



# Options for harmonising soil data obtained from different sources

ISRIC Report 2023/02

Niels H. Batjes



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Director, ISRIC - World Soil Information  
Droevendaalsesteeg 3 (building 101)  
6708 PB Wageningen  
The Netherlands  
E-mail: [soils@isric.org](mailto:soils@isric.org)

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## Summary

This review was prepared in conjunction with the EU HoliSoils project (Grant agreement No. 101000289) and ISRIC's ongoing WoSIS (World Information Service) programme. It investigates possibilities for harmonisation of discongruent soil data obtained from a diverse range of data providers. Various approaches, expressed in terms of tier levels, towards full harmonisation are discussed. The actual choice for, or feasibility of, a given approach is largely determined by the fact whether new soil monitoring programmes can be set up or whether only soil legacy data are available. These considerations determine the level of harmonisation of soil data achievable in the framework of the World Soil Information Service. WoSIS-derived data will underpin the digital soil mapping work of the HoliSoils project, with a focus on forest soils in Europe.

## 1. Introduction

Serious problems can arise with the comparison of soil analytical data derived from different sources. Many countries use specific analytical methods for their soil analyses, be it for agricultural soils or forest soils, and this generally according to the prevailing national standards (Higgins *et al.* 2021). In some instances different standards are used within countries, for example for 'states' (Höhle *et al.* 2016; Hoffmann *et al.* 2019). These 'original' data often meet specific goals and are not necessarily intended to contribute to international transboundary studies. Standardisation and harmonisation of such data for wider use may imply a loss of appropriateness for originally intended purposes (Batjes *et al.* 2017). However, once they are compiled under a common standard they importantly gain in appropriateness for use for cross border or international applications.

That being said, the quality and possible extent of the research and policy advice that can be based on such standardised data will strongly depend on the quality and comparability of the available (source) data (Van Egmond and Fantappiè 2021). Many projects and initiatives for Europe have underlined the difficulties that arise when comparing and sharing data from national soil monitoring systems (Bispo *et al.* 2021).

This report discusses the scope for harmonisation of soil data set, in the context of the EU-HoliSoils project and related mapping activities. As we are dealing with existing data, the possible approach to harmonisation will be largely driven by the actual availability and accessibility of datasets, in particular results from inter-laboratory comparisons throughout Europe. Another important factor in this respect is that 'soil monitoring for Europe is largely fragmented and under-resourced' (Morvan *et al.* 2008; Arrouays *et al.* 2012; Arrouays *et al.* 2021). Saby *et al.* (2008), for example, raised the important issue of whether 'European soil-monitoring networks [will] be able to detect changes in topsoil organic carbon content', which is an important consideration for the EU HoliSoils project.

## 2. Facing incongruent source data

Soil data for the HoliSoils mapping activities are derived from databases collated by the project itself (Wellbrock *et al.* 2021) as well as a large compilation of soil profile data managed in WoSIS (Batjes *et al.* 2020) complemented with recent acquisitions such as the LUCAS 2018 dataset (Fernandez-Ugalde *et al.* 2022).

For HoliSoils, all submitted datasets, pre-screened for completeness (e.g., lineage, license), are imported 'as is' in the ISRIC Data Repository keeping the original data model, naming and coding conventions, abbreviations, domains respectively units of measurement, and so on. Subsequently, these diverse 'source' datasets are converted into PostgreSQL format using a semi-automated ETL (extraction, transformation, load) procedure. During this process, the source data are 'mapped' to the WoSIS naming conventions, standard values and/or units of measurement (Ribeiro *et al.* 2020). This corresponds with the first major step of standardisation: make the originally incongruent data queryable and usable. The second step of standardisation involves importing the standardised data into the WoSIS data model itself; this process includes automated checks on the plausibility of the reported values (e.g., pH H<sub>2</sub>O should be between 1 and 13).

A desired third step, as a follow up to the standardisation, full data harmonisation would involve making similar soil chemical and physical data comparable, that is as 'if assessed by a commonly endorsed single reference method' (for pH, CEC, organic carbon, etc.). Worldwide, these reference (or target) methods still have to be agreed upon by the international soil science community, for example in the framework of the Global Soil Partnership (Baritz *et al.* 2014). Presently, GLOSOLAN (2020) is developing new Standard Operation Procedures (SOP) for wider application, yet these need not align with ISO/TC 190 (2016) standards.

Alternatively for Europe-wide soil monitoring, with a focus on agricultural soils, the list of analytical methods as implemented for LUCAS (Orgiazzi *et al.* 2018) are recommended (Bispo *et al.* 2021). Alternatively, EU-HoliSoils adopted the list of standard analytical methods described in ICP Forests (2021a). Both LUCAS and ICP Forests follow the same ISO standards for soil analyses, albeit with minor modifications. However, procedures for field sampling can vary substantially for example when sampling organic topsoils.

During the initial phase of the HoliSoils project and 2<sup>nd</sup> General Assembly (Helsinki, June 2022), the following soil properties were identified as being necessary for the subsequent mapping work, hence harmonisation (with consideration of organic versus mineral soil layers): bulk density, Carbon (Total and Organic), Calcium carbonate, Cation exchange capacity, Total Nitrogen, pH (H<sub>2</sub>O), Texture (sand, silt, clay), and possibly water retention (at specified tensions). The general principles and procedures outlined in Section 3 can be used for harmonising other soil properties as well.

### 3. Possible approaches to harmonisation

When starting a survey or monitoring programme from 'scratch' there is scope for *a priori* harmonisation of sampling design, field sampling, sample (pre-)treatment as well as the analytical analyses themselves. Conversely, when faced with existing laboratory data only *a posteriori* harmonisation may be considered. Many projects and initiatives for Europe have underlined the difficulties that arise when comparing and sharing data from national soil monitoring systems. This may be due to technical issues (e.g., sampling designs and protocols, analytical methods, data formats) and reasons for sharing (e.g., why share the data and for what purpose?) as well as legal requirements (Bispo *et al.* 2021).

#### 3.1 A priori harmonisation

In recent ICP Forests (ICPF) monitoring rounds, all soil properties were sampled and analysed according to the standards described in ICP Forests (2021b). Apparently, however, during the initial rounds individual countries still followed their own national standards, in a range of laboratories. These data

were later harmonised to the ICPF reference methods (Cools *et al.* 2004); the actual procedures used for this, however, are not easily traced (De Vos *et al.* 2015).

The past two decades, harmonised forest soil monitoring is applied across Europe (Fleck *et al.* 2016); all laboratories have to use the same reference methods (see Appendix), which follow ISO standards (ICP Forests 2021b). Apparently, no single reference laboratory is used at present yet round-robin rounds for between (and within) laboratory data inter-comparability are in place. Such procedures are essential in order to ensure the quality of soil chemical data can be assessed (Cynthia van Leeuwen *et al.* 2022). Alternatively, monitoring programmes such as LUCAS use ‘a common sampling procedure, single laboratory, and standard analytical methods’ in all rounds (Fernandez-Ugalde *et al.* 2022).

Use of uniform procedures over space and time is critical to reduce error sources and avoid the need for *a posteriori* harmonisation as required in the case of incongruent (national) soil datasets (Crawford *et al.* 2018; Bispo *et al.* 2021).

## 3.2 A posteriori harmonisation

As indicated, for the HoliSoils mapping component, we are faced with forest soil data from a wide range of sources. Early work at ISRIC by Vogel (1994) on the conversion (or harmonisation) of results determined by different analytical methods indicated that this can be accomplished in two ways. First by adding or subtracting the average difference between methods, see for example Höhle (2016, p. 385), or by using regionally-calibrated and validated regression equations or pedotransfer functions (PTF). In principle, the PTF-approach is considered more accurate, but development of regionally-applicable PTFs requires the availability of adequate ‘comparative’ input data sets for PTF-development (calibration) as well as their subsequent validation, and this for all soil properties under consideration (see Appendix).

### 3.2.1 Applying pedotransfer functions

#### *Development of pedotransfer functions*

The GlobalSoilMap (2015) consortium and others (e.g., Styc *et al.* 2021) indicated that the necessary pedotransfer functions are likely to be region and soil type specific. Probably, here again, the biggest challenge (besides the actual sharing of soil data) is the quality of the data used to build the regional transfer functions. Building a transfer function to describe the relationship between or among properties generally is not the main issue; when sufficient data are available it may be attractive to develop machine learning models (Tóth *et al.* 2014; Khaledian *et al.* 2018; Laura Poggio *et al.* 2021; Schillaci *et al.* 2021).

In practice, proficiency testing initiatives have revealed unacceptably variable control charts (for definition see e.g., van Reeuwijk and Houba 1998; ICP Forests 2021b), suggesting varying methods for the same *named* analysis across laboratories and/or varying quality control measures. Therefore, until

analytical methods and quality control are standardised (and enforced), errors of transfer functions built on variously sourced data of varying quality can be expected to be large. In principle, such issues can only be avoided when all soil data for a given region are collected and analysed using defined standard methods in a single reference laboratory as is the case with the EU-LUCAS topsoil programme (Orgiazzi *et al.* 2018; Fernandez-Ugalde *et al.* 2022), EU-Soils4Africa project (Paterson *et al.* 2020) and the US-NCSS soil database (USDA-NCSS 2018; Soil Survey Staff 2022).

Examples of PTFs for converting pH-results obtained using different methods are listed in Table 1 as example. Generally, such PTFs are not ‘portable’ or transferrable from one region to another (GlobalSoilMap 2015; Kabała *et al.* 2016; Santra *et al.* 2018; Sevastas *et al.* 2018; Hu *et al.* 2021; Styc *et al.* 2021). It should be noted that the original PTFs were often developed from single determinations lacking data on the precision of either method considered in the comparison (Crawford *et al.* 2018). Further, use of a PTF will introduce additional error (i.e., PTF-uncertainty) to the available (measured) data (Kotlar *et al.* 2019) and effects of such PTF-errors are not readily quantified (Van Looy *et al.* 2017; Cynthia van Leeuwen *et al.* 2022). Additional PTF-uncertainty is introduced when several PTFs are used in sequence to go from one source method X (e.g., pH<sub>H2O</sub> 1:2.5) via a PTF to pH<sub>H2O</sub> 1:5 first, and then use this value as input to a second PTF to arrive at the defined target method Y (e.g, pH<sub>CaCl2</sub>, 1:2.5 0.01 M) as done by Styc *et al.* (2021) in an example based on the GlobalSoilMap methodology.

Table 1. Example of PTFs for harmonising pH values obtained with different methods<sup>a</sup>

No.	Target Method (Y)	Source Method (X)	Equation	R <sup>2</sup>	Reference
1	pH (1:1 0.01 m CaCl2)	pH (1:1 water)	$y = 1.08(x) - 0.973$	0.98	Miller and Kissel (2010)
2	pH (1:1 0.01 m CaCl2)	pH (saturated paste)	$y = 1.10 (x) - 0.923$	0.98	Miller and Kissel (2010)
3	pH (1:1 0.01 m CaCl2)	pH (1:2 water)	$y = 1.05 (x) - 0.950$	0.97	Miller and Kissel (2010)
4	pH (1:1 water)	pH (1:1 0.01 m CaCl2)	$y = x + 0.267 (EC\ 1:1\ water)^{-0.445}$	0.99	Miller and Kissel (2010)
5	pH (1:2 water)	pH (1:1 0.01 m CaCl2)	$y = x + 0.239 (EC\ 1:1\ water)^{-0.505}$	0.98	Miller and Kissel (2010)
6	pH (1:5 0.01 m CaCl2)	pH (1:5 water)	$y = 1.012 (x) - 0.76$	0.99	Conyers and Davey (1988)
7	pH (1:5 0.01 m CaCl2)	pH (1:5 water)	$y = 0.979 (x) - 0.71$	0.68	Bruce <i>et al.</i> (1989)
8	pH (1:5 0.01 m CaCl2)	pH (1:5 water)	$y = 0.887 (x) - 0.199$	0.88	Aitken and Moody (1991)
9	pH (1:5 0.01 m CaCl2)	pH (1:5 water)	$y = 0.197 (x)^2 - 1.21 (x) + 5.78$	0.92	Aitken and Moody (1991)
10	pH (1:5 0.002 m CaCl2)	pH (1:5 water)	$y = 0.948 (x) - 0.308$	0.90	Aitken and Moody (1991)
11	pH (1:5 0.002 m CaCl2)	pH (1:5 water)	$y = 0.178 (x)^2 - 1.043 (x) + 5.10$	0.94	Aitken and Moody (1991)
12	pH (1:5 1 m KCl)	pH (1:5 water)	$y = 0.803 (x) + 0.077$	0.81	Aitken and Moody (1991)
13	pH (1:5 1 m KCl)	pH (1:5 water)	$y = 0.233 (x)^2 - 1.797 (x) + 7.143$	0.98	Aitken and Moody (1991)
14	pH (soil solution)	pH (1:5 water)	$y = 1.28 (x) - 0.613$	0.78	Aitken and Moody (1991)
15	pH (soil solution)	pH (1:5 0.01 m CaCl2)	$y = 1.105 (x) - 0.140$	0.79	Aitken and Moody (1991)
16	pH (soil solution)	pH (1:5 0.002 m CaCl2)	$y = 1.050 (x) - 0.112$	0.80	Aitken and Moody (1991)
17	pH (soil solution)	pH (1:5 1 m KCl)	$y = 1.175 (x) - 0.262$	0.80	Aitken and Moody (1991)

<sup>a</sup> Credit: For references see ‘Specifications Tiered<sup>1</sup> GlobalSoilMap products’ (GlobalSoilMap 2015).

### Measurement uncertainty

As indicated, errors in ‘traditional’ wet-chemistry measurements can be considerable; sampling and sample preparation errors are of the same order of magnitude as errors caused in the chemical analysis themselves (Wagner *et al.* 2001). Hence, sampling and sample preparation require the same attention and equivalent measures for quality assurance (QA) and quality control (QC) as the chemical analyses



themselves. However, this aspect is not always taken into consideration (Fernández-Ugalde *et al.* 2020). Tirez (2014), for example, reported that monitoring uncertainties derived from an interlaboratory trial (including sampling) amounted to  $\pm 20\%$  (95% confidence interval (CI)) for soil organic carbon and  $\pm 0.3$  pH units (95% CI) for soil acidity.

The above errors will carry over into calibrations and reduce the accuracy of spectrometric estimates (Viscarra Rossel and Bouma 2016; Poppiel *et al.* 2022; Viscarra Rossel *et al.* 2022). Performance of harmonisation functions further depends on parameters (soil properties) and sampling protocols (Louis *et al.* 2014; Shamrikova *et al.* 2022). With respect to MIR-DRS (Mid infrared, diffuse reflectance spectroscopy), Minasny (2009) indicated “local calibration is preferred to avoid the measurement biases between laboratories, and the calibrated functions are more specific to the soil types in an area”. Yet, there is a trade-off with sample size (i.e., local calibration is based on fewer data). Similarly, regression methods for bulk density are economical as they can make indirect ‘measurements’ (rather predictions), but again these depend on sufficient good quality data of soil texture and organic matter content as well as environmental properties. Also, like most of the other approaches, PTF-accuracy tends to decrease with sampling depth (Al-Shammary *et al.* 2018). Likely, this is due to the fact that generally fewer samples are taken at greater depth and the effect of environmental variables tends to be less at greater depth.

ICP Forests level-2 inventories provide information that permits to track the applied methods, the instrument detection limits, method detection limits, and the ring-test proficiency of the laboratories that produced the analytical data (ICP Forests 2021b, chapter 3). According to Fleck (2016), a limitation of the level-2 dataset is that analyses were carried out by different national laboratories in Europe instead of one central laboratory. Similarly, for ICP Forests level-1, for ‘analyses in the laboratory, all laboratories have to use the reference methods, which mainly follow ISO standards’ (ICP Forests 2021b), but no reference is made to a central, certified laboratory to ensure consistency.

### *Lack of standardised procedures*

Different monitoring systems, such as LUCAS and ICP Forests, use different approaches for sampling soils with respect to the consideration of a possible surficial organic layer. Further, use of different instruments and techniques for soil sampling (e.g., a spade versus gouge auger, or litter removal) will affect results to a varying extent (Fernández-Ugalde *et al.* 2020). This may lead to difficulties when comparing results from different (national) monitoring programmes (see Bispo *et al.* 2021).

The LUCAS 2022 sampling round will include a double sampling exercise to develop transfer functions between national and LUCAS-EU methods (with focus on agricultural land) (Bispo *et al.* 2021). Similarly, Ross (2015) indicated long-term forest soil monitoring and research often require a comparison of laboratory data generated at different times and in different laboratories. Quantifying the uncertainty associated with these analyses is necessary to assess temporal changes in soil properties, with consideration for differences between soil types (Ross *et al.* 2015). It should be noted here that temporal change can only be detected if it is larger than the uncertainty caused by measurement error (Crawford *et al.* 2018). Such efforts should be coupled with ongoing activities to review and standardise

laboratory methods by the ‘Global Soil Laboratory Network’ (GLOSOLAN 2020), the ‘Wageningen Evaluating Programmes for Analytical Laboratories’ (WEPAL 2022), EURACHEM (2015), ISO and similar.

Figure 1 (Nemes *et al.* 1999) shows that limits for defining particle size distribution vary widely throughout Europe. Conversion of soil textural data based on ‘< 1 mm’ and ‘< 2 mm’ schemas for defining the ‘fine earth’ fraction are problematic (Stolbovoi 2001, p. 15). Importantly, Yudina *et al.* (2018) suggest the upper boundary of the clay fraction as used in the Russian Federation and former satellite countries classification (i.e. Katschinski 1956) should be shifted from 1 to 2 mm. Further, data transformation between different national soil texture schemes (within a given ‘fine earth fraction’, e.g. ‘< 2 mm’) is particularly cumbersome and generally requires access to comparative assessments that consider at least 5 or 6 different particle size limits (Rousseva 1997; Makó *et al.* 2017; Moeys 2018; Sadovski and Ivanova 2020; Takahashi *et al.* 2020; Van Egmond *et al.* 2021), while commonly only three size classes are defined or presented in most datasets (as submitted for consideration in WoSIS). Nemes *et al.* (1999) indicated that ‘once an extensive external reference data set with well-quantified particle-size distributions is available, the similarity procedure becomes a very powerful tool for interpolations.’ Alternatively, GlobalSoilMap (2015) developed a prototype library of R functions that allows conversion from systems of particle size classification different from the USDA to the standard particle size classes of the USDA system (clay  $\leq 2 \mu\text{m}$ , silt = 2-50  $\mu\text{m}$  and sand = 50-2000  $\mu\text{m}$ , determined by the pipette method). Similar procedures would be needed for conversion to the class limits used by LUCAS and ICPF (i.e., clay  $\leq 2 \mu\text{m}$ , silt = 2-63  $\mu\text{m}$  and sand = 63-2000  $\mu\text{m}$ ).

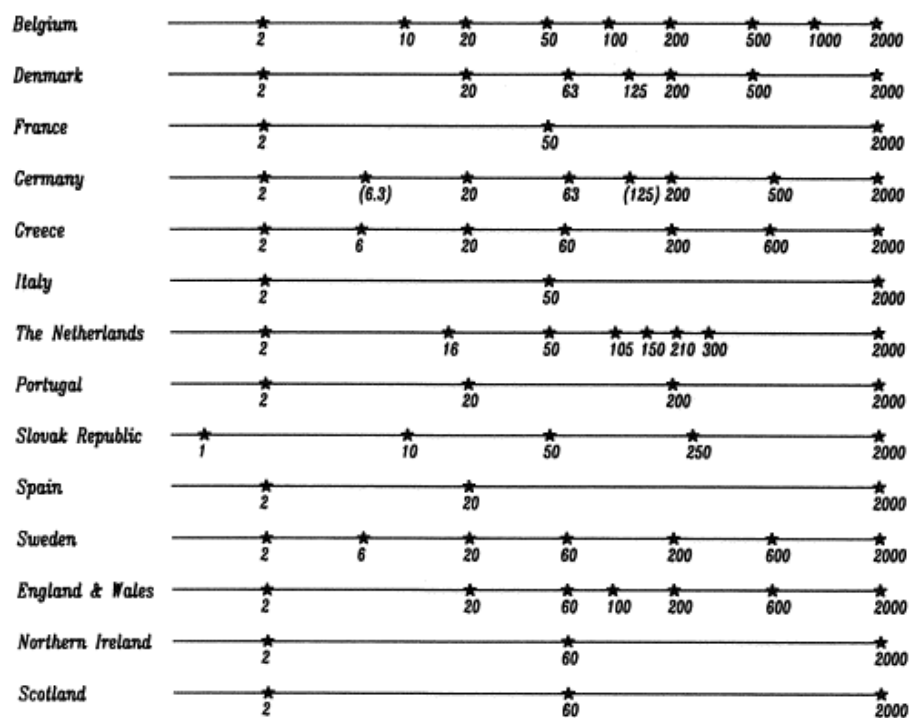


Figure 1. Differences in defining particle size distribution across Europe

Additional, generally unaccounted for uncertainties arise when differences in analytical or physical methods for the determination of particle size fractions themselves are also considered, such as use of

pipette, hydrometer or laser diffraction (Buurman and Van Doesburg 2007; Faé *et al.* 2019) as well as effects of different dispersing agents (Matar *et al.* 1980; Coates and Hulse 1985; Makó *et al.* 2017; Mwendwa 2022).

### 3.3.2 Grouping soil data according to operational definitions

When confronted with soil analytical data derived from a wide range of laboratories that use different national standards, and considering the observations made in Section 3.3.1, a practical solution towards harmonisation is to cluster the shared soil data according to key elements of the chemical procedures under consideration according to operational definitions (or functional properties, Soil Survey Staff 2022). In other words, analytical data obtained from different laboratories are grouped according to key criteria of the procedures themselves. This approach is illustrated in Table 2 using pH-KCl as an example. Of necessity, such an approach assumes interlaboratory differences for a given functional group are negligible, which is a simplification. Nonetheless, this approach is followed in SoilGrids mapping (L. Poggio *et al.* 2021) and similar broad scale approaches have been used in the framework of the HWSD (FAO *et al.* 2012) and an update thereof (WISE30sec, Batjes 2016). As indicated earlier, the desired final step of full harmonisation to an agreed reference method (Y) would require access to a wide range of comparative data sets that would permit development of PTFs between different 'functional groups' as discussed by Batjes (2020). Similarly, GLOSOLAN (Suvannang *et al.*, 2018, p. 10) wrote that "comparable and useful soil information (at the global level) will only be attainable once laboratories agree to follow common standards and norms". Further, "to allow comparison between data produced using different methods (for those laboratories unable to implement GLOSOLAN's SOPs), GLOSOLAN will develop conversion factors between the national methods and GLOSOLAN SOP's" (GLOSOLAN/FAO 2022). A similar exercise, with ISO SOP's as reference methods, is being undertaken in the framework of the 'LUCAS 2022' sampling programme. In all situations, it will be necessary to participate in international round-robin rounds (e.g., EURACHEM 2015; WEPAL 2022), and to consider comparisons between results obtained with ISO SOP's and GLOSOLAN SOP's on a representative set of reference samples.

Table 2. Characterising soil analytical methods for pH-KCl according to key criteria

Key	ISO <sup>a</sup>	ISRIC <sup>b</sup>	USDA <sup>c</sup>	WEPAL <sup>d</sup>	GLOSOLAN <sup>e</sup>
Pretreatment	< 2 mm	< 2 mm	< 2 mm	< 2 mm	< 2 mm
Solution	KCl	KCl	KCl	KCl	KCl
Concentration	1 M	1 M	1 M	1 M	1 M
Ratio	1:5	1:2.5	1:1	1:5	1:5
Ratio base	v/v	w/v	w/v	v/v	w/v
Instrument	Electrode	Electrode	Electrode	Electrode	Electrode

Source: Ribeiro (2020)

*Footnotes:* A) ISO 10390:2021 specifies an instrumental method for the routine determination of pH using a glass electrode in a 1:5 (volume fraction) suspension of soil in water (pH in H<sub>2</sub>O), in 1 mol/l potassium chloride solution (pH in KCl) or in 0.01 mol/l calcium chloride solution (pH in CaCl<sub>2</sub>) (ISO-10390 2021); this coding example is for pH KCl. B) ISRIC: Method 4-1 for pH-KCl (van Reeuwijk 2002), C) USDA: Method 4C1a2a3 (Soil Survey Staff 2022), D) WEPAL Wageningen Evaluating Programs for Analytical Laboratories – [www.wepal.nl](http://www.wepal.nl) – WEPAL is an accredited world-leading organiser of proficiency testing programmes in the fields of plants, soil, sediments and organic waste. Participants in the International Soil-Analytical Exchange programme

receive four times a year, four samples to be analysed for comparison of results. Participants describe the applied extraction/digestion, and the method of detection of the particular element of their method applied, E) GLOSOLAN/FAO (2021): The pH of the soil measured in either water, CaCl<sub>2</sub>, or KCl systems.

## 4. Towards a tier-based approach

Harmonisation of soil data determined using different sampling and analytical procedures is feasible, yet fraught with uncertainty. Strictly speaking, there should be no major technical difficulties to full data harmonisation when a new monitoring system is established. In practice, however, when faced with existing soil data the possible level of harmonisation (tier-level) will be determined largely by data availability and license constraints.

Basically, based on Section 3.3, there are four pathways towards full data harmonisation. In similarity to IPCC (2019), different levels of methodological complexity are proposed. Tier 1 would be the basic method (i.e., mainly crucial standardisation), while the Tier 4 approach would be the most demanding in terms of complexity and data requirements, as well as cost of implementation.

- Tier 1: Group analytical data sourced from different laboratories according to key criteria of the procedures themselves, that is according to their ‘functional properties’ or operational definitions. Users must then determine which subdivisions within a given functional group (e.g., silt size fraction or pH<sub>KCl</sub>) they consider to be comparable for their envisaged applications.
- Tier 2: Convert national data to the selected reference methods (*in casu* ICPF), based on a set of representative ‘reference’ soil samples analysed according to reference method (Y) and the national standards (X), using locally calibrated/validated conversion functions (with accounting of uncertainty).
- Tier 3: Collect all samples in a country using the accepted reference methods (*in casu* ICPF) and subsequently analyse the soil samples in national certified laboratories (using the adopted reference methods) with overall quality assurance/control enforced at a ‘main’ central laboratory, with ‘round-robin’ rounds.
- Tier 4: Collect all samples using an accepted reference method (e.g., LUCAS or ICPF) and subsequently prepare and analyse all soil samples in *one* specific, certified reference laboratory (with consideration of an adequate number of replicates and participation in international ‘round-robin’ rounds).

## 5. Concluding remarks

For consistent trans-national monitoring programmes a tier-4 approach would be preferred for highest accuracy, followed by a tier-3 approach. Inherently, adoption of such approaches would require long-term resources from funding agencies or international organisations. Countries with long-standing soil survey or monitoring programmes, however, may be reluctant to adopt such an approach (see Bispo *et al.* 2021; Soil Survey Staff 2022).

In practice, such as for the HoliSoils mapping component, disparate soil data will often have to be used. However, results of comparative (i.e., intra- and inter-laboratory) analyses are seldom available or shared thereby limiting the scope for PTF-development and full harmonisation. So in essence, when multiple source data need to be combined (e.g., ICPF, RQMS, LUCAS, and different national datasets) a tier 1 approach towards harmonisation may be the most realistic (and only feasible) option, particularly for broad scale assessments, pending the implementation of tier 3 or tier 4 types of trans-boundary monitoring programmes.

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## Appendix - Reference methods for chemical properties (ICPF)

Parameter	Reference Analysis Method <sup>1</sup>			Unit <sup>2</sup>			
	ISO	Extractant	Measurement method(s) <sup>3</sup>				
pH(CaCl <sub>2</sub> )	ISO 10390 (2005)	0.01 M CaCl <sub>2</sub>	pH-electrode				
pH(H <sub>2</sub> O)		H <sub>2</sub> O	pH-electrode				
Total nitrogen	ISO 13878 (1998)	-	Dry Combustion	g/kg			
	ISO 11261 (1995)	-	Modified Kjeldahl				
Total organic carbon <sup>4</sup>	ISO 10694 (1995)	-	Dry Combustion at 900 °C				
Carbonates	ISO 10693 (1994)	HCl	Calcimeter				
P	ISO 11466 (1995)	Aqua Regia by reflux digestion	ICP	Colorimetry	mg/kg		
K, Ca, Mg, Mn				AAS			
						Heavy metals: Cu, Cd, Pb, Zn	
Other: Al, Fe, Cr, Ni, Na							
			Hg	ICP	Cold vapour AAS		
ICP							
S		CNS - analyser					
Free Acidity (or sum of AC <sup>5</sup> ) and free H <sup>+</sup>	ISO 11254 (1994) modified	0.1 M BaCl <sub>2</sub> <sup>1</sup>	titration to pH 7.8 or 'German' method		cmol(+)/kg		
Exchangeable Cations	Al, Fe, Mn	ISO 11260 (1994) modified	0.1 M BaCl <sub>2</sub>	ICP	AAS	-	
	K, Ca, Mg, Na					FES	
Reactive Fe and Al Oxalate extractable P	ISRIC (2002)	Acid ammonium oxalate	AAS	ICP		mg/kg	
Total Elements: Ca, Mg, Na, K, Al, Fe, Mn	ISO 14869-1 (2001)	HF or LiBO <sub>2</sub>	AAS	ICP		mg/kg	

Source: ICP Forests (2021b), soil textural classes, pipette method, are defined as: clay ≤2 µm, silt = 2-63 µm and sand = 63-2000 µm. These are referred to as reference method Y in the text.

<sup>1</sup> Note: The ICPF reference method deviates from ISO 11260 & ISO 14254 in the sense that one single barium chloride extraction must be used instead of three extractions (<http://icp-forests.net/page/icp-forests-manual>).

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