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Tellus Border project

Geochemical Data User Guide

Version 1

Authors:

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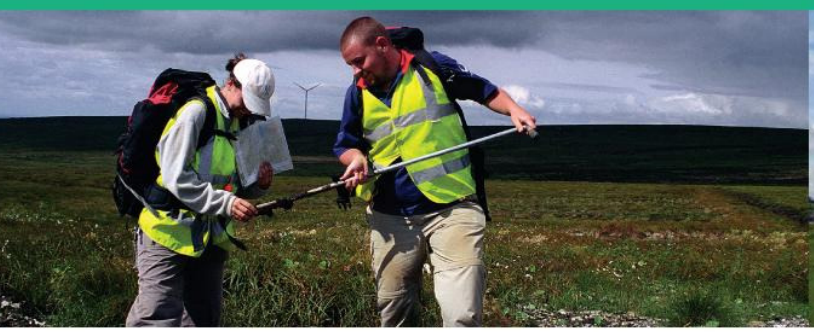

Geological Survey of Ireland and Geological Survey of Northern Ireland

Joint report

2013

Geological Survey of Ireland

The Geological Survey of Ireland is responsible for providing geological advice and information, and for the acquisition of data for this purpose. GSI produces a range of products including maps, reports and databases and acts as a knowledge centre and project partner in all aspects of Irish geology. GSI is a division of the Department of Communications, Energy & Natural Resources (DCENR).

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The Tellus Border project

Tellus Border is a geo-environmental mapping project which provides data on soils, waters and rocks across the border region of Ireland and integrates these with existing data in Northern Ireland. This cross-border collaboration between the Geological Survey of Ireland, the Geological Survey of Northern Ireland and research partners supports the assessment of natural resources, sustainable environmental management and improvement of geological mapping on the island of Ireland. For more information on Tellus Border please see www.tellusborder.eu.

Tellus Border is funded by the INTERREG IVA development programme of the European Regional Development Fund, which is managed by the Special EU Programmes Body (SEUPB). The SEUPB is a North/South Implementation Body sponsored by the Department of Finance and Personnel in Northern Ireland and the Department of Finance in Ireland. It is responsible for managing two EU structural funds Programmes PEACE III and INTERREG IV designed to enhance cross-border co-operation, promote reconciliation and create a more peaceful and prosperous society. For more information on the SEUPB please visit www.seupb.eu.

Tellus Border is additionally part funded by the Department of Environment, Community and Local Government in Ireland and the Department of the Environment in Northern Ireland.

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Document Information

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Executive Summary

This report describes how the Tellus Border geochemical samples were collected, prepared and analysed, describes the format of the results, and details how the data was conditioned before delivery.

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1 Introduction

The Tellus Border geochemical field survey collected samples of soil, stream water and stream sediment from approximately 7,000 locations across the border region in 2011–2012. These samples have been chemically analysed to investigate the baseline or background environmental concentrations of a range of inorganic chemical elements and chemical parameters.

This geochemical survey will help geologists and environmental scientists to better understand how chemical elements are naturally distributed in the surface environment of the northern region of Ireland. The data have a wide range of applications and uses including:

- Helping geologists to characterise the nature of rocks and minerals beneath the Earth's surface;
- Applications for improved land management and agriculture, such as understanding where trace elements and micronutrients may be deficient or excessive;
- Investigating potential ecological, animal and human health risks, such as potentially harmful elements in soil and water;
- Underpinning environmental policy and feeding into improved regulatory frameworks.

The Tellus Border geochemical baseline survey provides a snapshot in time, against which future environmental change and impacts on soil, sediment and water may be measured and mapped.

2 Sample collection

Tellus Border geochemical surveys were conducted by OCAE Consultants Ltd between August 2011 and June 2012 on behalf of the Geological Survey of Ireland (GSI). Reports on the sampling campaigns are given by OCAE Consultants Ltd (2012a and b). Samples of soil, stream sediment, stream water, panned mineral concentrates and vegetation were collected from a region spanning 12,339 km^2 at a typical density of one sample per 4 km^2. In total approximately 7,000 sites were visited over the course of the survey. The sampling methodologies were based on those developed by the British Geological Survey (BGS) G-BASE programme (Johnson, 2005), in order to ensure comparability with data from the Tellus survey of Northern Ireland (Smyth, 2007). The sampling density of the Tellus Border survey was approximately half that of Tellus. Figure 1 and Figure 2 show the sampling locations and densities for the Tellus Border and Tellus soil and drainage (stream) surveys respectively.

Two soil samples are taken at each site with a hand-held Dutch combination auger; the first at *c.*5–20 cm depth (nominally) denoted topsoil, and a second at *c.*35–50 cm depth and denoted subsoil. A composite sampling technique was used whereby five auger flight samples were taken at the corners and centre of a 20m x 20m square, typically from undisturbed land and away from evident point source contamination. Samples were collected into paper bags and a detailed site description of sample and site observational data was recorded and digitally captured to accompany the samples.

Stream water and sediment samples were typically collected from low (first- and second-) order streams. At each site, a wet-sieved sediment sample was sieved through nylon mesh to collect the <150 µm fraction. Additionally the <2 mm sediment fraction was panned for heavy mineral concentrates on-site. Stream water samples were filtered and collected into Nalgene® HDPE sample bottles, and stored in a dark place and kept refrigerated until analyses. Stream water pH, specific electrical conductivity (SEC) and total alkalinity were determined on the day of sample collection. Samples destined for ICP-MS analyses were acidified to 1% v/v with concentrated nitric acid on the day of sample collection in the field, and subsequently additionally acidified with 0.5% v/v concentrated hydrochloric acid prior to analyses.

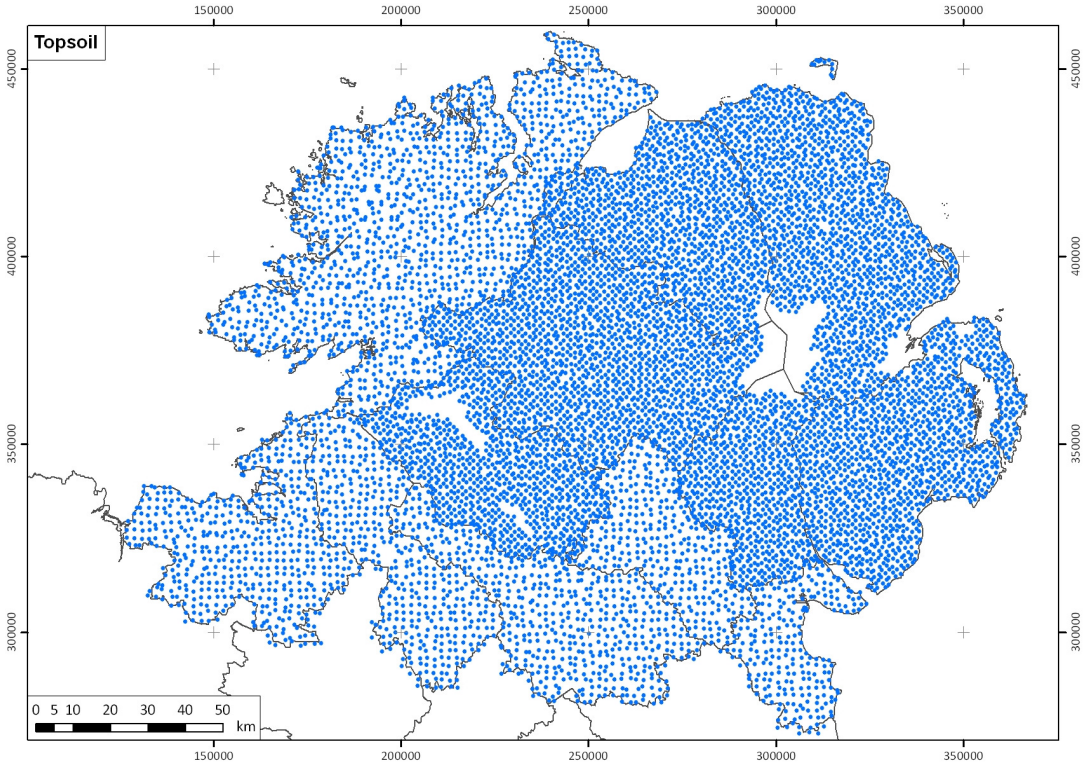

Figure 1. Soil sampling locations and densities for the Tellus Border (Rep. of Ireland) and Tellus (Northern Ireland) surveys. County boundary outlines are shown for reference. Irish National Grid (labelled).

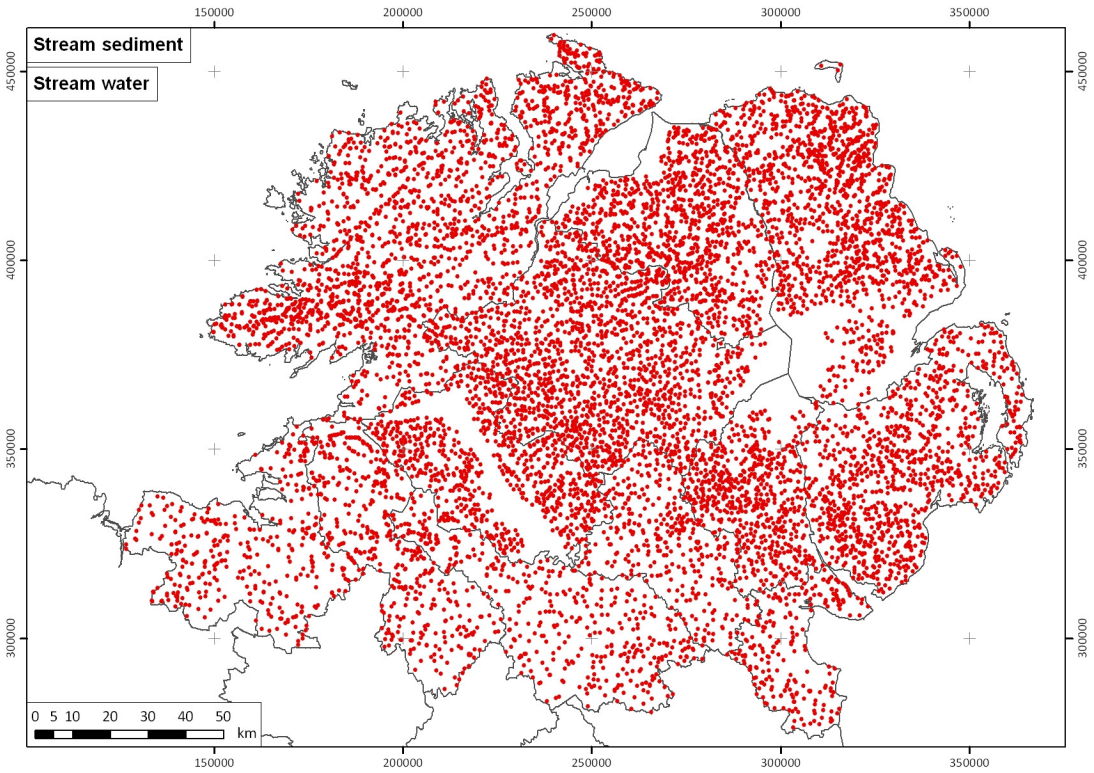

Figure 2. Stream sampling locations and densities for the Tellus Border (Rep. of Ireland) and Tellus (Northern Ireland) surveys. County boundary outlines are shown for reference. Irish National Grid (labelled).

At each stream location a vegetation (woody or twig material) sample was also taken. The vegetation samples, along with the panned mineral concentrates and subsoil samples, have not been routinely chemically analysed and have been archived at the Geological Survey of Ireland. The sample archive (of prepared and unprepared materials) is available on an application basis for research purposes and proposals for additional investigations.

3 Sample preparation and analysis

A summary of the chemical element determinands and analytical methods used in the Tellus Border project is shown in Table 1.

Sample type collected	Analytical method	Chemical determinands*
Stream sediment	X-ray fluorescence spectrometry (XRFS)	K, Ca, Ti, Mn, Fe, S, Cl, Sc, V, Cr, Co, Ni, Cu, Zn, Ga, Ge, As, Se, Br, Rb, Sr, Zr, Nb, Mo, Nd, Sm, Yb, Hf, Ta, W, Tl, Pb, Bi, Th, U, Ag, Cd, In, Sn, Sb, Te, I, Cs, Ba, La, Ce, Na, Mg, Al, Si, P, Ba, Y.
Stream water	Stream water pH, electrical conductivity, alkalinity (bicarbonate); organic carbon	NPOC; Cl^-, SO_4^{2-}, NO_3^-, Br^-, NO_2^-, HPO_4^{2-}, F^-; Li, Be, B, Na, Mg, Al, Si, P, S, K, Ca, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Ga, As, Se, Rb, Sr, Y, Zr, Nb, Ag,

	(NPOC) analyser; ion chromatography; ICP-MS	Cd, Sn, Sb, Cs, Ba, La, Ce, Pr, Nd, Sm, Eu, Tb, Tm, Yb, Lu, Hf, Ta, W, Tl, Pb, Bi, Th, U, Ti, Mo, Gd, Dy, Ho, Er.
Topsoil (*c*.5–20 cm deep)	ICP(-OES/-MS) following *aqua regia* digestion; soil pH, soil loss-on-ignition at 450°C	Al, B, Ba, Ca, Cr, Cu, Fe, K, Li, Mg, Mn, Na, Ni, P, S, Sr, Ti, V, Zn, Zr, Ag, As, Be, Bi, Cd, Ce, Co, Cs, Ga, Ge, Hf, Hg, In, La, Lu, Mo, Nb, Pb, Rb, Sb, Sc, Se, Sn, Ta, Tb, Te, Th, Tl, U, W, Y, Yb.

*Not all samples have data for all determinands, depending on the analytical capabilities and nature of the samples.

Table 1. List of chemical determinands and analytical methods for each sample medium.

3.1 Stream sediment

Approximately 3600 stream sediment samples were air dried on site/at GSI and then prepared for analyses. These works were contracted to the Sample Preparation Facility at the British Geological Survey, Keyworth, Nottingham, UK. Samples were typically frozen and freeze-dried and disaggregated using a non-metallic pestle and mortar, before agate planetary ball milling until at least 95% of material was milled to <53 µm. Milled samples were additionally milled with a wax binder until 95% of the sample was nominally <30 µm and prepared into pressed powder pellets for X-ray fluorescence spectrometry (XRFS) analyses, subcontracted to the PanAlytical laboratory based in Keyworth, Nottingham, UK. The XRFS analyses involved a multi-element sequence across wavelength dispersive (XRF-WD) and energy dispersive (XRF-ED) instruments. The list of chemical determinands and lower limits of detection for stream sediment XRFS analysis are given in Table 2. Note that major elements are typically expressed as weight % major oxide equivalents, but XRFS analyses are for the total element concentrations of all analytes.

Instrument Type	Analyte	Unit	Method LLD
XRFWD	K_2O	weight % equivalent	0.1
XRFWD	CaO	weight % equivalent	0.04
XRFWD	TiO_2	weight % equivalent	0.01
XRFWD	MnO	weight % equivalent	0.01
XRFWD	Fe_2O_3	weight % equivalent	0.01
XRFWD	S	mg kg^{-1}	0.1
XRFWD	Cl	mg kg^{-1}	0.02
XRFWD	Sc	mg kg^{-1}	3
XRFWD	V	mg kg^{-1}	3
XRFWD	Cr	mg kg^{-1}	3
XRFWD	Co	mg kg^{-1}	1.5
XRFWD	Ni	mg kg^{-1}	1.3
XRFWD	Cu	mg kg^{-1}	1.3
XRFWD	Zn	mg kg^{-1}	1.3
XRFWD	Ga	mg kg^{-1}	1

XRFWD	Ge	mg kg^{-1}	0.5
XRFWD	As	mg kg^{-1}	0.9
XRFWD	Se	mg kg^{-1}	0.2
XRFWD	Br	mg kg^{-1}	0.8
XRFWD	Rb	mg kg^{-1}	1
XRFWD	Sr	mg kg^{-1}	1
XRFWD	Y	mg kg^{-1}	1
XRFWD	Zr	mg kg^{-1}	1
XRFWD	Nb	mg kg^{-1}	1
XRFWD	Mo	mg kg^{-1}	0.2
XRFWD	Nd	mg kg^{-1}	4
XRFWD	Sm	mg kg^{-1}	3
XRFWD	Yb	mg kg^{-1}	1.5
XRFWD	Hf	mg kg^{-1}	1
XRFWD	Ta	mg kg^{-1}	1
XRFWD	W	mg kg^{-1}	0.6
XRFWD	Tl	mg kg^{-1}	0.5
XRFWD	Pb	mg kg^{-1}	1.3
XRFWD	Bi	mg kg^{-1}	0.3
XRFWD	Th	mg kg^{-1}	0.7
XRFWD	U	mg kg^{-1}	0.5
XRFED	Ag	mg kg^{-1}	0.5
XRFED	Cd	mg kg^{-1}	0.5
XRFED	In	mg kg^{-1}	0.5
XRFED	Sn	mg kg^{-1}	0.5
XRFED	Sb	mg kg^{-1}	0.5
XRFED	Te	mg kg^{-1}	0.5
XRFED	I	mg kg^{-1}	0.5
XRFED	Cs	mg kg^{-1}	1
XRFED	Ba	mg kg^{-1}	1
XRFED	La	mg kg^{-1}	1
XRFED	Ce	mg kg^{-1}	1
XRFWD	Na_2O	weight % equivalent	0.3
XRFWD	MgO	weight % equivalent	0.2
XRFWD	Al_2O_3	weight % equivalent	0.2
XRFWD	SiO_2	weight % equivalent	0.1
XRFWD	P_2O_5	weight % equivalent	0.03
XRFWD	SO_3	weight % equivalent	0.1
XRFWD	CaO	weight % equivalent	0.04
XRFWD	TiO_2	weight % equivalent	0.01
XRFWD	Fe_2O_3	weight % equivalent	0.01
XRFWD	Ba	weight % equivalent	1
XRFWD	Cl	weight % equivalent	0.02

Table 2. Chemical determinands and lower limits of detection (LLD) for stream sediment analyses by XRFS.

3.2 Stream water

Stream water samples were collected from approximately 3500 sites. Samples destined for chemical analyses were filtered at the point of collection to <0.45 µm. Multi-element, anions and non-purgeable organic carbon (NPOC) analyses were undertaken by ICP-MS, Ion Chromatography (IC) and Total Inorganic/Organic Carbon (TIC/TOC) analyser methods respectively by the Inorganic Analytical Aqueous Laboratories at the British Geological Survey, Keyworth, Nottingham, UK. Sample pH, conductivity and total alkalinity were also determined in the field. The list of chemical determinands and lower limits of detection for waters ICP, IC and NPOC analyses are given in Table 3.

Instrument Type	Analyte	Unit	Method LLD
TIC/TOC analyser	NPOC	mg L^{-1}	0.5
IC	Cl^-	mg L^{-1}	0.05
IC	SO_4^{2-}	mg L^{-1}	0.1
IC	NO_3^-	mg L^{-1}	0.05
IC	Br^-	mg L^{-1}	0.02
IC	NO_2^-	mg L^{-1}	0.01
IC	HPO_4^{2-}	mg L^{-1}	0.1
IC	F^-	mg L^{-1}	0.01
ICP-MS	Li	µg L^{-1}	1
ICP-MS	Be	µg L^{-1}	0.01
ICP-MS	B	µg L^{-1}	10
ICP-MS	Na	mg L^{-1}	0.2
ICP-MS	Mg	mg L^{-1}	0.01
ICP-MS	Al	µg L^{-1}	1
ICP-MS	Si	µg L^{-1}	50
ICP-MS	P	mg L^{-1}	0.01
ICP-MS	S	mg L^{-1}	1
ICP-MS	K	mg L^{-1}	0.02
ICP-MS	Ca	mg L^{-1}	0.3
ICP-MS	Ti	µg L^{-1}	0.05
ICP-MS	V	µg L^{-1}	0.1
ICP-MS	Cr	µg L^{-1}	0.05
ICP-MS	Mn	µg L^{-1}	0.2
ICP-MS	Fe	µg L^{-1}	1
ICP-MS	Co	µg L^{-1}	0.01
ICP-MS	Ni	µg L^{-1}	0.1
ICP-MS	Cu	µg L^{-1}	0.4
ICP-MS	Zn	µg L^{-1}	0.5
ICP-MS	Ga	µg L^{-1}	0.03
ICP-MS	As	µg L^{-1}	0.02
ICP-MS	Se	µg L^{-1}	0.1
ICP-MS	Rb	µg L^{-1}	0.01

ICP-MS	Sr	µg L^{-1}	0.1
ICP-MS	Y	µg L^{-1}	0.005
ICP-MS	Zr	µg L^{-1}	0.05
ICP-MS	Nb	µg L^{-1}	0.02
ICP-MS	Mo	µg L^{-1}	0.03
ICP-MS	Ag	µg L^{-1}	0.05
ICP-MS	Cd	µg L^{-1}	0.01
ICP-MS	Sn	µg L^{-1}	0.02
ICP-MS	Sb	µg L^{-1}	0.005
ICP-MS	Cs	µg L^{-1}	0.005
ICP-MS	Ba	µg L^{-1}	0.1
ICP-MS	La	µg L^{-1}	0.002
ICP-MS	Ce	µg L^{-1}	0.002
ICP-MS	Pr	µg L^{-1}	0.002
ICP-MS	Nd	µg L^{-1}	0.01
ICP-MS	Sm	µg L^{-1}	0.002
ICP-MS	Eu	µg L^{-1}	0.002
ICP-MS	Tb	µg L^{-1}	0.002
ICP-MS	Gd	µg L^{-1}	0.002
ICP-MS	Dy	µg L^{-1}	0.002
ICP-MS	Ho	µg L^{-1}	0.002
ICP-MS	Er	µg L^{-1}	0.002
ICP-MS	Tm	µg L^{-1}	0.002
ICP-MS	Yb	µg L^{-1}	0.002
ICP-MS	Lu	µg L^{-1}	0.002
ICP-MS	Hf	µg L^{-1}	0.01
ICP-MS	Ta	µg L^{-1}	0.02
ICP-MS	W	µg L^{-1}	0.05
ICP-MS	Tl	µg L^{-1}	0.01
ICP-MS	Pb	µg L^{-1}	0.02
ICP-MS	Bi	µg L^{-1}	n/a
ICP-MS	Th	µg L^{-1}	0.005
ICP-MS	U	µg L^{-1}	0.002
pH meter	pH	pH unit	-
Conductivity meter	SEC	µS cm^{-1}	-
Titration against H_2SO_4	HCO_3^-	mg L^{-1} HCO_3^- equivalent	

Table 3. Chemical determinands and lower limits of detection (LLD) for water ICP-MS, IC and NPOC analyses.

3.3 Topsoil

Following air drying (at maximum 35°C) approximately 3600 topsoil samples were prepared at the Sample Preparation Facility at the British Geological Survey, Keyworth, Nottingham, UK. Samples were first disaggregated using a non-metallic pestle and mortar before passing through nylon mesh to <2 mm, discarding lithic fragments. The <2mm fraction samples were agate ball milled until at least 95% of material

was milled to <53 µm. Prepared samples were shipped to SGS Laboratories in Toronto, Canada for Inductively Coupled Plasma (ICP), pH and Loss on Ignition (LOI) analyses. Note that soil pH was determined on the <2 mm sample fraction.

Multi-element ICP analyses (SGS method code ICM11D) were conducted on a 1 g milled sub-sample following an *aqua regia* two-acid sample digestion (2:1 HNO_3:HCl) extraction. ICP analysis was undertaken with optical emission spectrometry (ICP-OES) and mass spectrometry (ICP-MS) instruments. Soil pH (SGS method code ISE15V) was conducted on a 5 g subsample by a $CaCl_2$ slurry method with calibrated pH electrode. Soil LOI (SGS method code PHY01D) was conducted on a 1 g subsample, calculating the proportion of mass lost by ignition at 450℃. The list of chemical determinands and lower limits of detection for soil ICP, pH and LOI analyses are given in Table 4.

Instrument Type	Analyte	Unit	Method LLD
ICP-OES	Al	weight %	0.01
ICP-OES	B	mg kg^{-1}	10
ICP-OES	Ba	mg kg^{-1}	5
ICP-OES	Ca	weight %	0.01
ICP-OES	Cr	mg kg^{-1}	1
ICP-OES	Cu	mg kg^{-1}	0.5
ICP-OES	Fe	weight %	0.01
ICP-OES	K	weight %	0.01
ICP-OES	Li	mg kg^{-1}	1
ICP-OES	Mg	mg kg^{-1}	0.01
ICP-OES	Mn	mg kg^{-1}	2
ICP-OES	Na	weight %	0.01
ICP-OES	Ni	mg kg^{-1}	0.5
ICP-OES	P	mg kg^{-1}	50
ICP-OES	S	weight %	0.01
ICP-OES	Sr	mg kg^{-1}	0.5
ICP-OES	Ti	weight %	0.01
ICP-OES	V	mg kg^{-1}	1
ICP-OES	Zn	mg kg^{-1}	1
ICP-OES	Zr	mg kg^{-1}	0.5
ICP-MS	Ag	mg kg^{-1}	0.01
ICP-MS	As	mg kg^{-1}	1
ICP-MS	Be	mg kg^{-1}	0.1
ICP-MS	Bi	mg kg^{-1}	0.02
ICP-MS	Cd	mg kg^{-1}	0.01
ICP-MS	Ce	mg kg^{-1}	0.05
ICP-MS	Co	mg kg^{-1}	0.1
ICP-MS	Cs	mg kg^{-1}	0.05
ICP-MS	Ga	mg kg^{-1}	0.1

ICP-MS	Ge	mg kg^{-1}	0.1
ICP-MS	Hf	mg kg^{-1}	0.05
ICP-MS	Hg	mg kg^{-1}	0.01
ICP-MS	In	mg kg^{-1}	0.02
ICP-MS	La	mg kg^{-1}	0.1
ICP-MS	Lu	mg kg^{-1}	0.01
ICP-MS	Mo	mg kg^{-1}	0.05
ICP-MS	Nb	mg kg^{-1}	0.05
ICP-MS	Pb	mg kg^{-1}	0.2
ICP-MS	Rb	mg kg^{-1}	0.2
ICP-MS	Sb	mg kg^{-1}	0.05
ICP-MS	Sc	mg kg^{-1}	0.1
ICP-MS	Se	mg kg^{-1}	1
ICP-MS	Sn	mg kg^{-1}	0.3
ICP-MS	Ta	mg kg^{-1}	0.05
ICP-MS	Tb	mg kg^{-1}	0.02
ICP-MS	Te	mg kg^{-1}	0.05
ICP-MS	Th	mg kg^{-1}	0.1
ICP-MS	Tl	mg kg^{-1}	0.02
ICP-MS	U	mg kg^{-1}	0.05
ICP-MS	W	mg kg^{-1}	0.1
ICP-MS	Y	mg kg^{-1}	0.05
ICP-MS	Yb	mg kg^{-1}	0.1
Loss-on-ignition	-	%	0.01
pH	-	pH unit	-

Table 4. Chemical determinands and lower limits of detection (LLD) for topsoil by ICP, pH and LOI analyses.

4 Data conditioning

Samples submitted to the analytical laboratories included a range of quality control (QC) samples in order to assess the performance of the analyses and to determine that the data were 'fit-for-purpose'. The range of control samples include duplicate and replicate samples to assess the proportions of sampling, analytical and spatial variability in the datasets; certified and secondary reference materials that are submitted blind to the analytical facilities, and blank and calibration samples that are used for calibration and instrument QA/QC by the analysts. These control samples are removed from the final datasets released for general use.

A range of quality control samples are used are used to check for a range of potential issues including but not limited to sample and sampling reproducibility; sampling, preparation and analytical bias; analytical drift and carry-over; and potential contamination arising from the sampling, handling, preparation and analytical processes. A range of certified and secondary reference materials were inserted into analytical

batches at a rate of approximately 7% and analysed along with the water, sediment and soil samples to assess analytical precision and accuracy. In general, comprehensive QA/QC procedures are undertaken prior to any data releases. Merging datasets that span a significant period of time, for example the geochemical data from the Republic of Ireland with that from Northern Ireland, adds additional complexity and data are assessed to ensure that they are conditioned, or levelled, as appropriate.

5 Data availability and licensing information

Conditioned and quality-controlled analytical datasets shall be made freely availably. Please see www.tellusborder.eu for updates on future data releases. Documented accounts of the quality control procedures and guidance on the data shall be published to accompany the data releases.

It is anticipated that datasets shall be made available in .csv and/or .xls file formats, plus a selection of interpolated mapped images for use in Geographical Information Systems software.

Tellus Border geochemical data is available to view and will shortly be available for download from the Tellus Border online viewer at www.tellusborder.eu. The data and viewer were developed by the Department of Communications, Energy and Natural Resources (DCENR).

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