Originalarbeit

Raghad Shwiekh, Judith Schick, Sylvia Kratz, Daniel Rückamp, Ewald Schnug

Comparative investigation and inter-calibration of different soil P tests

Vergleichende Untersuchung und Interkalibrierung verschiedener P-Extraktionsmethoden

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Abstract

To allow the comparison and interpretation of data obtained by different soil P tests, different established extraction methods were investigated and statistically inter-calibrated. All soil P tests applied in this study were significantly correlated with each other. Their extracting force varied considerably, decreasing in the order $P_{AR} > P_{AL}, P_{M3} \geq P_{PAAAc-EDTA}, P_{DL} \geq P_{CAL} \geq P_{Olsen}, P_{AAAc} \geq P_W$. Generally, it was possible to transform data from one soil P test into another one. However, the quality of the resulting values depended on the pair of soil tests at question. Based on the present set of data, values from CAL, AL, M3, AAAc-EDTA and water extracts showed strong correlations and consequently allowed for the calculation of highly significant regression equations with a strong coefficient of determination. While in some cases, simple regressions already yielded a coefficient of determination > 80%, in other cases additional soil parameters (such as soil-pH, Zn_{WH}, Fe_{WH}, Al_{WH}, Ca_{WH} and C_{total}) had to be included in order to achieve this high level of accuracy. In contrast, values obtained from extractions with $NaHCO_3$ (Olsen), DL, and AAAc displayed weaker correlations. Accordingly, no satisfactory regression equations (i.e. with $R^2 > 80\%$) could be produced for these three methods. As major obstacles, differences in chemical composition, acidity and extraction force of the various extractants were identified.

Key words: Phosphorus, soil P tests, inter-calibration

Zusammenfassung

Ziel dieser Arbeit war es, zu ermitteln inwieweit die Ergebnisse verschiedener P-Extraktionsmethoden miteinander vergleichbar sind. Dazu wurden 8 Methoden untersucht und interkalibriert. Alle untersuchten P Tests waren signifikant miteinander korreliert. Die Extraktionsstärke nahm in der Reihenfolge $P_{AR} > P_{AL}, P_{M3} \geq P_{AAAc-EDTA}, P_{DL} \geq P_{CAL} \geq P_{Olsen}, P_{AAAc} \geq P_W$ ab. Grundsätzlich war es möglich, die Werte einer Methode in die einer anderen zu übertragen, allerdings hing die Qualität der Ergebnisse stark von den jeweils verglichenen Methoden ab. Basierend auf dem in dieser Arbeit verwendeten Datenmaterial zeigten die Extraktionen mit CAL, AL, M3, AAAc-EDTA und Wasser die stärksten Korrelationen und waren entsprechend am besten für die Berechnung hoch signifikanter Regressionsgleichungen mit hohem Bestimmtheitsmaß geeignet. Während für einige dieser Methoden einfache Regressionsgleichungen bereits zu einem Bestimmtheitsmaß von > 80% führten, mussten in einigen Fällen weitere Bodenparameter (z.B. Bodenreaktion, Zn_{WH}, Fe_{WH}, Al_{WH}, Ca_{WH} und C_{total}) mitberücksichtigt werden, um diese Genauigkeit zu erlangen. Die Ergebnisse der Extraktion mit $NaHCO_3$ (Olsen), DL, und AAAc zeigten dagegen schwächere Korrelationen. Entsprechend konnten für diese drei Methoden keine Regressionsgleichungen mit einem ausreichend hohen Bestimmtheitsmaß (z.B. $R^2 > 80\%$) abgeleitet werden. Als wichtigste Hindernisse wurden die Unterschiede in der chemischen Zusammensetzung, dem Säuregrad und der Extraktionsstärke der Extrakte identifiziert.

Introduction

High amounts of available P fractions in agricultural soils, e.g. as a result of excessive manure application, are usually strongly correlated with high amounts of dissolved reactive P (DRP) in drainage water and leachate. Correspondingly, lower amounts of plant-available P in the soil result in lower levels of P in the drainage water (HESKETH and BROOKES, 2000; WITHERS et al., 2000; SIMS et al., 2002; MAGUIRE et al., 2005). Based on the observation that very little P (< 0.15 mg P/l) can be found in drainage water from soils with a concentration of < 60 mg Olsen P/kg, while the P concentrations in drainage water from soils above this level increases linearly with soil Olsen P concentrations, HESKETH and BROOKES (2000) determined the so-called "change-point" (60 mg Olsen P/kg) which can be applied to assess the probability of P leaching from agricultural soils. In this case, the accurate assessment of available P in soils is fundamental to identify those spots which are prone to P-leaching. Additionally, soil P testing is important to formulate fertiliser recommendations which are adjusted to the actual P supply in the soil and the demand of the particular crop. A balanced fertiliser application is a major prerequisite to reduce the amount of available P in soils and thus prevent P-losses from agricultural fields.

In the past, a multitude of different extraction methods were developed to assess the amounts of available P in the soil to reflect the plant availability, and to predict potential environmental impacts. Due to different processes such as precipitation/dissolution and sorption/desorption, P exists in a wide range of chemical forms in the soil. Thus, the concept of the various soil tests is to mirror different soil processes such as dissolution, desorption and chelation of the different P forms to dissolve P (MAGUIRE et al., 2005). In different countries, different extraction methods are used as a standard to assess the available P amounts in soils. The methods differ concerning parameters such as pH, extraction time, soil-solution ratio, temperature and concentration of active agents (SIBBESEN, 1983; MAGUIRE et al., 2005) which leads to the extraction of different amounts of P. The choice of a soil test as the national standard method usually is the result of the suitability of the extractant for the majority of soils in that particular country.

As international research cooperation is nowadays intensified, a harmonised interpretation of results obtained from different soil P tests is required. This will be the only way to formulate common fertiliser recommendations and to facilitate international research and environmental control (OTTABONG et al., 2009). However, it is still questionable whether soil P tests are comparable at all and if the results can be transferred into one another (OTTABONG et al., 2009).

One important area where international research cooperation is urgently needed is the Baltic Sea Region (BSR). The Baltic Sea is one of the most highly polluted marine bodies in the world, particularly so with regard to N and P eutrophication. With agriculture being by far the largest emitter of diffuse P losses into marine environments, there is a pressing demand for concerted action of all Baltic Sea member states. This study was therefore conducted to elaborate if the results obtained by different standard soil P tests applied in the Baltic countries are generally comparable and transferrable and, where possible, provide equations for their "translation", taking into account some of the key soil parameters if necessary.

This shall allow for a more reliable assessment of P supply and turnover in agricultural soils under different environmental conditions, which facilitates the estimation of P losses, particularly for hotspots in the BSR. The information gained from this can be used to adapt precision farming soil tillage and fertilisation practices and to adjust policy instruments.[1]

Material and Methods

Soil samples and analysis

183 soil samples were examined within the framework of this study: 83 samples originated from Estonia and Finland and represented different sampling sites throughout these two countries. 100 samples originated from Germany and Poland and were taken during a field sampling campaign in 2011. Air-dried soil samples which were previously sieved to ≤ 2 mm were used for analysis. The extraction methods applied in this study are listed in Tab. 1.

Besides extractable P, "so-called total" P was determined by *aqua regia* digestion. Furthermore, C_{total}, soil pH and Al, Ca, Cu, Fe and Zn dissolved by the Westerhoff extract (1954/1955) were analysed (Tab. 2).

Statistical analyses

Comparisons between the different extraction methods were conducted by applying a one-way ANOVA and Tukey post hoc test. Correlation analyses were performed and simple and multiple regression equations calculated. All statistical analyses were done by employing SPSS version 17.0 and Microsoft Office EXCEL 2007.

Results and Discussion

The pH-values of the soil samples ranged from 3.7 (very strongly acid) to 7.4 (slightly alkaline). Most of the soil samples (71%) were classified as being moderately or slightly acidic (pH 5.0–6.9) (Tab. 3).

The results of the extraction methods and other chemical soil parameters are listed in Tab. 4. The wide range indicates the heterogeneity of the samples used in this study. Furthermore, significant differences in the extraction force

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[1] The detailed report ("Comparison and Inter-Calibration of Different Soil P Tests Used in the Baltic Sea Countries") about this study which was written within the framework of the EU-project Baltic Manure can be found at www.balticmanure.eu.

Tab. 1. Standard P-extraction methods employed (modified after ERIKSSON, 2009 and JANSSEN, 2004)

Agent	Reference	Short	Wave-length (nm)	Used as standard P-extraction method in:
Ammonium lactate	EGNÉR (1954)	P_{AL}	772	Lithuania, Sweden, Norway, Slovakia
Double lactate	RIEHM (1942)	P_{DL}	580	Latvia, Poland, Germany, Austria
Calcium lactate	SCHÜLLER (1969)	P_{CAL}	882	Germany, Austria
Mehlich 3	MEHLICH (1984)	P_{M3}	882	Estonia, Czech Republic
Olsen	OLSEN et al. (1954)	P_{Olsen}	820	Denmark, Italy, Greece, UK, Spain
Acid ammonium acetate	VUORINEN and MÄKITIE (1955)	P_{AAAc}	712	Finland
Acid ammonium acetate + EDTA	LAKANEN and ERVIÖ (1971)	$P_{AAAc\text{-}EDTA}$	882	Switzerland, Belgium
Water	VAN DER PAAUW et al. (1971)	P_W	882	Netherlands

Tab. 2. Additional analytical methods

Parameter	Short	Extractant	Method	Analysis
"So-called total" P	P_{AR}	conc. HNO_3 + conc. HCl (1 + 3)	ABFKLÄRV (1992)	SPECORD® 50 spectrometer; 882 nm
Al	Al_{WH}	0.43M HNO_3	WESTERHOFF (1954/1955)	ICP-OES
Ca	Ca_{WH}			icap 6000 (Thermo)
Cu	Cu_{WH}			
Fe	Fe_{WH}			
Zn	Zn_{WH}			
Total C	C_{total}		High temperature combustion at 1150°C (with oxygen supply)	C/N-Analyzer variomax
	N_{total}			
pH			Suspension with 0,01 M $CaCl_2$, potentiomentric measurement with glass electrode (VDLUFA-method, HOFFMANN, 1991)	

Tab. 3. Soil classification (excerpt) based on soil pH (determined in 0.01 M CaCl2) (SCHEFFER and SCHACHTSCHABEL, 2010) (n = 183)

pH-value ($CaCl_2$)	Classification	n
3.0–3.9	very strongly acid	1
4.0–4.9	strongly acid	33
5.0–5.9	moderately acid	76
6.0–6.9	slightly acid	54
7.0	neutral	5
7.1–8.0	slightly alkaline	14

of the different methods were found. The extraction force decreased in the order: P_{AR} > P_{AL}, P_{M3} ≥ $P_{AAAc\text{-}EDTA}$, P_{DL} ≥ P_{CAL} ≥ P_{Olsen}, P_{AAAc} ≥ P_W (Fig. 1). As expected, AR extracted by far the highest amounts of P, while water extracted the lowest. It is known that extractants which are more acid or alkaline than the soil solution will also extract P forms with a low plant-availability (SELF-DAVIS et al., 2000). In contrast, the water extraction maintains the soil pH within one unit of its original value. Furthermore, it is expected to simulate the release of P to run-off or leaching by water more accurately than stronger chemical extractants and can thus be used for agronomic purposes from an environmental perspective (MOORE et al., 1998). Accordingly, a close correlation of water with DRP (= dissolved reactive P)-concentrations in run-off from agricultural land has been identified (POTE et al., 1996). Furthermore, it could also be observed in the present study that the addition of EDTA to the AAAc-extract increases the amount of extracted P significantly. This confirms the findings of LAKANEN and ERVIÖ (1971) who also observed a higher extraction force of AAAc-EDTA. The purpose of the application of EDTA is the complexation of phosphate binding cations such Ca, Al or Fe and a number of trace metals (e.g. Cu) in order to prevent their re-precipitation with phosphates. Otherwise, this

Tab. 4.   Soil properties of the samples used and P-concentrations extracted by different extractants

| Parameter	n	Unit	Min	Max	Mean	STD	Median
P_{AR}	183	mg/kg	46	3895	764	± 811	665
P_{AL}	183	mg/kg	14	2136	178	± 423	129
P_{DL}	177	mg/kg	5.3	571	120	± 135	92
P_{CAL}	183	mg/kg	3.0	1110	96	± 219	65
P_{M3}	183	mg/kg	2.5	1936	165	± 381	139
P_{Olsen}	183	mg/kg	0.8	219	58	± 48	54
P_{AAAC}	161	mg/kg	2.6	132	35	± 32	18
$P_{AAAC\text{-}EDTA}$	183	mg/kg	4.0	2096	147	± 406	96
P_W	183	mg/kg	2.9	180	19	± 31	17
pH	183		3.7	7.4	5.8	± 0.8	5.8
C	183	%	0.10	20	2.8	± 2.8	1.7
Cu_{WH}	183	mg/kg	0.5	19	5.7	± 2.7	4.3
Zn_{WH}	182	mg/kg	0.5	139	10	± 26	6.7
Al_{WH}	183	mg/kg	467	8486	1726	± 331	984
Fe_{WH}	183	mg/kg	296	6482	1688	± 1165	1293
Ca_{WH}	183	mg/kg	387	66400	4365	± 6309	2561

± expresses the standard deviation (=STD)
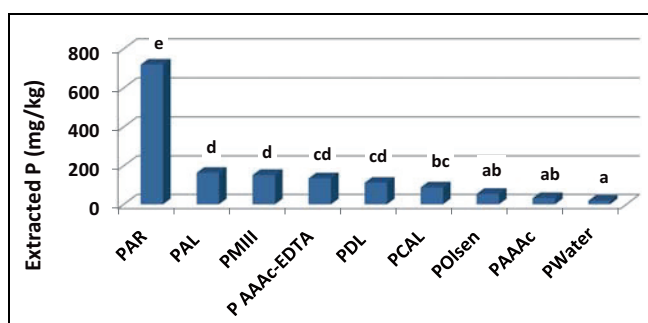
Fig. 1. Extractable P (mean) (mg/kg) determined by 9 different methods (significant differences between groups were determined by Tukey post-hoc test (p < 0.05) and are denoted by different letters).

re-precipitation might occur during the extraction process as a secondary reaction (Gallet, 2001; Cottenie et al., 1979).

The results shown in Tab. 4 and Fig. 1 are in good agreement with the findings of Neyroud and Lischer (2003), who compared the extraction force of 16 different extraction procedures. The amount of extracted P in their study decreased in the order[1]: **P_{total}** > ($P_{oxal.}$) > **P_{AL}** > **P_{M3}** (> P_{Bray}) > **$P_{AAAC\text{-}EDTA}$, P_{DL}, P_{CAL}** > **P_{Olsen}** (> $P_{Paper\ Strip}$) > **P_{AAAc}**, (P_{Morgan}) > **P_W**, (P_{CO2}, P_{CaCl2}).

The differences in extraction force between the various methods can be mainly attributed to different extraction mechanisms which base on individual active components in the extract. The three strongest methods, AL, M3 and AAAc-EDTA, all contain one or more strong chelating agents such as lactate, EDTA, NH_3F and acetate (the latter, however, according to Eriksson (2009) displaying only mild chelating properties), which are able to release P from Al- and Fe-P-compounds. In addition, AL and M3 have a low pH (< 4), leading to the hydrolysis of P in insoluble Al-humic-P substances and the dissolution of sparingly soluble Ca-P compounds (Ottabong et al., 2009). In contrast, the two methods with medium strength, DL and CAL, both contain only one chelating agent, lactate in combination with Ca as active cation. Ca^{2+} may precipitate some of the extracted P in the extraction solution and thus will lead to lower P-values compared to those extractants containing NH_4^+ as active cation (like AL) (Eriksson, 2009). The weaker extractants, Olsen and water, only hydrolyse those Fe-/Al-P compounds which are easily soluble. In addition, the alkaline Olsen-extract releases P from Ca-oxides and some of the P found in Ca-phosphates (Olsen et al., 1954). Due to its alkaline pH, Olsen may show an advantage for soils rich in organic P such as peat soils, as it accesses the organic P pool more aggressively than acid extractants (Ottabong et al., 2009). As only very few of the soil samples investigated in this study showed an OM-content higher than 20%, this finding can neither be confirmed nor disproved here. The extraction force of AAAc is mainly based on its content of acetic acid. Therefore it can also be included into the group of weak P extractants. Besides the active components in the extract, factors such as soil: solution ratio as well as duration and power of shaking the samples influence the extraction force of the different methods (Eriksson, 2009).

The differing extraction forces of the various methods are useful for different purposes and also ask for different

[1] **in bold**: methods that were researched within this study.

interpretation strategies for each method (Neyroud and Lischer, 2003). Weak extraction methods tend to reflect immediately available P and may be useful for immediate fertilisation recommendations. In contrast, the stronger extractants are rather related to P species which are more strongly bound and will become available on a medium to long-term basis, and thus essential for planning fertilisation on a mid to long-term basis. Regarding the assessment of the actual risk of P loss and environmental pollution (eutrophication of ground and surface waters), the weak extraction methods appear to be more suitable to the authors of this study.

Of course, soil characteristics also play an important role in the performance of the different extraction methods. Thus, key factors influencing the type of P binding such as soil pH, organic carbon, and contents of Fe_{WH}, Al_{WH}, Ca_{WH}, Zn_{WH} and Cu_{WH} were also included into the regression equations.

The main soil parameters for the soil samples examined per country are presented in Tab. 5.

The average pH of the soil samples from Estonia was 6.3. Worth mentioning were the significantly elevated Ca_{WH}-concentrations and comparatively low Cu_{WH} and Zn_{WH} concentrations in these soils.

The parameters of the Finnish soil samples differed significantly from those of the three other countries; the soils were most acidic (pH 5.0), the C-contents were by far the highest and the results for Cu_{WH}, Zn_{WH}, Fe_{WH} and Al_{WH} were elevated, as well. The average pH of the soil samples from Germany was 6.2 (slightly acidic) and Cu_{WH}- and Zn_{WH} concentrations were elevated in comparison to the Estonian soil samples. This tendency was also observed for the Polish soils.

Tab. 6 shows the average amounts of P extracted by different extraction methods. Among all soil samples, both, the "so-called total" P-content and extracted P were significantly higher in the samples from Poland. In contrast, the mean total P content in the Finnish soils was also elevated, but the share of extractable P in these soils was on a very low level for all methods. Since the pH of the Finnish soils was moderately acid and the Fe_{WH}- and Al_{WH}-concentrations were distinctively elevated (Tab. 5), it can be assumed that the formation of insoluble Fe_{WH}- and Al_{WH}-complexes was responsible for an increased P-fixation and resulted in the low share of extractable P. The average amount of extractable P in the Estonian soils was also very low and comparable to the Finnish soils. With view to high Ca_{WH}-concentrations this might be explained by the precipitation of sparingly soluble Ca-P-compounds (Tab. 5). The amounts of extractable P in the German soils ranged between those of Finland/ Estonia and Poland.

At present, different standard methods are used among different countries to assess and to interpret the amount of available P in agricultural soils (see Tab. 1). Exemplary, the standard P extraction methods and the respective classification systems for Estonia, Germany, Poland and Sweden are listed in Tab. 7.

Tab. 5. Mean concentration of soil parameters for the soils from Estonia, Finland, Germany and Poland

| | pH | C (%) | Westerhoff | | | | |
			Ca (mg/kg)	Fe (mg/kg)	Al (mg/kg)	Cu (mg/kg)	Zn (mg/kg)
Estonia (n = 22)	6.3	1.6	10051	647	1155	1.5	2.7
Finland (n = 61)	5.0	5.1	2983	2852	3439	10	9.1
Germany (n = 60)	6.2	1.5	3034	1128	789	4.3	12
Poland (n = 40)	6.0	1.9	5341	1323	831	3.2	13

Tab. 6. Mean concentration (± expresses the standard deviation) of soil parameters on 7 sampling sites in Germany and Poland and the two sets of soils samples from Estonia and Finland (mg/kg)

	P_{AR}	P_{AL}	P_{DL}	P_{CAL}	P_{M3}	P_{Olsen}	P_{AAAC}	$P_{AAAc-EDTA}$	P_{Water}
Estonia (n = 22)	580 ± 52	107 ± 65	64 ± 6	49 ± 41	115 ± 72	33 ± 23	n.a.	66 ± 46	12 ± 6.9
Finland (n = 61)	859 ± 57	92 ± 50	68 ± 7	50 ± 26	87 ± 78	52 ± 20	11 ± 8	66 ± 41	13 ± 6
Germany (n = 60)	548 ± 136	136 ± 88	130 ± 78	98 ± 64	164 ± 72	55 ± 20	44 ± 35	138 ± 90	22 ± 8
Poland (n = 40)	1042 ± 769	370 ± 97	209 ± 33	189 ± 205	311 ± 349	83 ± 47	56 ± 35	329 ± 379	30 ± 29

Tab. 7. Country-specific differences in the classification of the P-supply of soils

Country	National extraction method	Classification (mg P/100 g soil)				
		Strongly deficient	Deficient	Sufficient	Excessive	Strongly excessive
Estonia*	M3	< 1.5	1.5–4.0	4.1–9.5	9.6–20.5	> 20.6
Germany	CAL	≦ 2.2	2.2–3.9	4.9–7.2	7.3–10.4	≧ 10.5
Poland	DL	< 2.2	2.2–4.4	4.4–6.5	6.5– 8.7	> 8.7
Sweden	AL	< 2	2–4	4–8	8–16	> 16

* < 2% humus content

Differing reference methods and classification systems exist in each country which consequently results in a divergent assessment of the P supply of agricultural soils (Fig. 2). While the German classification tends to group sites into lower (deficient) classes, they are assessed as being sufficiently or even excessively supplied with P if one of the other national procedures is applied. These findings underline the necessity for a harmonisation of P-extraction and classification standards.

Inter-Calibration
For the direct comparison of different extraction methods, suitable equations were calculated to allow for the transformation of the results of one method into another. In some cases, satisfactory coefficients of determination were already achieved by simple single-parameter regression equations, while in other cases the inclusion of one or more soil parameters into multiple regression equations was required in order to account for the influence of soil characteristics on P dissolution. The selection of variables will always be a compromise between keeping the number of analyses required as low as possible and reaching a satisfactory level of determination for the calculation (R^2 should be at least 80%). The strategy applied in this study was to work with a stepwise multiple regression, starting with one soil parameter and then successively including more parameters until a satisfactory level of determination was reached. The SPSS routine "stepwise regression" was used, applying a limit value of 0.05 for the significance of F-probability for the inclusion, and of 0.1 for the exclusion of a variable from the regression equation. In addition to practical considerations (number of additional analyses required), the final decision to keep or discard soil parameters in/from the equations was also based on their relative importance as revealed by the beta correlation coefficient (β). As extra soil parameters, soil-pH, total C (%) and Westerhoff (WH)-extractable Zn, Cu, Al, Fe and Ca were tested for having a potential influence on the extractability/solubility of P by different extractants. At this point, the final multiple regression equations are only presented for those pairs of methods for which no satisfying simple regression equations could be calculated. A step-wise delineation of the multiple regression equations for all methods is given by SCHICK et al. (2013).

Satisfactory results ($R^2 > 80\%$) were already achieved with simple regression equations for the calculation of P_{CAL} from P_{AL}, P_{M3}, $P_{AAAc\text{-}EDTA}$ and P_{water}, which also showed very strong bivariate correlations (r > 0.9) between each two respective extraction methods (Tab. 8).
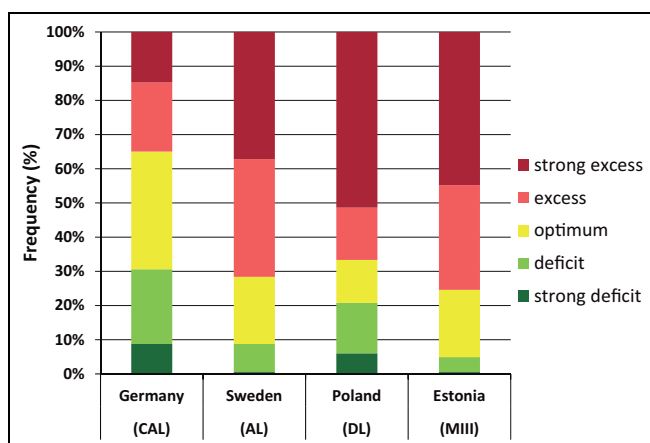

Fig. 2. Percentage distribution of P-classes obtained by applying different national standard extraction methods and the corresponding classification system (see also Tab. 7) (n = 183).

Tab. 8. Calculation of PCAL from other reference methods by simple regression equations (including correlation r between PCAL and respective P-extract, β coefficients for the regressor variable and coefficient of determination R^2 for the equation)

P_{CAL} =	r	β	R^2 (%)
−66.6 + 2.85 * P_{Olsen}	0.810**	0.810	66.5
3.26 + 0.52 * P_{AL}	**0.976****	**0.976**	**95.2**
−29.6 + 1.05 * P_{DL}	0.891**	0.891	79.2
2.68 + 0.56 * P_{M3}	**0.952****	**0.952**	**85.6**
14.1 + 0.55 * $P_{AAAc\text{-}EDTA}$	**0.982****	**0.982**	**96.5**
−32.6 + 6.56 * P_{water}	**0.919****	**0.919**	**84.5**
16.2 + 2.33 * P_{AAAc}	0.652**	0.652	42.2

Significances: * p < 0.05, ** p < 0.01; equations with $R^2 > 80\%$ are marked in bold

Tab. 9.   Calculation of PCAL from other reference methods by multiple regression equations (including β coefficients for the regressor variables and coefficient of determination R^2 for the equation)

P_{CAL} =	β (in the order of parameters given by the equation)				R^2 (%)
$-202 + 2.32 * P_{Olsen} + 27.0 * soil\text{-}pH + 2.17 * Zn_{WH} - 0.010 * Fe_{WH}$					77.1[a]
$-35.1 + 0.88 * P_{DL} + 2.33 * Zn_{WH}$	0.707	0.303			86.3
$21.6 + 0.93 * P_{DL} + 2.21 * Zn_{WH} - 10.4 * soil\text{-}pH$	0.787	0.287	−0.077		86.7[b]
$130 + 2.46 * P_{AAAc} + 4.12 * Zn_{WH} - 28.3 * soil\text{-}pH$	0.688	0.533	−0.208		73.4[a]

Significances: * $p < 0.05$, ** $p < 0.01$; chosen equations are marked in bold and, if $R^2 > 80\%$, marked in italic
[a] no further soil parameters fulfilling the significance criteria for inclusion available
[b] soil-pH obviously has a rather low relative weight (β), so the preceding equation may also be used if soil-pH is not available

Despite different extraction mechanisms, these methods seem to be easily convertible into each other. This is also confirmed with the simple regression equations calculated for each of them (Tables 10, 13, 15, 17). A multiple regression equation revealed satisfactory results for the calculation of P_{CAL} from P_{DL}. However, for P_{Olsen} and P_{AAAc} no coefficient of determination < 80% could be obtained by a multiple regression equation (Tab. 9).

The bivariate correlations (r) between P_{Olsen} and the other P-tests applied in this study were highly significant, but not as strong as those between P_{CAL} and other P-tests ($0.512 \leq r \leq 0.810$). This can be attributed to the alkaline nature of the Olsen-extract, a unique feature among the extractants tested in this study. Accordingly, different soil reactions can be expected, rendering the direct interchangeability with acidic extracts less likely. Consequently, the generation of simple regression equations for transferring Olsen-data into data obtained by acid extracts displayed comparably low coefficients of determination, ranging between $R^2 = 25.8$ and 65.5%. The inclusion of additional soil parameters into multiple regressions did not improve the outcome; coefficients of determination still remained below $R^2 = 80\%$, which is considered too low to apply these equations for a reliable transfer of P-Olsen into another soil test (data not shown here).

P_{AL} showed highly significant and very strong (r > 0.9) bivariate correlations with P_{CAL}, P_{M3}, $P_{AAAc-EDTA}$ and P_{water}. In this case, simple regression equations with only the corresponding P-test as regressor proved to be fully viable for estimating P_{AL} with a satisfactory level of determination (R > 82%) (Tab. 10).

Tab. 10. Calculation of PAL from other reference methods by simple regression equations (including correlation r between PAL and respective P-extract, β coefficients for the regressor variable and coefficient of determination R^2 for the equation)

P_{AL} =	r	β	R^2 (%)
$1.80 + 1.85 * P_{CAL}$	0.976**	0.976	95.2
$-114 + 5.12 * P_{Olsen}$	0.770**	0.770	59.0
$-54.5 + 1.96 * P_{DL}$	0.877**	0.877	76.7
$-0.234 + 1.08 * P_{M3}$	0.943**	0.943	88.9
$23.7 + 1.04 * P_{AAAc-EDTA}$	0.988**	0.988	97.6
$-60.7 + 12.2 * P_{water}$	0.907**	0.907	82.1
$35.8 + 4.11 * P_{AAAc}$	0.606**	0.606	34.6

Significances: * $p < 0.05$, ** $p < 0.01$; equations with $R^2 > 80\%$ are marked in bold

For the relation P_{AL}-P_{DL}, the multiple regression yielded a satisfactory level of determination (85.2%) which was not the case for the relations P_{AL}-P_{Olsen} and P_{AL}-P_{AAAc} (Tab. 11). As indicated before, the reason for this can be most likely attributed to the chemical composition of the extractants (AL − acid/Olsen − alkaline). The main difference between AL and AAAc is assumed to lie in the higher chelating power of AL, which contains lactate as a chelating agent, while AAAc contains acetate, having only mild chelating properties and serving mainly as a pH-reducing component. Furthermore, the ammonium cation in the AL extract results in a high desorption of P (ERIKSSON, 2009).

Tab. 11. Calculation of PAL from other reference methods by multiple regression equations (including β coefficients for the regressor variables and coefficient of determination R^2 for the equation)

P_{AL} =	β (in the order of parameters given by the equation)					R^2 (%)
$-320 + 4.05 * P_{Olsen} + 4.45 * Zn_{WH} + 43.9 * soil\text{-}pH - 0.024 * Fe_{WH}$	0.609	0.305	0.173	−0.127		71.0
$190 + 1.74 * P_{DL} + 4.26 * Zn_{WH} - 42.3 * soil\text{-}pH - 0.019 * Fe_{WH} + 0.003 * Ca_{WH}$	0.777	0.292	−0.165	−0.104	0.086	85.2
$374 + 4.21 * P_{AAAc} + 7.98 * Zn_{WH} - 71.1 * soil\text{-}pH - 0.022 * Fe_{WH} + 0.005 * Ca_{WH}$	0.621	0.543	−0.275	−0.116	0.111	68.6

Significances: * $p < 0.05$, ** $p < 0.01$; chosen equations are marked in bold and, if $R^2 > 80\%$, marked in italic

Tab. 12. Calculation of PDL from other reference methods by multiple regression equations (including β coefficients for the regressor variables and coefficient of determination R² for the equation)

P_{DL} =	β (in the order of parameters given by the equation)					R² (%)
$-64.2 + 0.800 * P_{CAL} + 19.9 * soil\text{-}pH - 1.006 * Zn_{WH}$	*0.947*	*0.175*	*-0.154*			*84.0*
$-286 + 2.25 * P_{Olsen} + 46.5 * soil\text{-}pH$	*0.782*	*0.407*				*78.2*
$-228 + 0.442 * P_{M3} + 43.3 * soil\text{-}pH + 0.022 * Fe_{WH} + 5.08 * C_{total} - 0.012 * Al_{WH} - 0.654 * Zn_{WH}$	0.830	0.379	0.262	0.154 -0.171	-0.101	77.4
$-126 + 0.413 * P_{AL} + 29.5 * soil\text{-}pH - 0.921 * Zn_{WH} + 0.008 * Fe_{WH} - 0.001 * Ca_{WH}$	*0.926*	*0.259*	*-0.141*	*0.091*	*-0.070*	*82.3*
$-117 + 0.415 * P_{AAAc\text{-}EDTA} + 29.3 * soil\text{-}pH - 0.855 * Zn_{WH} + 1.96 * Cu_{WH}$	*0.855*	*0.257*	*-0.131*	*0.083*		*80.1*
$-224 + 4.88 * P_{water} + 40.8 * soil\text{-}pH + 5.99 * C_{total} - 0.734 * Zn_{WH}$	0.808	0.258	0.182	-0.113		70.2
$89.3 + 2.45 * P_{AAAc} + 2.03 * Zn_{WH} - 13.1 * soil\text{-}pH$	0.821	0.313	-0.115			74.9

Significances: * p < 0.05, ** p < 0.01; chosen equations are marked in bold and, if R² > 80%, marked in italic

Bivariate correlations (r) for DL with other extracts were strong and highly significant, but remained below 0.9 (data not shown here). Consequently, the coefficients of determination for the simple regression equations were below 80%, i.e. estimates based on these equations will not be precise enough (data not shown here). Even with multiple regression equations, only levels of determination below 80% were achieved for P_{DL} estimated from P_{Olsen}, P_{M3}, P_{water} or P_{AAAc}. Reliable estimates (R² > 80%) could only be calculated for P_{CAL}, P_{AL} or $P_{AAAc\text{-}EDTA}$ from P_{DL} data when additional soil parameters were included (Tab. 12).

As mentioned earlier, M3, water and AAAc-EDTA were easily convertible into each other as well as into CAL or AL-values by simple regression equations (see Tab. 13, 15 and 17). If further soil parameters are available, in some cases even higher coefficients of determination were achieved by using multiple regression equations (data not shown here).

The application of multiple regression equations did not lead to satisfying results for the conversion of M3 or AAAc-EDTA into Olsen, DL and AAAc (Tab. 14 and 16).

The application of multiple regression equations leads to a satisfying coefficient of determination for AAAc-EDTA-DL. For Olsen and AAAc no coefficient < 80% could be achieved (Tab. 18).

Tab. 13. Calculation of PM3 from other reference methods by simple regression equations (including correlation r between PM3 and respective P-extract, β coefficients for the regressor variable and coefficient of determination R² for the equation)

P_{M3} =	r	β	R² (%)
$17.8 + 1.54 * P_{CAL}$	**0.925****	**0.925**	**85.6**
$-84.0 + 4.36 * P_{Olsen}$	0.748**	0.748	55.7
$17.0 + 0.827 * P_{AL}$	**0.943****	**0.943**	**88.9**
$-21.6 + 1.56 * P_{DL}$	0.796**	0.796	63.1
$35.3 + 0.868 * P_{AAAc\text{-}EDTA}$	**0.942****	**0.942**	**88.7**
$-50.2 + 11.0 * P_{water}$	**0.934****	**0.934**	**87.1**
$58.0 + 3.02 * P_{AAAc}$	0.508**	0.508	25.4

Significances: * p < 0.05, ** p < 0.01; equations with R² > 80% are marked in bold

The weakest bivariate correlations were displayed by AAAc when related to other extraction methods. Hence, neither simple nor multiple regression equations could be generated for a reliable conversion of AAAc-data into data for other methods with a sufficiently high coefficient of determination (data not shown here).

Tab. 14. Calculation of PM3 from other reference methods by multiple regression equations (including β coefficients for the regressor variables and coefficient of determination R² for the equation)

P_{M3} =	β (in the order of parameters given by the equation)				R² (%)
$-0.720 + 3.60 * P_{Olsen} - 0.051 * Fe_{WH} + 3.81 * Zn_{WH}$	0.618	-0.310	0.298		69.4[a]
$420 + 1.47 * P_{DL} + 3.94 * Zn_{WH} - 68.3 * soil\text{-}pH - 0.046 * Fe_{WH}$	0.746	0.308	-0.304	-0.277	78.8[a]
$551 + 3.51 * P_{AAAc} + 7.14 * Zn_{WH} - 88.2 * soil\text{-}pH - 0.046 * Fe_{WH}$	0.591	0.554	-0.389	-0.274	63.2[a]

Significances: * p < 0.05, ** p < 0.01; chosen equations are marked in bold
[a] no further soil parameters fulfilling the significance criteria for inclusion available

Tab. 15. Calculation of Pwater from other reference methods by simple regression equations (including correlation r between Pwater and respective P-extract, β coefficients for the regressor variable and coefficient of determination R² for the equation)

P_{water} =	r	β	R^2 (%)
7.03 + 0.129 * P_{CAL}	**0.919****	**0.919**	**84.5**
−2.57 + 0.386 * P_{Olsen}	0.783**	0.783	61.1
7.34 + 0.067 * P_{AL}	**0.907****	**0.907**	**82.1**
4.19 + 0.128 * P_{DL}	0.771**	0.771	59.3
6.31 + 0.079 * P_{M3}	**0.934****	**0.934**	**87.1**
8.72 + 0.071 * $P_{AAAc-EDTA}$	**0.915****	**0.915**	**83.7**
10.5 + 0.260 * P_{AAAc}	0.523**	0.523	27.0

Significances: * $p < 0.05$, ** $p < 0.01$; equations with $R^2 > 80\%$ are marked in bold

Tab. 17. Calculation of PAAAc-EDTA from other reference methods by simple regression equations (including correlation r between PAAAc-EDTA and respective P-extract, β coefficients for the regressor variable and coefficient of determination R² for the equation)

$P_{AAAcEDTA}$ =	r	β	R^2 (%)
−20.2 + 1.77 * P_{CAL}	**0.982****	**0.982**	**96.5**
−128 + 4.86 * P_{Olsen}	0.768**	0.768	58.7
−19.0 + 0.940 * P_{AL}	**0.988****	**0.988**	**97.6**
−69.5 + 1.84 * P_{DL}	0.863**	0.863	74.4
20.9 + 1.02 * P_{M3}	**0.942****	**0.942**	**88.7**
−80.7 + 11.8 * P_{water}	**0.915****	**0.915**	**83.7**
58.0 + 3.02 * P_{AAAc}	0.606**	0.606	40.6

Significances: * $p < 0.05$, ** $p < 0.01$; equations with $R^2 > 80\%$ are marked in bold

For a first evaluation and validation of the models, the application of the best regression equations for each pair of methods to the soil samples they were derived from, and then regressing measured data on predicted data is suitable (Ottabong et al., 2009). In this study, a first model evaluation showed promising results with view to the validity of the derived regression equations (data not shown here). The respective coefficients of determination (of transformation regression equation and regression lines for measured vs. predicted values) were almost identical, indicating that both regressions are based on the same basic data set (data not shown here). Thus, an additional data set containing samples which were not used for calculating the regression equations will be used for a statistically sound validation.

Validation of derived regression equations with independent soil data
An independent data set produced from 93 soil samples, which were taken from long term field trials in Mariensee

Tab. 16. Calculation of Pwater from other reference methods by multiple regression equations (including β coefficients for the regressor variables and coefficient of determination R² for the equation)

P_{water} =	β (in the order of parameters given by the equation)					R^2 (%)
2.42 + 0.311 * P_{Olsen} + 0.355 * Zn_{WH} − 1.23 * C_{total} − 0.003 * Fe_{WH} + 0.545 * Cu_{WH}	0.630	0.329	0.224	−0.197	0.140	76.4[a]
24.8 + 0.106 * P_{DL} + 0.454 * Zn_{WH} − 0.00018 * Ca_{WH} − 0.001 * Fe_{WH} − 3.23 * soil-pH − 0.594 * C_{total}	0.640	0.421	−0.059	−0.087 −0.171	−0.109	76.6[a]
40.7 + 0.295 * P_{AAAc} + 0.656 * Zn_{WH} − 6.16 * soil-pH − 1.13 * C_{total}	0.592	0.609	−0.324	−0.207		68.6[a]

Significances: * $p < 0.05$, ** $p < 0.01$; chosen equations are marked in bold
[a] no further soil parameters fulfilling the significance criteria for inclusion available

Tab. 18. Calculation of PAAAc-EDTA from other reference methods by multiple regression equations (including β coefficients for the regressor variables and coefficient of determination R² for the equation)

$P_{AAAcEDTA}$ =	β (in the order of parameters given by the equation)				R^2 (%)
−307 + 3.83 * P_{Olsen} + 4.26 * Zn_{WH} + 38.6 * soil-pH− 0.022 * Fe_{WH}	0.606	0.307	0.160	−0.123	70.0[a]
*126 + 1.64 * P_{DL} + 4.12 * Zn_{WH} − 33.0 * soil-pH − 0.015 * Fe_{WH}*	*0.770*	*0.297*	*−0.135*	*−0.085*	*82.7*
302 + 4.15 * P_{AAAc} + 7.58 * Zn_{WH} − 60.6 * soil-pH − 0.017 * Fe_{WH}	0.643	0.542	−0.246	−0.093	67.7[a]

Significances: * $p < 0.05$, ** $p < 0.01$; chosen equations are marked in bold and, if $R^2 > 80\%$, marked in italic
[a] no further soil parameters fulfilling the significance criteria for inclusion available

and Braunschweig supervised by members of Julius Kühn-Institut, Federal Research Centre for Cultivated Plants, Institute for Crop and Soil Science, was used for validation. The samples were selected to represent a wide range of available P concentrations and soils comparable to those used in the present study. Where necessary, missing soil parameters needed for calculation were analysed in the samples following the procedures described in "Material and Methods". All regression equations displaying coefficients of determination $R^2 > 80\%$ were checked for their performance, i.e. used to predict available soil P in the independent samples. Predicted values were then plotted against the original values measured in the samples and regression equations were calculated to evaluate their quality. Fig. 3 shows the examples of the plots with the best predicted values for each soil test.

Coefficients of determination for the regressions between predicted and measured values ranged from 47 up to 97% (Tab. 19). Out of the 26 equations tested, 8 produced predictions with a coefficient of determination $R^2 < 80\%$, i.e. a transformation of STP using these equations would not yield a statistically satisfactory result.

Conclusion

The soil samples analysed in this study showed a wide range of variation in their elemental composition, soil pH, and organic matter content. All soil P tests applied in this study were significantly correlated with each other. Their extracting force varied considerably, decreasing in the order $P_{AR} > P_{AL}$, $P_{M3} \geq P_{AAAc-EDTA}$, $P_{DL} \geq P_{CAL} \geq P_{Olsen}$, $P_{AAAc} \geq P_W$. Generally, it was possible to transform data
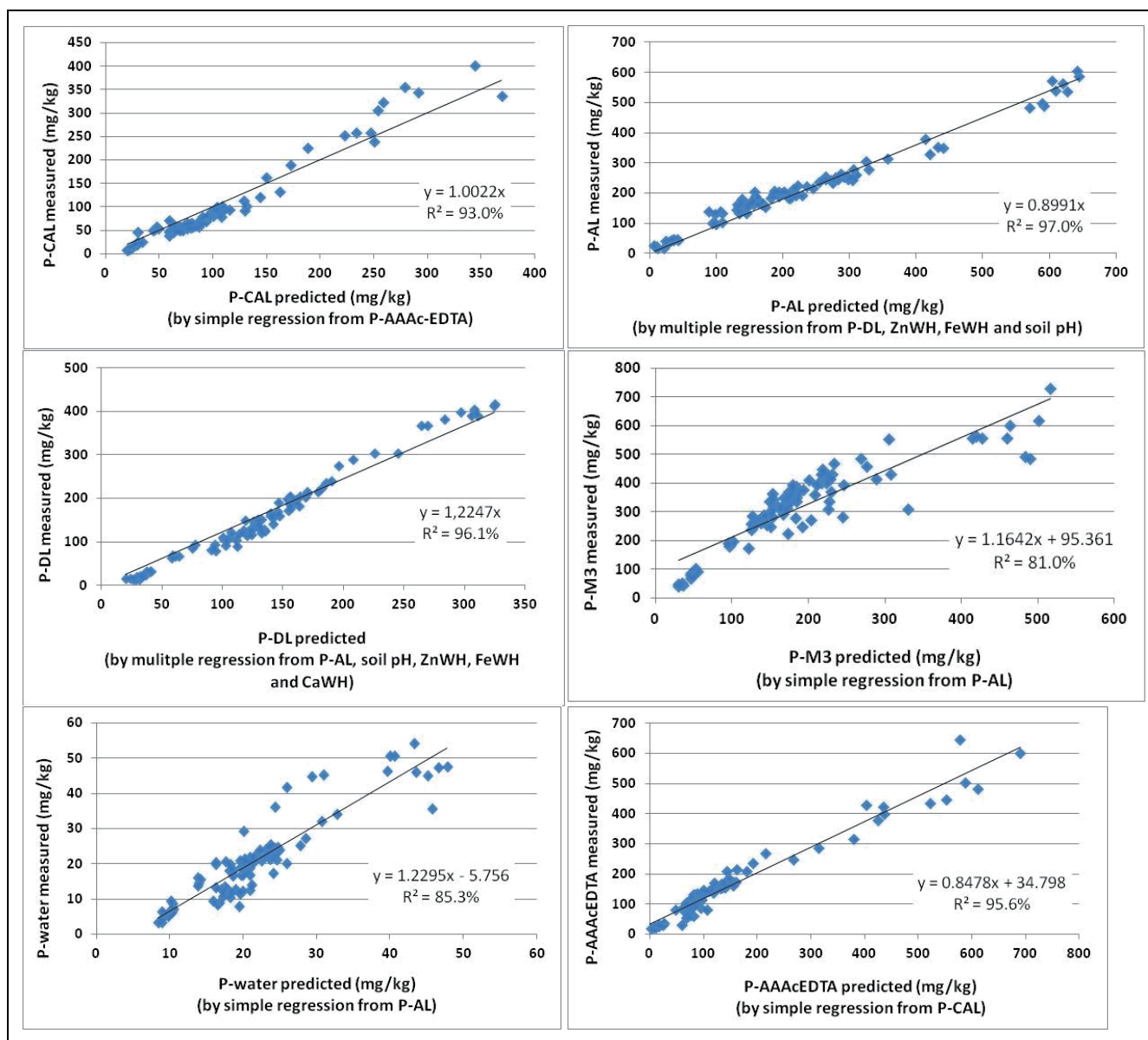

Fig. 3. Regression equations and coefficients of determination for measured (y) vs. predicted (x) P-values (mg/kg air-dried soil) using an independent data set for validation.

Tab. 19. Regression equations for measured versus predicted P concentrations as estimated from the equations selected in Tables 8–18, using an independent data set for validation (cases where $R^2 > 80\%$ are marked in bold)

Parameters used for prediction	Regression measured (y) vs. predicted (x)	R^2
CAL		
AL	**y = 0.9008x**	**83.9**
M3	y = 0.5478x	47.1
AAAc-EDTA	**y = 1.0022x**	**93.0**
Water	**y = 0.9160x**	**80.2**
DL, ZnWH, soil pH	**y = 0.7670x**	**86.5**
AL		
CAL	y = 1.0379x	75.5
M3	y = 0.6419x	78.0
AAAc-EDTA	**y = 1.1141x**	**91.5**
Water	y = 1.0018x	79.0
DL, ZnWH, soil pH, FeWH, CaWH	**y = 0.8991x**	**97.0**
DL		
CAL, soil pH, ZnWH	**y = 1.3472x**	**89.9**
AL, soil pH, Zn, FeWH, CaWH	**y = 1.2247x**	**96.1**
AAAc-EDTA, soil pH, ZnWH, CuWH	**y = 1.3998x**	**93.9**
M3		
CAL	y = 0.8041x + 189	51.2
AL	**y = 1.1642x + 95.4**	**81.0**
AAAc-EDTA	y = 1.0082x + 145	60.2
Water	y = 0.8823x + 162	66.3
Water		
CAL	**y = 1.0122x + 1.47**	**81.0**
AL	**y = 1.2295x + 5.76**	**85.3**
M3	y = 0.8647x − 6.58	66.3
AAAc-EDTA	**y = 1.1943x − 3.04**	**80.3**
AAAc-EDTA		
CAL	**y = 0.8478x + 34.8**	**95.6**
AL	**y = 0.9738x − 18.2**	**93.1**
DL, ZnWH, pH, FeWH	**y = 0.7876x − 1.82**	**92.1**
M3	**y = 0.9738x − 18.2**	**93.1**
Water	**y = 0.8028x + 25.4**	**80.3**

from one soil P test into another one. However, the quality of the resulting values depended strongly on the pair of soil tests at question. Based on the present set of data, values from CAL, AL, M3, AAAc-EDTA and water showed strong correlations and consequently allowed for the calculation of highly significant regression equations with a strong coefficient of determination. While in some cases, simple regressions already yielded a coefficient of determination > 80%, in other cases additional soil parameters such as soil-pH, Zn_{WH}, Fe_{WH}, Al_{WH}, Ca_{WH}, and C_{total} were included in order to achieve this level of accuracy. Extractions with $NaHCO_3$ (Olsen), DL, and AAAc

displayed somewhat weaker correlations. The weakest relations with other methods were found for the AAAc extract. Accordingly, no satisfactory regression equations (i.e. with $R^2 > 80\%$) could be produced for the latter set of methods. A validation based on independent soil data demonstrated that even regression equations derived with a high level of determination ($R^2 > 80\%$) may not necessarily perform well when applied to another set of soil samples. As major obstacles for a reliable transformation of values obtained by different soil tests, differences in chemical composition, acidity and extraction force between methods were identified.

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