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# Assessing the ability of soil tests to estimate labile phosphorus in agricultural soils: Evidence from isotopic exchange

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## ABSTRACT

Efficient phosphorus (P) fertilization strategies are essential for intensive crop production with minimal negative environmental impacts. A key factor in sustainable P use is assessment of the plant available soil P pool using soil P tests. This study determined isotopically exchangeable P after six days of reaction with  $^{33}\text{PO}_4$  (P-E (6 d)) to determine how accurately two commonly used P tests, Olsen and AL (acid ammonium acetate lactate) can quantify the amount of labile P. Soil samples were taken from both highly P-amended and unamended plots at six sites within the Swedish long-term soil fertility experiments. According to P K-edge XANES spectroscopy, the P speciation was dominated by Al-bound P and organic P, with additional contributions from Fe-bound P and Ca phosphates in most soils. The results showed that the AL test overestimated P-E (6 d) by a factor of 1.70 on average. In contrast, the Olsen test underestimated P-E (6 d), with the mean ratios of P-Olsen to P-E (6 d) being 0.52 for high-P and 0.19 for low-P soils. The  $^{33}\text{P}/^{31}\text{P}$  ratio in the Olsen extract of a  $^{33}\text{PO}_4$  spiked soil was closer to that of a  $0.005 \text{ mol L}^{-1}$   $\text{CaCl}_2$  soil extract than the corresponding ratio in the AL extract, suggesting that AL extraction solubilized more non-labile P. In conclusion, the AL and Olsen methods are not suitable for direct quantification of the isotopically exchangeable soil P pool after 6 days of equilibration. However, based on the results, Olsen may be superior to AL for classification of soil P status, due to its even performance for calcareous and non-calcareous soils and lower extraction of non-labile P.

## 1. Introduction

Application of organic or inorganic phosphorus (P) fertilizer is essential to achieve maximum yield in intensive cropping systems, but is often an expensive and time-consuming process. Overuse of fertilizers can lead to economic losses, eutrophication (Schindler, 1977), and exhaustion of limited resources (Cordell et al., 2009). To encourage sustainable P use, many countries have developed P fertilizer management strategies (Jordan-Meille et al., 2012). The purpose is to advise farmers on the amount of P necessary for maximum yield with minimum negative impact. Different countries may implement these strategies in different ways, but P fertilization management is commonly divided into two steps (Jordan-Meille et al., 2012): (1) The readily available amount of P in the soil is determined with a suitable soil P test. (2) The test result is combined with knowledge of crop P requirements and expected yield. This information is then used to calculate the P fertilization requirement. A reliable, rapid and accurate soil

P test that assesses the plant-available P pool is a key factor for sustainable P fertilization.

In Europe > 10 different soil P tests are currently in use (Jordan-Meille et al., 2012). This study focuses on two of the more common types used in northern Europe, acid ammonium acetate lactate extraction (AL method; Egnér et al., 1960) and sodium bicarbonate extraction (Olsen method; Olsen et al., 1954). The AL method is the standard in Belgium (Flanders), Hungary, Lithuania, Norway, Slovenia and Sweden (Jordan-Meille et al., 2012). However, the Olsen method is probably the most commonly used soil P extraction test globally (van Raij, 1998) and is the official soil P test in, e.g., Denmark, England, France, Italy and Spain (Jordan-Meille et al., 2012). The AL and Olsen methods are both designed to quantify plant-available P, but they extract P in different ways. In the Olsen test, extraction with alkaline  $\text{NaHCO}_3$  solubilizes P primarily through addition of  $\text{HCO}_3^-$  ions, which displace phosphate sorbed on particle surfaces. Moreover,  $\text{CaCO}_3$  may be formed, which removes Ca from solution and promotes dissolution of highly soluble

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calcium phosphates (Barrow and Shaw, 1976). The relatively high pH of the extractant (8.5) also promotes desorption of PO<sub>4</sub> from aluminum (Al) and iron (Fe) oxides (Olsen et al., 1954; Barrow and Shaw, 1976). The acidic AL extractant solubilizes adsorbed P but can also dissolve poorly crystalline Fe and Al oxides, and in alkaline soils possible also calcium phosphates. This means that the AL method may overestimate plant-available P in alkaline soils, especially in soils rich in carbonates, as P species not directly available to plants are extracted (Zbiral, 2000; Eriksson et al., 2013). However, even in acidic soils low in carbonates the AL method extracts significantly more P than the alkaline Olsen method (Neyroud and Lischer, 2003; Eriksson et al., 2013). A recent multi-site calibration effort for five established soil tests in long-term field trials in Europe found that the Olsen method explained P related yield responses slightly better than the AL method when evaluated by a Mitscherlich curve, although the differences were rather small (Nawara et al., 2017).

Isotopic exchange of <sup>31</sup>PO<sub>4</sub> with radioactive phosphate ions (<sup>32</sup>PO<sub>4</sub> or <sup>33</sup>PO<sub>4</sub>) can be used to quantify the amount of isotopically exchangeable P (E), which can be regarded as the fraction of P transferable to the soil solution at a given time after addition of the isotope (Frossard and Sinaj, 1997; Hamon et al., 2002). The E value represents the total isotopically exchangeable pool, which is the sum of the isotopically exchangeable pool associated with the solid phase and the isotopically exchangeable P in solution. Several studies have shown that plants grown in soil with added radioactive P isotopes have a specific activity (SA, <sup>33</sup>P/<sup>31</sup>P) equal to that of the soil solution, meaning that plant uptake occurs preferentially from isotopically exchangeable P (Frossard et al., 1994; Morel and Plenchette, 1994). Therefore, a soil P test for predicting plant-available P should preferably extract P only from this isotopically exchangeable pool. This was investigated for eight common soil P tests, including Olsen, on tropical soils by Six et al. (2012). They found that the Olsen test extracted some non-isotopically exchangeable P, and that this fraction varied between soils and was dependent on the fertilizer P dose applied. This agrees with the findings by Demaria et al. (2005), that between 66.0 and 91.7% of the Olsen-extractable P was derived from the isotopically exchangeable P pool when the test was applied on a number of Swiss soils. Extraction with 0.01 mol L<sup>-1</sup> CaCl<sub>2</sub> was assumed to exclusively extract P from the isotopically exchangeable P pool (Six et al., 2012). This means that comparison of the SA in the CaCl<sub>2</sub> extract and the SA in other soil P extracts can be used to assess whether a soil P test samples from the isotopically exchangeable P pool (Six et al., 2012). However, plants may also utilize non-labile P, i.e. P that is not isotopically exchangeable, especially under extreme P deficiency (Pypers et al., 2006).

Many studies have investigated the relationship between Olsen and AL (Lončarić et al., 2006; Ottagio et al., 2009; do Horta et al., 2010).

However, to our knowledge no previous study has used isotopic techniques to investigate the plant availability of P extracted with the AL method. The objectives of this study were thus: 1) to compare the quantity of isotopically exchangeable phosphate after 6 days (P-E (6 d)) with the quantity of phosphate extracted with the AL and Olsen soil P tests, 2) to determine whether the Olsen and AL tests sample only from the isotopically exchangeable pool, and 3) to examine whether the fertilizer P history of the soil affects these results. In addition, further insights into the fate of the spiked <sup>33</sup>PO<sub>4</sub> were sought using five commonly applied chemical extraction methods (water, CaCl<sub>2</sub>, Olsen, AL and ammonium oxalate). The tests were applied to a range of Swedish agricultural soils under different fertilizer regimes in the Swedish long term soil fertility experiments. This set of experiments is a unique field trial as concerns its long time frame (from the 1950s until today), and range of intrinsic soil characteristics (e.g. texture, soil chemistry) representing Swedish agricultural soils (Carlgren and Mattsson, 2001).

## 2. Materials and methods

### 2.1. Soil sampling and soil properties

Topsoil (0–20 cm) was collected from six different sites within the long-term Swedish soil fertility experiments. The experimental design at these sites includes two different crop rotation schemes typical for the area, one intended to mimic a farm with livestock and the other a farm with crop production only. In this study the plots with no-livestock crop rotation were sampled. Among the different fertilizer treatments comprising four phosphorus and potassium (PK) levels (A, B, C and D) and four nitrogen (N) levels (1, 2, 3 and 4), we selected the following two fertilization regimes: no P or K fertilization (A3), and replacement P and K removed by the crop plus 30 kg P and 80 kg K ha<sup>-1</sup> yr<sup>-1</sup> (D3) in the form of triple superphosphate and potassium chloride. Level 3 of N fertilization comprises an average application of 150 kg N ha<sup>-1</sup> year<sup>-1</sup> at two of the selected sites (Fjärdingslöv and Ekebo) and 125 kg N ha<sup>-1</sup> year<sup>-1</sup> at the four other sites (Vreta Kloster, Högåsa, Kungsängen, and Fors). All field treatments are performed in duplicate, leading to a total of 24 topsoil (0–20 cm) plots, all of which were sampled. The sites were chosen to give a range of soil properties such as pH, Fe and Al (hydr)oxide content, and particle-size distribution (Table 1), and they have different soil P concentrations (Table 2). The soil samples were collected in autumn 2015, after harvest and immediately air-dried at 40 °C, hand-crushed with a wooden pestle, and sieved (< 2 mm).

For the A3 and D3 plots on all soils except Högåsa, phosphorus speciation has been investigated previously using P K-edge XANES spectroscopy (Eriksson et al., 2016). The results are summarized

**Table 1**  
General characteristics of the soil at the six long-term field trial sites selected for this study.

Site	Treatment	pH (H <sub>2</sub> O)	Clay	Silt	Sand	Carbonate C	Org C	Al <sub>ox</sub>	Al <sub>py</sub>	Fe <sub>ox</sub>	Fe <sub>py</sub>
			(% of mineral fraction)			(% of soil)		(mmol P kg <sup>-1</sup> )			
Ekebo	A3	6.98	15.3	35.7	49.1	0.01	2.42	71.0	39.5	34.7	17.8
	D3	7.06	14.9	35.6	59.6	0.01	2.34	77.5	46.7	39.2	22.5
Fjärdingslöv	A3	6.81	17.6	26.4	56.1	0.01	1.23	30.3	6.00	32.1	5.80
	D3	6.79	16.9	25.0	58.1	0.01	1.84	29.9	7.57	27.1	10.0
Vreta Kloster	A3	6.84	43.5	48.9	7.60	0.01	1.87	60.0	10.3	29.0	4.50
	D3	6.84	49.9	41.0	9.15	0.01	2.02	66.2	10.2	34.8	7.80
Högåsa	A3	5.97	6.10	16.8	77.2	0.01	1.92	72.1	45.0	53.2	23.2
	D3	6.41	8.10	22.7	69.3	0.01	2.06	77.9	49.7	52.3	28.7
Kungsängen	A3	6.58	55.4	41.0	3.56	0.08	2.23	55.0	4.96	152	18.4
	D3	6.50	54.0	42.5	3.54	0.03	2.07	55.8	9.31	163	24.2
Fors	A3	8.32	16.3	57.8	26.0	0.47	1.56	31.5	5.23	28.7	2.06
	D3	7.83	14.4	59.1	26.6	0.77	1.71	29.2	5.00	30.9	3.47

Al<sub>ox</sub> = oxalate-extractable Al, Al<sub>py</sub> = pyrophosphate-extractable Al, Fe<sub>ox</sub> = oxalate-extractable Fe, Fe<sub>py</sub> = pyrophosphate-extractable Fe. Carbon analysis was performed by dry combustion using a LECO TruMac CNS Analyzer.

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