unrealistic (e.g., available P). Furthermore, extractable (not total) concentrations are used in agriculture to assess the fertility status of soil and make nutrient input decisions appropriate to the next crop.

BGS has experience of creating reference materials across a range of rock, sediment, and soil matrices. Ten soil Reference Materials are available (BGS110-BGS119) which are optimised for major, minor and trace element variation from nine contrasting soil parent materials, and one anthropogenically contaminated soil (Kalra et al., 2020). BGS therefore set out to create suite of new reference materials that will help to support high-quality analysis for agriculturally relevant parameters, to augment the soil Reference Materials already offered for sale. This suite comprises a range of sample matrices from temperate silt-rich to peat-rich (BGS120 to BGS124), and equatorial agricultural soils (BGS125 to BGS126).

2 Methods

2.1 SAMPLE INFORMATION

Reference Materials BGS120-BGS124 were prepared using excess sample material collected from agricultural land in Ireland. These were bulked into five classes based on observation of soil colour and texture. A further two bulk samples were collected from agricultural land in Kenya to form BGS125 and BGS126, to support availability of representative materials for tropical soil testing. In both cases, access to and collection of soil for the creation of reference materials was supported by local partners.

2.2 SAMPLE HANDLING

The detailed approach to the creation of the seven soil reference materials in the BGS120-BGS126 series is provided on each Data Sheet (Appendix 3).

Briefly, for materials BGS120-BGS124 the soil sample materials were dried at 30°C, disaggregated and sieved to <2 mm. These were supplied in individual ~0.5 kg sample bags, which were combined after sieving and homogenised using barrel rotation. The bulk material was divided first by riffle splitter, and then by rotary splitter to create the final reference material portions. These are provided in labelled plastic bottles.

For materials BGS125-BGS126 the samples were autoclaved to ensure they were sterile, prior to processing. Processing then proceeded by the same approach as BGS120-BGS124.

2.3 HOMOGENEITY TESTING

2.3.1 BGS120-BGS124

Sample homogeneity testing was carried out using eight bottles of material selected at random. From each randomly selected bottle, two subsamples were analysed for loss-on-ignition (LOI), pH and total P concentration. Samples were analysed using single analytical batches.

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

Where standard deviation was less than or equal to the detection limit (DL), or 0.1 pH units, the homogeneity was deemed acceptable. Where standard deviation >DL, and the RSD ≤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 bottles was not significantly greater ($P \leq 0.05$) than the variation within bottles, thus the homogeneity was deemed acceptable. No tested data failed the homogeneity testing criteria.

2.3.2 BGS125-BGS126

Sample homogeneity testing was carried out using eight bottles of material selected at random. From each randomly selected bottle, three subsamples were analysed for loss-on-ignition (LOI), and two subsamples for pH and total P concentration. Samples were analysed using single analytical batches.

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

Where standard deviation was less than or equal to the detection limit (DL) or 0.1 pH units, the homogeneity was deemed acceptable. Where standard deviation >DL, and the RSD ≤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 bottles was not significantly greater ($P \leq 0.05$) than the

variation within bottles, thus the homogeneity was deemed acceptable. No tested data failed the homogeneity testing criteria.

2.4 SELECTION OF TARGET METHODS FOR CANDIDATE REFERENCE MATERIAL CHARACTERISATION

The soils were characterised for parameters selected for their relevance to agriculture and geochemistry. Methods of interest were determined through market research and consultation with the FAO GLOSOLAN Ring test (<https://www.slideshare.net/Soils2012/glosolan-pt-2019>, last accessed 10/11/2023), from which a list of the most widely used methods was assembled for use in this characterisation study. A prioritised range of methods based on the frequency of use was provided to participating laboratories who carried out measurement of the specified parameter using their preferred methods, thereby highlighting the most widely used analyses for each analyte in our European testing laboratories. These parameters, with methodological categories, are summarised in Table 1.

2.5 COLLABORATIVE CHARACTERISATION APPROACH

The characterisation of the materials was achieved through an inter-laboratory analysis scheme in which 13 separate European laboratories regularly analysing agricultural soil samples for research or commercial purposes, all of whom voluntarily participated.

Laboratories were contacted by email, and once participation was confirmed, they were sent sufficient material (two or three bottles) of each material for testing using their preferred methods. Recognising the large resource commitment this could entail, laboratories were not asked to supply data for all methods, rather to supply data only for those methods as they felt able.

The laboratories were provided with a template in which to report the results and the methods used, to facilitate subsequent data processing. The reported data represent the parameters for which there is reasonable consistency, and thus interoperability of results.

Analytical performance and laboratory data quality was assessed using blind analysis of a commercially available soil QC material (*AgroMAT* AG-1, from SCP Science, Canada) that had documented data for many, but not all, of the tests requested. One bottle of this material was supplied to all participating laboratories.

Laboratories were requested to undertake three replicate analyses of each candidate reference material and a single analysis of the QC material, by all their usual methods in the scope of this exercise. See Appendix 1 for the 'instructions to analysts' sent to each participating laboratory and Appendix 2 provides the proforma data reporting structure which they were requested to use.

2.6 SAMPLE CHARACTERISATION METHODS

The prioritised methods offered to the participating laboratories and those from which sufficient data were received to permit calculation of reference values are shown in Table 1. A wide range of methods are used for measuring extractable concentrations of analyte suites relevant to agricultural applications. Many of these were used by the contributing laboratories, however the methods exhibited variable recoveries and there was insufficient data for any one method, such that it was not possible to calculate consensus values. Consequently, consensus data for these parameters cannot currently be reported (Table 1, footnote).

Table 1. Analytical methods employed in the characterisation of BGS120 to BGS126, prioritised by frequency of provision.

Priority	Analytical method	Method included
1	pH	Water slurry CaCl ₂ slurry KCl slurry
2	Loss on ignition	LOI at 450°C
3	Total carbon	Combustion
4	Organic carbon	Walkley Black, & Tyurin Combustion
5	Total nitrogen	Kjeldahl Combustion
6	Available nitrogen	No*
7	Extractable/available phosphorus	Olsen*
8	Exchangeable/available agronomic cations and anions	No*
9	Electrical conductivity	1:5 & 1:2.5 soil:water ratio

* Insufficient number of data values were reported for:
 available nitrogen using either potassium sulfate or potassium chloride
 available phosphorus using Mehlich 3, Morgan or Machigin extraction
 exchangeable cations using Mehlich 1, Mehlich 3, Morgan, acid oxalate (Tamm),
 ammonium nitrate, ammonium acetate and barium chloride extraction

2.7 DATA HANDLING

When data were received from the contributing laboratories, an archive of the original version was stored before processing commenced on a working copy. Formatting the datasets into a common standard was facilitated by having provided a reporting template including specified reporting units. Data and metadata were imported into, and managed within, a relational database (MS Access) before being exported for data handling using MS Excel, and subsequent visualisations in R (R Core Team, 2023). Post-database, all data were managed independently for each reference material, using a standardised workflow, and finally, validated against the submitted data as part of the quality assurance workflow.

A large amount of variation in both the general and specific (reagent strength/quantity) method used by the participating laboratories was observed. The methods excluded from further consideration on this basis that insufficient laboratories were reporting it, are summarised in the footnote to Table 1. Of particular note, is that too few consistent methods were used for measurement of extractable cations. Each method is optimised for different soil types, and produces a different recovery so the data cannot be compiled as a single dataset. No one method was used by enough laboratories to provide sufficient confidence in proposing reference values.

Further inspection of the remaining analytical methods was undertaken to ensure that the data arising could be treated as a single parameter for statistical summary purposes. The methods are described more fully below and are summarised in Table 2.

Table 2: Analytical methods used.

Analyte	Method	QC data available?
pH	Soil:water 1:2 to 1:5	Yes
	Soil:KCl 1:2.5 to 1:5	No
	Soil:CaCl ₂ 1:2 to 1:5	No
Electrical conductivity	Soil:water 1:5	No
Loss on ignition (LOI)	LOI @ 450°C	Yes
Carbon	Total - Dry combustion	No
	Organic - Dry combustion	No
	Organic - Walkley Black or Tyurin	Yes
Nitrogen	Dry combustion	No
	Kjeldahl	No
Phosphorus	Olsen	Yes

2.7.1 pH

Three pH methods were reported by participating laboratories, pH in water, pH in KCl, and pH in CaCl₂, in sufficient numbers that these parameters could all be incorporated. The water pH was determined by shaking or stirring 5-20 g of soil with deionised water mixed in a ratio of 1:2 to 1:5 for 20 minutes to 24 hours. The KCl pH was determined by shaking or stirring 5-20 g of soil with 1 or 2 mol L⁻¹ KCl mixed in a ratio of 1:2.5 to 1:5 for 20 minutes to 24 hours. The CaCl₂ pH was determined by shaking or stirring 5-10 g of soil with 0.01 mol L⁻¹ CaCl₂ mixed in a ratio of 1:2 to 1:5 for 20-60 minutes. The within parameter data indicated that differences in the soil:solution ratios did not preclude their joint use to form a Data Sheet value.

2.7.2 Electrical conductivity

The majority of laboratories providing EC data had used a 1:5 soil:water ratio by shaking or stirring 5-10 g of soil with deionised water mixed for 60 minutes. Only one laboratory reported data determined using a 1:2.5 ratio, consequently, this data was not incorporated into the final dataset.

2.7.3 Loss-on-ignition

Loss on ignition (LOI) determined at 450°C (as a proxy for organic matter) was reported by a large proportion of the laboratories. The methods calculated the relative weight loss after heating 1-5 g of sample in a muffle furnace at 450°C for periods of 3-12 hours.

2.7.4 Carbon – total and organic

Although there are recorded differences between the values derived from different organic carbon methods, the differences between organic carbon concentration data determined by either the Walkley Black or Tyurin methods were indistinguishable, so the datasets have been processed together to provide a single consensus value (*organic carbon - wet*) for each reference material. The methods used a sample mass of 0.5-3 g, and the Walkley Black method incorporated either potentiometric titration or colorimetric determination.

Organic carbon concentration determined by dry combustion has been processed independently to provide a separate value (*organic carbon - dry*). The methods used a sample mass of 0.01-0.025 g and combustion at 500-900°C.

2.7.5 Nitrogen - total

Total nitrogen was determined by two methods, so each dataset has been processed independently to provide method specific values for Kjeldahl (*total nitrogen - wet*) and dry

combustion (*total nitrogen - dry*). Sample masses of 0.2-10 g were used for Kjeldahl analysis with determination by colorimetry, ion selective electrode or flow injection analysis. Sample masses of 0.015-1 g were used for combustion analysis at 500-1350°C.

2.7.6 Phosphorus – available

Available phosphorus was determined five different methods: flow analysis, Machigin, Mehlich 3, Morgan and Olsen methods. Only the Olsen method was used by enough laboratories to be incorporated into the final dataset. Sample masses of 1-5 g were used with colorimetric determination.

2.8 QUALITY CONTROL

Subsequent to excluding any methods which were reported by fewer than three laboratories, the quality of the remaining data was assessed.

Benchmarking resulted in the exclusion of data falling outside the defined confidence intervals provided with the quality control sample for parameters where there were reference values available: pH (water); LOI @450°C; carbon – organic; phosphorus – available. Data supplied for organic carbon from one laboratory were excluded on this basis.

Outlying data were identified where they exceeded two standard deviations from the mean. This resulted in the removal of ~5% of data submitted for all materials from all laboratories. Additionally for analytes without quality control reference values, data from a given laboratory which consistently lay at the upper or lower extreme of the data population for all materials were also excluded; this affected data supplied by one laboratory, data for total carbon were excluded for all materials.

The Reference Value, comprising the mean and the expanded uncertainty (twice the standard deviation of the analysis), were then calculated. Where fewer than 15 results remained after any exclusions, the data are provided as Information Values, with only the mean concentration provided.

3 The BGS120-BGS126 series Soil Reference Materials

The Data Sheets for each reference material, providing a summary of all methods and the Reference and Information Values, are presented in Appendix 3. Data is presented for 11 parameters: soil pH determined in water, 0.01 mol L⁻¹ CaCl₂ and 1 mol L⁻¹ KCl; loss on ignition (LOI) determined at 450°C (as a proxy for organic matter); total carbon by combustion; organic carbon (dry) determined by combustion; organic carbon (wet) determined by the Walkley Black or Tyurin methods; total nitrogen (dry) determined by combustion electrical conductivity; total nitrogen(wet) determined by the Kjeldahl method; available phosphorus determined by the Olsen method; and electrical conductivity.

A summary of the data is shown in Table 3 and Table 4, showing the mean and the expanded uncertainty for Reference Values and the mean for Information Values. Comparisons of these data are shown in Figure 1.

Data for the BGS120 to BGS123 illustrate a trend of increasing total carbon, organic carbon (by both methods), LOI and total nitrogen (by both methods) and decreasing pH (by all methods). BGS124 has values lower than any of these and the highest pH of the temperate soils. These parameters offer distinct differences between these five soils, which should suit different analytical programmes. For practical purposes there is no difference in terms of electrical conductivity and available phosphorus concentrations for BGS120-BGS124.

Of the two tropical soils, the total carbon content and organic carbon content of BGS126 are consistently greater than BGS125. Additionally, the total carbon and organic carbon content of both these soils is generally lower than temperate soils BGS120 to BGS123. The mean total nitrogen content and mean electrical conductivity are generally lower, and mean available phosphorous higher, than in the temperate soils; available phosphorus in BGS125 has the highest mean value of the whole suite. The pH of BGS126 is consistently lower than BGS125 but otherwise the data align with the pH of BGS120-BGS122 and BGS124.

Other noticeable differences between measured parameters in all of the soils are seen for BGS123 compared to the other materials: total carbon, where six reference materials ranged from 18,300 mg kg⁻¹ to 146,000 mg kg⁻¹ with BGS123 having particularly high concentration at 394,000 mg kg⁻¹; LOI was generally 5.2% to 26.3%, and exceptionally a value of 72.7% in BGS123; and soil pH where BGS123 was 4.2 to 4.8 (method dependent), compared to values of 5.4 to 7.2 for the other materials and methods.

The relatively large confidence intervals for Reference Values reported for total nitrogen (wet), electrical conductivity and available phosphorus could be due to factors such as bias between the methods employed by the individual laboratories whilst fulfilling the quality checks employed. The future participation of additional laboratories using these methods would lead to improved estimates of confidence intervals that could be reduced.

Table 3: Reference Values and Information Values for BGS120-BGS123.

Analyte	BGS120	BGS121	BGS122	BGS123
pH in water	6.99 ± 0.28	6.91 ± 0.31	6.17 ± 0.38	4.76 ± 0.22
pH in KCl	6.55 ± 0.42	6.52 ± 0.51	5.80 ± 0.27	4.21
pH in CaCl ₂	6.64	6.58	5.84	4.39
LOI @ 450°C (wt %)	8.78 ± 1.13	14.9 ± 1.7	26.3 ± 4.1	72.7 ± 7.7
Total carbon (mg kg ⁻¹)	45500 ± 2500	85600 ± 9800	146000 ± 25000	394000 ± 48000
Organic carbon – dry (mg kg ⁻¹)	42900	70300	122000	344000
Organic carbon – wet (mg kg ⁻¹)	50300	69700	123000	242000
Total nitrogen – dry (mg kg ⁻¹)	4140 ± 410	6280 ± 1070	9070 ± 1320	17900 ± 2600
Total nitrogen – wet (mg kg ⁻¹)	4160	6630 ± 5530	8070 ± 2420	16500
Available phosphorus (mg kg ⁻¹)	23.7 ± 14.3	24.8 ± 14	25.3 ± 17	25.3 ± 8.3
Electrical conductivity (µS cm ⁻¹)	288 ± 201	412 ± 239	384 ± 205	420 ± 123

Table 4: Reference Values and Information Values for BGS124-BGS126.

Analyte	BGS124	BGS125	BGS126
pH in water	7.12 ± 0.25	6.98 ± 0.16	6.44 ± 0.15
pH in KCl	6.67 ± 0.49	5.83	5.40
pH in CaCl ₂	6.70	6.42	5.89
LOI @ 450°C (wt %)	6.10 ± 0.64	5.25 ± 0.67	9.54 ± 1.02
Total carbon (mg kg ⁻¹)	29900 ± 2000	18300 ± 900	28800 ± 1300
Organic carbon – dry (mg kg ⁻¹)	28200	18500	29100
Organic carbon – wet (mg kg ⁻¹)	30900	20900	26000
Total nitrogen – dry (mg kg ⁻¹)	2710 ± 90	1230 ± 140	1790 ± 150
Total nitrogen – wet (mg kg ⁻¹)	3080 ± 3200	1450	1710
Available phosphorus (mg kg ⁻¹)	16.8 ± 10.2	60.8 ± 31.3	37.9 ± 39
Electrical conductivity (µS cm ⁻¹)	276 ± 152	128 ± 26	128 ± 23

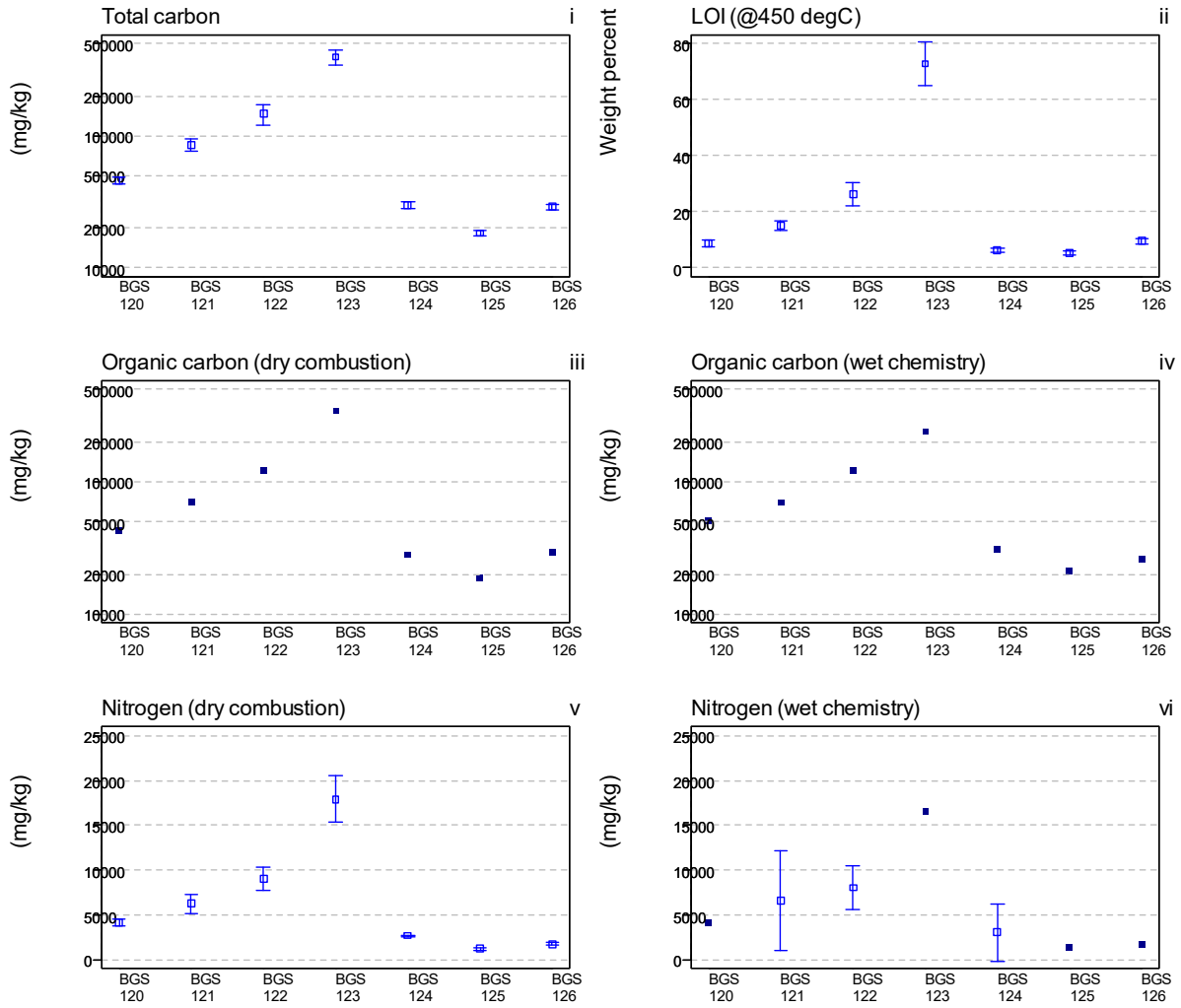


Figure 1 (i-vi): BGS120-BGS126 Reference Values (open blue symbols) and Information Values (dark blue squares) for total carbon, loss on ignition (LOI) at 450°C, organic carbon and total nitrogen. Error bars show expanded uncertainty (twice standard deviation of the dataset).

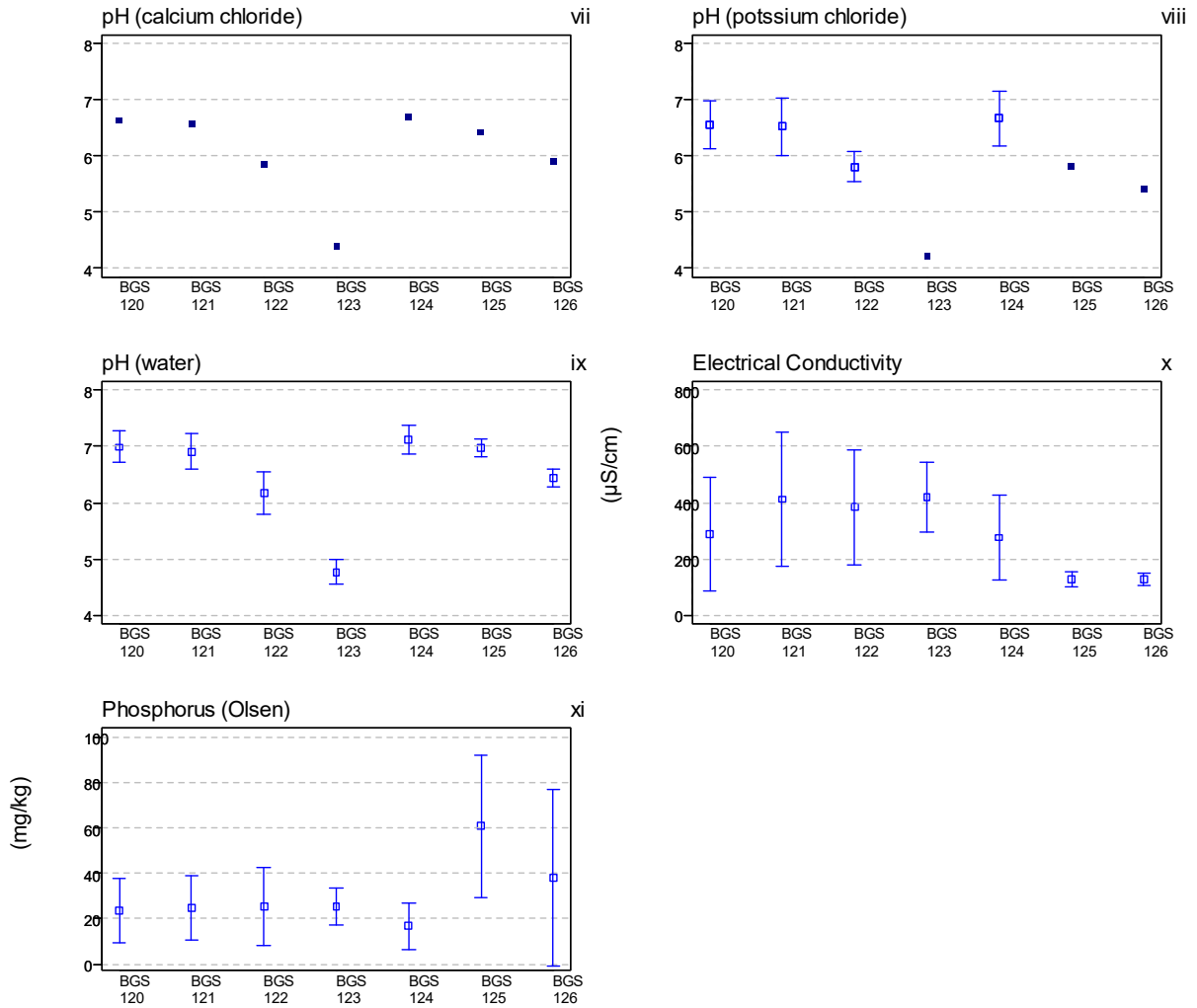


Figure 1 (vii-xi): BGS120-BGS126 Reference Values (open blue symbols) and Information Values (dark blue squares) for pH, electrical conductivity and available phosphorous. Error bars show expanded uncertainty (twice standard deviation of the dataset).

3.1 USE AND DISTRIBUTION OF THE REFERENCE MATERIALS

The larger particle size of these reference materials means that specific care needs to be taken to homogenise the sample bottle by shaking and repeated inversion prior to taking a subsample for analysis. They have thus been provided in plastic bottles with sufficient space above the soil to ensure complete mixing can be achieved.

Sets of sterile gamma irradiated soils are available for international shipping.

4 Lessons learned for future reference material production

The inclusion of a QC material to be analysed alongside the candidate reference materials provided an independent benchmark of individual laboratory data and a degree of traceability for the values calculated for these reference materials. This additional QC check afforded an improvement beyond simply examining reported datasets for instances of repeatedly extreme data values. The reference material used was *AgroMAT AG-1*. This approach should be adopted in all future production of reference values.

Supplying a data reporting template to the participating laboratories had the anticipated benefit of streamlining initial data handling and encouraging reporting information on analytical methodologies employed. Previous exercises where laboratories reported data in their routine format had required considerable input to format the data into a suitable and consistent layout.

The use of Excel macros, or other reproducible data processing approaches, may improve the speed and traceability of the workflow when preparing data for multiple reference materials.

5 Further potential benefits of these materials

The large disparity in procedures for measurement of extractable cations (e.g., Ca, K, Mg, Na) means that although data were generally reported by the 13 laboratories, none of these were sufficiently consistent across three or more laboratories to enable reporting of Reference Values or Information Values. This existing dataset could be strengthened by future participation of additional laboratories to enable specific methods for these analytes to be reported on these seven reference materials, widening their usefulness and appeal for agricultural users.

Three reference materials were also tested to screen them as candidates to provide quality control data for stable isotope analysis of soils, specifically chosen to reflect a range of C and N concentrations, as well as $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ values. The new materials BGS123 and BGS125, as well as BGS119, were used for this investigation. These data will be separately reported, and the materials are separately packaged for this purpose.

Appendix 1 Instructions to Analysts for participating laboratories

Thank you for agreeing to participate in the characterisation of this suite of Candidate Reference Materials using the methods you have previously volunteered.

Sample description and prior preparation

The materials have been dried at <40°C, disaggregated, and sieved to pass a 2 mm sieve. They have been homogenised, and the bulk representatively split by riffle and rotary divider to achieve the portions supplied. The QC material has been milled to pass a 75 µm sieve.

Instructions to analysts

These materials are intended for laboratory testing, and should only be used by trained personnel.

Before weighing, the material should be well mixed by shaking the sealed container multiple times to ensure that subsequent sub-sampling is representative. They should be stored in a cool, dark, dry environment, with the lid securely sealed.

Safety: Usual precautions apply for handling; material may contain elements or other substances at concentrations potentially harmful to health. Use only with adequate ventilation and/or personal protective equipment. Wash thoroughly after using. Avoid contact with skin and eyes.

Please find enclosed, bottles of seven Candidate Reference Materials and a single container of a QC material.

Candidate RM name	Mass per bottle (approx.)
14511-0001	58 g
Medium	95 g
Dark	95 g
Peaty	95 g
Very Light	95 g
14636-0005	95 g
14636-0006	95 g
QC material	
BGS QC-A	43 g

The number of methods offered by laboratories participating in this exercise was varied, as was the amount of material requested. To maximise return on available material, you are invited to conduct the targeted methods listed in priority order in the table below, using the suggested masses for each method. If you require more material than suggested, please use the mass you need for the higher priority methods at the exclusion of a lower priority method(s).

Complete as many tests as you can. Analyse each Candidate Reference Material in triplicate, using your chosen analytical methods. For the QC material, carry out a single analysis (not in triplicate) for the six tests indicated in the table below.

Conduct all analysis on a dry weight basis.

Priority	Analytical method	Suggested maximum mass of RM to allow for triplicate analysis (g)	Comments	Suggested maximum mass of QC to allow for single analysis (g)
1	pH	30	pH in water	10
2	Loss on ignition	15	Ignition at 450°C	3
3	Total carbon	5	Use preferred method	N/A
4	Organic carbon	5	Use preferred method	3
5	Total nitrogen	10	Use preferred method	3
6	Available nitrogen	50	Use preferred method	N/A
7	Extractable/available phosphorus	20	Use Olsen method if possible, or your preferred method	10
8	Exchangeable/available agronomic cations and anions	30	For as many analytes as possible from your chosen method	10
9	Electrical conductivity	25	Use 1:5 ratio in water	N/A

If you have material remaining, please also determine pH using an alternative extractant (CaCl₂ or KCl), and extractable/available species using your alternative method.

Reporting

- To facilitate subsequent data handling, report the results from each of the triplicate analyses (not the mean) using the Excel template provided by email.
- Report data using one more significant figure than you would use routinely, using the units specified. Include as many exchangeable/available agronomic cations and anions as possible.
- Report by email to inorganicgeochemistry@bgs.ac.uk by **12 February 2021**.
- Please provide a brief description of each analytical method used, e.g. drying temperature, sample mass analysed, volume/concentration of reagents, temperature, time, analytical finish, etc. (a page is included in the Excel template provided by email).

We intend to have recommended values generated by March 2021, and then to publish the results of the round-robin characterisation. With your permission, we will acknowledge your participation in this exercise, but data will not be identifiable to the laboratory that produced it.

Thank you for participating.



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Appendix 2 Data reporting template provided to participating laboratories

Analytical data reporting notes:

- 1 Please enter the name of your laboratory at the top of each tab used.
- 2 Please provide a brief description of each analytical method used in the "Analytical method description" tab.
- 3 Please report data to one more decimal place than you would routinely, using the units specified.
- 4 Data for most of the determinands should be reported using the template in the "Data" tab. The columns for available cations and anions can be found in the "Cation and anion data" tab.
- 5 If you are able to conduct analysis of pH using an alternative extractant, please report data in the columns shaded grey at the right hand end of the "Data" tab.
- 6 If you conduct analysis for available cations and anions using more than one method, please make a second copy of the blank "Cation and anion data" tab for entry of data for the second method.
- 7 Please report by email to inorganicgeochemistry@bgs.ac.uk

Thank you

Candidate RM reporting template

Analytical method description

Laboratory Name:

Method	Mass used (g)	Method description (e.g. volume/concentration of reagents, temperature, time, analytical finish, etc.)
pH in water		
LOI at 450°C		
Total carbon		
Organic carbon		
Total nitrogen		
Available nitrogen		
Available phosphorus		
Available cations and anions 1		
Electrical conductivity		
pH in CaCl ₂		
pH in KCl		
Available cations and anions 2		

Laboratory Name:

Method	pH in water	Loss On Ignition (LOI at 450°C)	Total carbon	Organic carbon	Total nitrogen	Available nitrogen	Available phosphorus	Electrical conductivity	pH in CaCl ₂	pH in KCl
	pH units	wt %	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	µS/cm	pH units	pH units
BGS QC-A			N/A			N/A		N/A		
14511-0001 1										
14511-0001 2										
14511-0001 3										
Medium 1										
Medium 2										
Medium 3										
Dark 1										
Dark 2										
Dark 3										
Peaty 1										
Peaty 2										
Peaty 3										
Very Light 1										
Very Light 2										
Very Light 3										
14636-0005 1										
14636-0005 2										
14636-0005 3										
14636-0006 1										
14636-0006 2										
14636-0006 3										

Laboratory Name:

Method	Available Na mg/kg	Available Mg mg/kg	Available K mg/kg	Available Ca mg/kg	Available B mg/kg	Available fluoride mg/kg	Available Al mg/kg	Available Si mg/kg	Available S mg/kg	Available sulphate mg/kg	Available chloride mg/kg	Available Cr mg/kg	Available Mn mg/kg	Available Fe mg/kg
BGS QC-A														
14511-0001 1														
14511-0001 2														
14511-0001 3														
Medium 1														
Medium 2														
Medium 3														
Dark 1														
Dark 2														
Dark 3														
Peaty 1														
Peaty 2														
Peaty 3														
Very Light 1														
Very Light 2														
Very Light 3														
14636-0005 1														
14636-0005 2														
14636-0005 3														
14636-0006 1														
14636-0006 2														
14636-0006 3														

Laboratory Name:

Method	Available Co mg/kg	Available Ni mg/kg	Available Cu mg/kg	Available Zn mg/kg	Available As mg/kg	Available Se mg/kg	Available Mo mg/kg	Available Cd mg/kg	Available Sb mg/kg	Available Te mg/kg	Available I mg/kg	Available Hg mg/kg	Available Pb mg/kg	Available Bi mg/kg
BGS QC-A														
14511-0001 1														
14511-0001 2														
14511-0001 3														
Medium 1														
Medium 2														
Medium 3														
Dark 1														
Dark 2														
Dark 3														
Peaty 1														
Peaty 2														
Peaty 3														
Very Light 1														
Very Light 2														
Very Light 3														
14636-0005 1														
14636-0005 2														
14636-0005 3														
14636-0006 1														
14636-0006 2														
14636-0006 3														

Appendix 3 Data sheets for BGS120 to BGS126

Reference Soil BGS120

Temperate clay loam soil

Sample information

The British Geological Survey has prepared a reference material (sieved to <2 mm), collected with local partners (Geological Survey Ireland) from agricultural land in the Republic of Ireland.

Particle size distribution was determined by laser diffraction: clay 35%, silt 28%, sand 37%. The textural class for this soil is clay loam¹.

Sample handling

This composite soil was created by amalgamation of a number of individual soil samples, each weighing approximately 500 g. Samples were dried at 30°C, disaggregated, and sieved to <2 mm. The combined materials provided a final mass of 30 kg, which was homogenised by rotation in a 70 L barrel.

A riffle splitter was repeatedly used to separate the homogenised material to achieve eight portions of approximately 3.7 kg, each of which was divided using a rotary splitter into interim portions of 0.47 kg.

Each of the interim portions were further divided using a rotary splitter to create final reference material portions of approximately 58 g. The final portions are provided in 60 mL labelled plastic bottles.

Homogeneity testing

Sample homogeneity testing was carried out using eight bottles of material selected at random. From each randomly selected bottle, two subsamples were analysed for loss-on-ignition (LOI), pH and total P concentration. Samples were analysed using single analytical batches.

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

Where standard deviation was less than or equal to the detection limit (DL), or 0.1 pH units, the homogeneity was deemed acceptable. Where standard deviation >DL, and the RSD ≤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 bottles was not significantly greater ($P \leq 0.05$) than the variation within bottles, 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 analysis of agriculturally relevant soil properties.

Storage

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

¹ <2 µm grain size defined by the pipette method corresponds with a grain size of <8 µm as defined by laser diffraction (Konert and Vandenberghe 1997). Particle size fractions were determined using the 2000-63-2-µm system (FAO 2006).

Reference Soil BGS120

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 materials (BGS120 to BGS126) using their standard procedures for analytes of agricultural interest (Table 1). Additionally, analysis of a quality control (QC) sample was requested to enable the reference material analysis to be benchmarked. Where there exists QC data (Table 1), benchmarking resulted in the exclusion of data falling outside the defined confidence intervals for the QC sample; this affected data supplied by one laboratory for organic carbon analysis. Outlying data were identified where they exceeded two standard deviations from the mean, and excluded from calculation of the Reference Values. For determinands without QC 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 all materials were also excluded; this affected data supplied by one laboratory for total carbon analysis.

The Reference Value (the mean value) 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 any exclusions, 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 parameters set out in Tables 2 and 3.

Table 1: Analytical methods used

Analyte	Method	QC data available	Method code
pH	Soil:water 1:2 to 1:5	Yes	a
	Soil:KCl 1:2.5 to 1:5	No	b
	Soil:CaCl ₂ 1:2 to 1:5	No	c
Electrical conductivity	Soil:water 1:5	No	d
Loss on ignition (LOI)	LOI @ 450°C	Yes	e
Carbon	Total - Dry combustion	No	f
	Organic - Dry combustion	No	g
	Organic - Walkley Black or Tyurin	Yes	h
Nitrogen	Dry combustion	No	i
	Kjeldahl	No	j
Phosphorus	Olsen	Yes	k

Reference Soil BGS120

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 parameter.

Table 2. Reference Values

Parameter	Mean value \pm expanded uncertainty	Units	Method code [§]	Number of data points
pH in water	6.99 \pm 0.28		a	31
pH in KCl	6.55 \pm 0.42		b	15
Electrical conductivity	288 \pm 201	$\mu\text{S cm}^{-1}$	d	18
LOI @ 450°C	8.78 \pm 1.13	wt %	e	21
Total carbon	45500 \pm 2500	mg kg^{-1}	f	15
Total nitrogen - dry	4140 \pm 410	mg kg^{-1}	i	16
Available phosphorus	23.7 \pm 14.3	mg kg^{-1}	k	15

[§] Defined in Table 1.

Table 3. Information Values

Parameter	Mean value	Units	Method code [§]	Number of data points
pH in CaCl ₂	6.64		c	12
Organic carbon - dry	42900	mg kg^{-1}	g	10
Organic carbon - wet	50300	mg kg^{-1}	h	9
Total nitrogen - wet	4160	mg kg^{-1}	j	14

[§] Defined in Table 1.

References

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

Konert, M. and Vandenberghe, J. (1997). Comparison of laser grain size analysis with pipette and sieve analysis: a solution for the underestimation of the clay fraction. *Sedimentology*, 44, 523-535.

Food and Agriculture Organisation of the United Nations (2006). *Guidelines for Soil Description* (fourth edition). FAO, Rome, 97pp

<https://www.fao.org/publications/card/en/c/903943c7-f56a-521a-8d32-459e7e0cdae9/>

Reference Soil BGS121

Temperate sandy clay loam soil

Sample information

The British Geological Survey has prepared a reference material (sieved to <2 mm), collected with local partners (Geological Survey Ireland) from agricultural land in the Republic of Ireland.

Particle size distribution was determined by laser diffraction: clay 25%, silt 26%, and sand 49%. The textural class for this soil is sandy clay loam¹.

Sample handling

This composite soil was created by amalgamation of a number of individual soil samples, each weighing approximately 500 g. Samples were dried at 30°C, disaggregated, and sieved to <2 mm. The combined materials provided a final mass of 80 kg, which was homogenised by rotation in a 290 L barrel.

A riffle splitter was repeatedly used to separate the homogenised material to achieve eight portions of approximately 10 kg, each of which was divided using a rotary splitter into interim portions of 1 kg.

Each of the interim portions were further divided using a rotary splitter to create final reference material portions of approximately 95 g. The final portions are provided in 125 mL labelled plastic bottles.

Homogeneity testing

Sample homogeneity testing was carried out using eight bottles of material selected at random. From each randomly selected bottle, two subsamples were analysed for loss-on-ignition (LOI), pH and total P concentration. Samples were analysed using single analytical batches.

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

Where standard deviation was less than or equal to the detection limit (DL), or 0.1 pH units, the homogeneity was deemed acceptable. Where standard deviation >DL, and the RSD ≤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 bottles was not significantly greater ($P \leq 0.05$) than the variation within bottles, 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 analysis of agriculturally relevant soil properties.

Storage

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

¹ <2 µm grain size defined by the pipette method corresponds with a grain size of <8 µm as defined by laser diffraction (Konert and Vandenberghe 1997). Particle size fractions were determined using the 2000-63-2-µm system (FAO 2006).

Reference Soil BGS121

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 materials (BGS120 to BGS126) using their standard procedures for analytes of agricultural interest (Table 1). Additionally, analysis of a quality control (QC) sample was requested to enable the reference material analysis to be benchmarked. Where there exists QC data (Table 1), benchmarking resulted in the exclusion of data falling outside the defined confidence intervals for the QC sample; this affected data supplied by one laboratory for organic carbon analysis. Outlying data were identified where they exceeded two standard deviations from the mean, and excluded from calculation of the Reference Values. For determinands without QC 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 all materials were also excluded; this affected data supplied by one laboratory for total carbon analysis.

The Reference Value (the mean value) 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 any exclusions, 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 parameters set out in Tables 2 and 3.

Table 1: Analytical methods used

Analyte	Method	QC data available	Method code
pH	Soil:water 1:2 to 1:5	Yes	a
	Soil:KCl 1:2.5 to 1:5	No	b
	Soil:CaCl ₂ 1:2 to 1:5	No	c
Electrical conductivity	Soil:water 1:5	No	d
Loss on ignition (LOI)	LOI @ 450°C	Yes	e
Carbon	Total - Dry combustion	No	f
	Organic - Dry combustion	No	g
	Organic - Walkley Black or Tyurin	Yes	h
Nitrogen	Dry combustion	No	i
	Kjeldahl	No	j
Phosphorus	Olsen	Yes	k

Reference Soil BGS121

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 parameter.

Table 2. Reference Values

Parameter	Mean value \pm expanded uncertainty	Units	Method code [§]	Number of data points
pH in water	6.91 \pm 0.31		a	30
pH in KCl	6.52 \pm 0.51		b	15
Electrical conductivity	412 \pm 239	$\mu\text{S cm}^{-1}$	d	18
LOI @ 450°C	14.9 \pm 1.7	wt %	e	20
Total carbon	85600 \pm 9800	mg kg ⁻¹	f	15
Total nitrogen - dry	6280 \pm 1070	mg kg ⁻¹	i	18
Total nitrogen - wet	6630 \pm 5530	mg kg ⁻¹	j	15
Available phosphorus	24.8 \pm 14.0	mg kg ⁻¹	k	15

[§] Defined in Table 1.

Table 3. Information Values

Parameter	Mean value	Units	Method code [§]	Number of data points
pH in CaCl ₂	6.58		c	11
Organic carbon - dry	70300	mg kg ⁻¹	g	12
Organic carbon - wet	69700	mg kg ⁻¹	h	9

[§] Defined in Table 1.

References

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

Konert, M. and Vandenberghe, J. (1997). Comparison of laser grain size analysis with pipette and sieve analysis: a solution for the underestimation of the clay fraction. *Sedimentology*, 44, 523-535.

Food and Agriculture Organisation of the United Nations (2006). *Guidelines for Soil Description* (fourth edition). FAO, Rome, 97pp

<https://www.fao.org/publications/card/en/c/903943c7-f56a-521a-8d32-459e7e0cdae9/>

Reference Soil BGS122

Temperate sandy loam soil

Sample information

The British Geological Survey has prepared a reference material (sieved to <2 mm), collected with local partners (Geological Survey Ireland) from agricultural land in the Republic of Ireland.

Particle size distribution was determined by laser diffraction: clay 17%, silt 25%, and sand 58%. The textural class for this soil is sandy loam¹.

Sample handling

This composite soil was created by amalgamation of a number of individual soil samples, each weighing approximately 500 g. Samples were dried at 30°C, disaggregated, and sieved to <2 mm. The combined materials provided a final mass of 80 kg, which was homogenised by rotation in a 290 L barrel.

A riffle splitter was repeatedly used to separate the homogenised material to achieve eight portions of approximately 10 kg, each of which was divided using a rotary splitter into interim portions of 1 kg.

Each of the interim portions were further divided using a rotary splitter to create final reference material portions of approximately 95 g. The final portions are provided in 250 mL labelled plastic bottles.

Homogeneity testing

Sample homogeneity testing was carried out using eight bottles of material selected at random. From each randomly selected bottle, two subsamples were analysed for loss-on-ignition (LOI), pH and total P concentration. Samples were analysed using single analytical batches.

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

Where standard deviation was less than or equal to the detection limit (DL), or 0.1 pH units, the homogeneity was deemed acceptable. Where standard deviation >DL, and the RSD ≤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 bottles was not significantly greater ($P \leq 0.05$) than the variation within bottles, 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 analysis of agriculturally relevant soil properties.

Storage

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

¹ <2 µm grain size defined by the pipette method corresponds with a grain size of <8 µm as defined by laser diffraction (Konert and Vandenberghe 1997). Particle size fractions were determined using the 2000-63-2-µm system (FAO 2006).

Reference Soil BGS122

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 materials (BGS120 to BGS126) using their standard procedures for analytes of agricultural interest (Table 1). Additionally, analysis of a quality control (QC) sample was requested to enable the reference material analysis to be benchmarked. Where there exists QC data (Table 1), benchmarking resulted in the exclusion of data falling outside the defined confidence intervals for the QC sample; this affected data supplied by one laboratory for organic carbon analysis. Outlying data were identified where they exceeded two standard deviations from the mean, and excluded from calculation of the Reference Values. For determinands without QC 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 all materials were also excluded; this affected data supplied by one laboratory for total carbon analysis.

The Reference Value (the mean value) 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 any exclusions, 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 parameters set out in Tables 2 and 3.

Table 1: Analytical methods used

Analyte	Method	QC data available	Method code
pH	Soil:water 1:2 to 1:5	Yes	a
	Soil:KCl 1:2.5 to 1:5	No	b
	Soil:CaCl ₂ 1:2 to 1:5	No	c
Electrical conductivity	Soil:water 1:5	No	d
Loss on ignition (LOI)	LOI @ 450°C	Yes	e
Carbon	Total - Dry combustion	No	f
	Organic - Dry combustion	No	g
	Organic - Walkley Black or Tyurin	Yes	h
Nitrogen	Dry combustion	No	i
	Kjeldahl	No	j
Phosphorus	Olsen	Yes	k

Reference Soil BGS122

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 parameter.

Table 2. Reference Values

Parameter	Mean value \pm expanded uncertainty	Units	Method code [§]	Number of data points
pH in water	6.17 \pm 0.38		a	33
pH in KCl	5.80 \pm 0.27		b	15
Electrical conductivity	384 \pm 205	$\mu\text{S cm}^{-1}$	d	16
LOI @ 450°C	26.3 \pm 4.1	wt %	e	19
Total carbon	146000 \pm 25000	mg kg ⁻¹	f	15
Total nitrogen - dry	9070 \pm 1320	mg kg ⁻¹	i	18
Total nitrogen - wet	8070 \pm 2420	mg kg ⁻¹	j	15
Available phosphorus	25.3 \pm 17.0	mg kg ⁻¹	k	15

[§] Defined in Table 1.

Table 3. Information Values

Parameter	Mean value	Units	Method code [§]	Number of data points
pH in CaCl ₂	5.84		c	12
Organic carbon - dry	122000	mg kg ⁻¹	g	12
Organic carbon - wet	123000	mg kg ⁻¹	h	9

[§] Defined in Table 1.

References

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

Konert, M. and Vandenberghe, J. (1997). Comparison of laser grain size analysis with pipette and sieve analysis: a solution for the underestimation of the clay fraction. *Sedimentology*, 44, 523-535.

Food and Agriculture Organisation of the United Nations (2006). *Guidelines for Soil Description* (fourth edition). FAO, Rome, 97pp

<https://www.fao.org/publications/card/en/c/903943c7-f56a-521a-8d32-459e7e0cdae9/>

Temperate organic-rich soil

Sample information

The British Geological Survey has prepared a reference material (sieved to <2 mm), collected with local partners (Geological Survey Ireland) from agricultural land in the Republic of Ireland.

Soil texture classification has not been applied to this material due to its high organic matter content¹.

Sample handling

This composite soil was created by amalgamation of a number of individual soil samples, each weighing approximately 200 g. Samples were dried at 30°C, disaggregated, and sieved to <2 mm. The combined materials provided a final mass of 50 kg, which was homogenised by rotation in a 290 L barrel.

A riffle splitter was repeatedly used to separate the homogenised material to achieve eight portions of approximately 6.2 kg, each of which was divided using a rotary splitter into interim portions of 0.8 kg.

Each of the interim portions were further divided using a rotary splitter to create final reference material portions of approximately 95 g. The final portions are provided in 250 mL labelled plastic bottles.

Homogeneity testing

Sample homogeneity testing was carried out using ten bottles of material selected at random. From each randomly selected bottle, two subsamples were analysed for loss-on-ignition (LOI), pH and total P concentration. Samples were analysed using single analytical batches.

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

Where standard deviation was less than or equal to the detection limit (DL), or 0.1 pH units, the homogeneity was deemed acceptable. Where standard deviation >DL, and the RSD ≤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 bottles was not significantly greater (P≤0.05) than the variation within bottles, 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 analysis of agriculturally relevant soil properties.

Storage

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

¹ <2 µm grain size defined by the pipette method corresponds with a grain size of <8 µm as defined by laser diffraction (Konert and Vandenberghe 1997). Particle size fractions were determined using the 2000-63-2-µm system (FAO 2006).

Reference Soil BGS123

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 materials (BGS120 to BGS126) using their standard procedures for analytes of agricultural interest (Table 1). Additionally, analysis of a quality control (QC) sample was requested to enable the reference material analysis to be benchmarked. Where there exists QC data (Table 1), benchmarking resulted in the exclusion of data falling outside the defined confidence intervals for the QC sample; this affected data supplied by one laboratory for organic carbon analysis. Outlying data were identified where they exceeded two standard deviations from the mean, and excluded from calculation of the Reference Values. For determinands without QC 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 all materials were also excluded; this affected data supplied by one laboratory for total carbon analysis.

The Reference Value (the mean value) 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 any exclusions, 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 parameters set out in Tables 2 and 3.

Table 1: Analytical methods used

Analyte	Method	QC data available	Method code
pH	Soil:water 1:2 to 1:5	Yes	a
	Soil:KCl 1:2.5 to 1:5	No	b
	Soil:CaCl ₂ 1:2 to 1:5	No	c
Electrical conductivity	Soil:water 1:5	No	d
Loss on ignition (LOI)	LOI @ 450°C	Yes	e
Carbon	Total - Dry combustion	No	f
	Organic - Dry combustion	No	g
	Organic - Walkley Black or Tyurin	Yes	h
Nitrogen	Dry combustion	No	i
	Kjeldahl	No	j
Phosphorus	Olsen	Yes	k

Reference Soil BGS123

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 parameter.

Table 2. Reference Values

Parameter	Mean value \pm expanded uncertainty	Units	Method code [§]	Number of data points
pH in water	4.76 \pm 0.22		a	31
Electrical conductivity	420 \pm 123	$\mu\text{S cm}^{-1}$	d	18
LOI @ 450°C	72.7 \pm 7.7	wt %	e	21
Total carbon	394000 \pm 48000	mg kg^{-1}	f	15
Total nitrogen - dry	17900 \pm 2600	mg kg^{-1}	i	18
Available phosphorus	25.3 \pm 8.3	mg kg^{-1}	k	15

[§] Defined in Table 1.

Table 3. Information Values

Parameter	Mean value	Units	Method code [§]	Number of data points
pH in KCl	4.21		b	14
pH in CaCl ₂	4.39		c	12
Organic carbon - dry	344000	mg kg^{-1}	g	11
Organic carbon - wet	242000	mg kg^{-1}	h	9
Total nitrogen - wet	16500	mg kg^{-1}	j	14

[§] Defined in Table 1.

References

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

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Reference Soil BGS124

Temperate clay loam soil

Sample information

The British Geological Survey has prepared a reference material (sieved to <2 mm), collected with local partners (Geological Survey Ireland) from agricultural land in the Republic of Ireland.

Particle size distribution was determined by laser diffraction: clay 33%, silt 30%, and sand 37%. The textural class for this soil is clay loam¹.

Sample handling

This composite soil was created by amalgamation of a number of individual soil samples, each weighing approximately 500 g. Samples were dried at 30°C, disaggregated, and sieved to <2 mm. The combined materials provided a final mass of 80 kg, which was homogenised by rotation in a 290 L barrel.

A riffle splitter was repeatedly used to separate the homogenised material to achieve eight portions of approximately 10 kg, each of which was divided using a rotary splitter into interim portions of 1 kg.

Each of the interim portions were further divided using a rotary splitter to create final reference material portions of approximately 95 g. The final portions are provided in 125 mL labelled plastic bottles.

Homogeneity testing

Sample homogeneity testing was carried out using eight bottles of material selected at random. From each randomly selected bottle, two subsamples were analysed for loss-on-ignition (LOI), pH and total P concentration. Samples were analysed using single analytical batches.

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

Where standard deviation was less than or equal to the detection limit (DL), or 0.1 pH units, the homogeneity was deemed acceptable. Where standard deviation >DL, and the RSD ≤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 bottles was not significantly greater ($P \leq 0.05$) than the variation within bottles, 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 analysis of agriculturally relevant soil properties.

Storage

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

¹ <2 µm grain size defined by the pipette method corresponds with a grain size of <8 µm as defined by laser diffraction (Konert and Vandenberghe 1997). Particle size fractions were determined using the 2000-63-2-µm system (FAO 2006).

Reference Soil BGS124

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 materials (BGS120 to BGS126) using their standard procedures for analytes of agricultural interest (Table 1). Additionally, analysis of a quality control (QC) sample was requested to enable the reference material analysis to be benchmarked. Where there exists QC data (Table 1), benchmarking resulted in the exclusion of data falling outside the defined confidence intervals for the QC sample; this affected data supplied by one laboratory for organic carbon analysis. Outlying data were identified where they exceeded two standard deviations from the mean, and excluded from calculation of the Reference Values. For determinands without QC 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 all materials were also excluded; this affected data supplied by one laboratory for total carbon analysis.

The Reference Value (the mean value) 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 any exclusions, 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 parameters set out in Tables 2 and 3.

Table 1: Analytical methods used

Analyte	Method	QC data available	Method code
pH	Soil:water 1:2 to 1:5	Yes	a
	Soil:KCl 1:2.5 to 1:5	No	b
	Soil:CaCl ₂ 1:2 to 1:5	No	c
Electrical conductivity	Soil:water 1:5	No	d
Loss on ignition (LOI)	LOI @ 450°C	Yes	e
Carbon	Total - Dry combustion	No	f
	Organic - Dry combustion	No	g
	Organic - Walkley Black or Tyurin	Yes	h
Nitrogen	Dry combustion	No	i
	Kjeldahl	No	j
Phosphorus	Olsen	Yes	k

Reference Soil BGS124

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 parameter.

Table 2. Reference Values

Parameter	Mean value \pm expanded uncertainty	Units	Method code [§]	Number of data points
pH in water	7.12 \pm 0.25		a	30
pH in KCl	6.67 \pm 0.49		b	15
Electrical conductivity	276 \pm 152	$\mu\text{S cm}^{-1}$	d	17
LOI @ 450°C	6.10 \pm 0.64	wt %	e	20
Total carbon	29900 \pm 2000	mg kg ⁻¹	f	15
Total nitrogen - dry	2710 \pm 90	mg kg ⁻¹	i	15
Total nitrogen - wet	3080 \pm 3200	mg kg ⁻¹	j	15
Available phosphorus	16.8 \pm 10.2	mg kg ⁻¹	k	15

[§] Defined in Table 1.

Table 3. Information Values

Parameter	Mean value	Units	Method code [§]	Number of data points
pH in CaCl ₂	6.70		c	12
Organic carbon - dry	28200	mg kg ⁻¹	g	11
Organic carbon - wet	30900	mg kg ⁻¹	h	9

[§] Defined in Table 1.

References

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

Konert, M. and Vandenberghe, J. (1997). Comparison of laser grain size analysis with pipette and sieve analysis: a solution for the underestimation of the clay fraction. *Sedimentology*, 44, 523-535.

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<https://www.fao.org/publications/card/en/c/903943c7-f56a-521a-8d32-459e7e0cdae9/>

Reference Soil BGS125

Tropical iron rich clay loam soil

Sample information

The British Geological Survey has prepared a reference material (sieved to <2 mm), collected with local partners (University of Eldoret) from agricultural land in the Republic of Kenya.

Particle size distribution was determined by laser diffraction: clay 29%, silt 27%, sand 44%. The textural class for this soil is clay loam¹.

Sample handling

The bulk sample was sterilised by autoclaving (30 min @121 °C), dried at 30°C, disaggregated, and sieved to <2 mm, giving a final mass of 39 kg.

The sieved 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 four portions of approximately 10 kg, each of which was divided using a rotary splitter into interim portions of 1 kg.

Each of the interim portions were further divided using a rotary splitter to create final reference material portions of approximately 95 g. The final portions are provided in 125 mL labelled plastic bottles.

Homogeneity testing

Sample homogeneity testing was carried out using eight bottles of material selected at random. From each randomly selected bottle, three subsamples were analysed for loss-on-ignition (LOI), and two subsamples for pH and total P concentration. Samples were analysed using single analytical batches.

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

Where standard deviation was less than or equal to the detection limit (DL) or 0.1 pH units, the homogeneity was deemed acceptable. Where standard deviation >DL, and the RSD ≤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 bottles was not significantly greater ($P \leq 0.05$) than the variation within bottles, 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 analysis of agriculturally relevant soil properties.

Storage

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

¹ <2 µm grain size defined by the pipette method corresponds with a grain size of <8 µm as defined by laser diffraction (Konert and Vandenberghe 1997). Particle size fractions were determined using the 2000-63-2-µm system (FAO 2006).

Reference Soil BGS125

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 materials (BGS120 to BGS126) using their standard procedures for analytes of agricultural interest (Table 1). Additionally, analysis of a quality control (QC) sample was requested to enable the reference material analysis to be benchmarked. Where there exists QC data (Table 1), benchmarking resulted in the exclusion of data falling outside the defined confidence intervals for the QC sample; this affected data supplied by one laboratory for organic carbon analysis. Outlying data were identified where they exceeded two standard deviations from the mean, and excluded from calculation of the Reference Values. For determinands without QC 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 all materials were also excluded; this affected data supplied by one laboratory for total carbon analysis.

The Reference Value (the mean value) 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 any exclusions, 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 parameters set out in Tables 2 and 3.

Table 1: Analytical methods used

Analyte	Method	QC data available	Method code
pH	Soil:water 1:2 to 1:5	Yes	a
	Soil:KCl 1:2.5 to 1:5	No	b
	Soil:CaCl ₂ 1:2 to 1:5	No	c
Electrical conductivity	Soil:water 1:5	No	d
Loss on ignition (LOI)	LOI @ 450°C	Yes	e
Carbon	Total - Dry combustion	No	f
	Organic - Dry combustion	No	g
	Organic - Walkley Black or Tyurin	Yes	h
Nitrogen	Dry combustion	No	i
	Kjeldahl	No	j
Phosphorus	Olsen	Yes	k

Reference Soil BGS125

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 parameter.

Table 2. Reference Values

Parameter	Mean value \pm expanded uncertainty	Units	Method code [§]	Number of data points
pH in water	6.98 \pm 0.16		a	31
Electrical conductivity	128 \pm 26	$\mu\text{S cm}^{-1}$	d	17
LOI @ 450°C	5.25 \pm 0.67	wt %	e	20
Total carbon	18300 \pm 900	mg kg^{-1}	f	15
Total nitrogen - dry	1230 \pm 140	mg kg^{-1}	i	18
Available phosphorus	60.8 \pm 31.3	mg kg^{-1}	k	15

[§] Defined in Table 1.

Table 3. Information Values

Parameter	Mean value	Units	Method code [§]	Number of data points
pH in KCl	5.83		b	14
pH in CaCl ₂	6.42		c	11
Organic carbon - dry	18500	mg kg^{-1}	g	12
Organic carbon - wet	20900	mg kg^{-1}	h	9
Total nitrogen - wet	1450	mg kg^{-1}	j	14

[§] Defined in Table 1.

References

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

Konert, M. and Vandenberghe, J. (1997). Comparison of laser grain size analysis with pipette and sieve analysis: a solution for the underestimation of the clay fraction. *Sedimentology*, 44, 523-535.

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<https://www.fao.org/publications/card/en/c/903943c7-f56a-521a-8d32-459e7e0cdae9/>

Reference Soil BGS126

Tropical iron rich clay loam soil

Sample information

The British Geological Survey has prepared a reference material (sieved to <2 mm), collected with local partners (University of Eldoret) from agricultural land in the Republic of Kenya.

Particle size distribution was determined by laser diffraction: clay 32%, silt 26%, sand 42%. The textural class for this soil is clay loam¹.

Sample handling

The bulk sample was sterilised by autoclaving (30 min @121 °C), dried at 30°C, disaggregated, and sieved to <2 mm, giving a final mass of 24 kg.

The sieved material was then combined and homogenised by rotation in a 70 L barrel.

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

Each of the interim portions were further divided using a rotary splitter to create final reference material portions of approximately 95 g. The final portions are provided in 125 mL labelled plastic bottles.

Homogeneity testing

Sample homogeneity testing was carried out using eight bottles of material selected at random. From each randomly selected bottle, three subsamples were analysed for loss-on-ignition (LOI), and two subsamples for pH and total P concentration. Samples were analysed using single analytical batches.

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

Where standard deviation was less than or equal to the detection limit (DL) or 0.1 pH units, the homogeneity was deemed acceptable. Where standard deviation >DL, and the RSD ≤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 bottles was not significantly greater ($P \leq 0.05$) than the variation within bottles, 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 analysis of agriculturally relevant soil properties.

Storage

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

¹ <2 µm grain size defined by the pipette method corresponds with a grain size of <8 µm as defined by laser diffraction (Konert and Vandenberghe 1997). Particle size fractions were determined using the 2000-63-2-µm system (FAO 2006).

Reference Soil BGS126

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 materials (BGS120 to BGS126) using their standard procedures for analytes of agricultural interest (Table 1). Additionally, analysis of a quality control (QC) sample was requested to enable the reference material analysis to be benchmarked. Where there exists QC data (Table 1), benchmarking resulted in the exclusion of data falling outside the defined confidence intervals for the QC sample; this affected data supplied by one laboratory for organic carbon analysis. Outlying data were identified where they exceeded two standard deviations from the mean, and excluded from calculation of the Reference Values. For determinands without QC 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 all materials were also excluded; this affected data supplied by one laboratory for total carbon analysis.

The Reference Value (the mean value) 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 any exclusions, the data are provided as Information Values with only the mean concentration given in Table 3.

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Table 1: Analytical methods used

Analyte	Method	QC data available	Method code
pH	Soil:water 1:2 to 1:5	Yes	a
	Soil:KCl 1:2.5 to 1:5	No	b
	Soil:CaCl ₂ 1:2 to 1:5	No	c
Electrical conductivity	Soil:water 1:5	No	d
Loss on ignition (LOI)	LOI @ 450°C	Yes	e
Carbon	Total - Dry combustion	No	f
	Organic - Dry combustion	No	g
	Organic - Walkley Black or Tyurin	Yes	h
Nitrogen	Dry combustion	No	i
	Kjeldahl	No	j
Phosphorus	Olsen	Yes	k

Reference Soil BGS126

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 parameter.

Table 2. Reference Values

Parameter	Mean value \pm expanded uncertainty	Units	Method code [§]	Number of data points
pH in water	6.44 \pm 0.15		a	32
Electrical conductivity	128 \pm 23	$\mu\text{S cm}^{-1}$	d	18
LOI @ 450°C	9.54 \pm 1.02	wt %	e	21
Total carbon	28800 \pm 1300	mg kg^{-1}	f	15
Total nitrogen - dry	1790 \pm 150	mg kg^{-1}	i	17
Available phosphorus	37.9 \pm 39.0	mg kg^{-1}	k	15

[§] Defined in Table 1.

Table 3. Information Values

Parameter	Mean value	Units	Method code [§]	Number of data points
pH in KCl	5.40		b	14
pH in CaCl_2	5.89		c	11
Organic carbon - dry	29100	mg kg^{-1}	g	11
Organic carbon - wet	26000	mg kg^{-1}	h	9
Total nitrogen - wet	1710	mg kg^{-1}	j	14

[§] Defined in Table 1.

References

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

Konert, M. and Vandenberghe, J. (1997). Comparison of laser grain size analysis with pipette and sieve analysis: a solution for the underestimation of the clay fraction. *Sedimentology*, 44, 523-535.

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