Assessment of fertilizer P residues in a calcareous Vertisol*

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Abstract

The amounts of available P in a Typic Pellustert containing different levels of residual fertilizer P were assessed in a field experiment and chemically with cation-anion-exchange resin, using: (i) P desorbed after 1 extraction, (ii) cumulative P from 8 extractions [Resin8-P] and (iii) the asymptote of the extraction curve. P exchangeable to ^{32}P, Olsen-P and P extracted by dilute salt solution (0.03 M KCl) were also measured. P available to sorghum in 1989 was strongly influenced by fertilizer P applied in 1988, but not by P applied in 1987. Olsen-P and Resin8-P both reflected well the effects of residual P on yield and P uptake and could therefore be used to predict available residual P. The amounts of Resin8-P were much larger than Olsen-P, so it was easier to observe the differences between soil treatments, but Olsen-P gave the better correlations with grain yield and P uptake.

Multiple regressions of crop yield or P uptake with amounts of P applied in 1987 and 1988 gave the best correlations, without the need for practical work. Thus, the development of an appropriate model based on records of P fertilization is seen as the most effective way to predict availability of residual P. Assessments related to the P intensity and desorption rate using resin were not able to improve predictions of available P, indicating that within one soil the quantity of residual P is most important, but for comparisons between soils, account will need to be taken of differences in soil characteristics.

Introduction

Until recently, lack of phosphate was not generally recognised as a major limitation to crop production on tropical Vertisols and there was relatively little chemical investigation of the nature of their P [10]. Fertilizer P use on Vertisols in developing countries is becoming more common, so it is important that the residual value should be allowed for. A chemical extractant for available soil P should, therefore, correctly reflect the value of the fertilizer residues.

It appears that P is more freely available to crops on calcareous Vertisols than is suggested by the usual calibration of Olsen-P [18], so it was considered important to compare abilities to assess available residual P for some chemical methods in this soil type.

For long-term (up to 100 year old) experiments on neutral and calcareous soils of the UK, Olsen-P provided a rapid and reliable method for assessment of fertilizer P residues [12]. Isotopically exchangeable phosphate and P extracted by dilute $CaCl_2$ solution also increased in response to fertilizer P residues. The P extracted by water or a dilute salt solution is very loosely held and the amounts extracted in water are greatly increased in the presence of anion-ex-

change resin, which acts as a sink for phosphate. Because of the mild nature of extraction by resin, it can also be used to assess rates of P desorption, which may improve the prediction of available P [4]. It has been suggested that P desorbed by anion-exchange resin is a useful measure of the availability of residual P [15]. Great variations exist in the conditions for P desorption with resin and maximum extraction times range from 2 hours [4] to 21 days [16]. Various equations have been used to describe the time course of desorption, such as those following:

$$P = r\sqrt{T} + b \qquad \text{Reference [4]}$$

$$dP/dT = abT^{(b-1)} \qquad \text{Reference [5]}$$

$$P = \frac{k(P_{max})}{1 + kT} \qquad \text{Reference [16]}$$

$$P = B - (B - a).\exp(-kT) \qquad \text{Reference [11]}$$

where P represents phosphate extracted at time T, and a, b, B, k, r and P_{max} are constants, the meaning of which may vary between equations. In consequence, mechanistic interpretations vary between authors. The overall rate of P desorp-
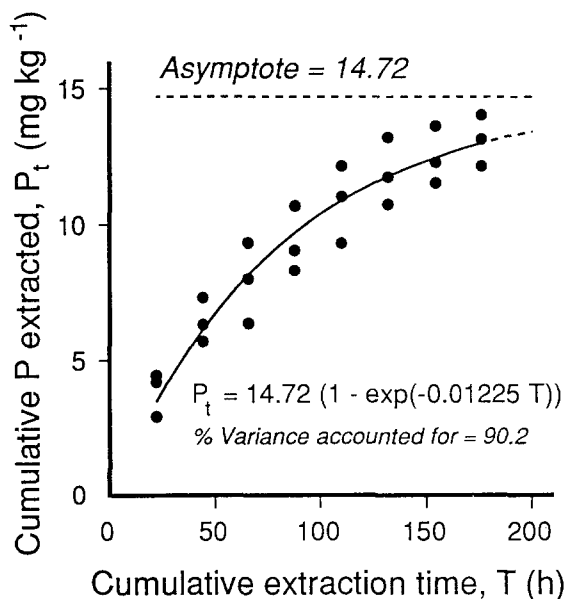
tion depends on two stages: (i) the release of P from soil to solution and then (ii) the uptake of P by resin from solution, and in practice a true value for the first rate cannot be determined [1].

However, our major objectives in this work were (i) to assess the value of fertilizer P residues by sorghum in the field, and Olsen-P and ^{32}P, and (ii) to assess the ultimate amount of P that could be removed by a sequence of resin extractions, by means of the asymptote of a fitted curve (Fig. 1). A monobed mixed cation-anion-exchange resin (CAER) was used, since it gives a better measure of plant available P than anion-exchange resin alone [5]).

Materials and methods

Soil samples and field experiment

The soil used was of the Kasireddipalli series, a benchmark Vertisol (Typic Pellustert) at the ICRISAT Center, (pH 8.3; 5.6% $CaCO_3$). Samples were drawn from treatments of a field experiment commenced in 1987 at the ICRISAT Center, the objective of which was to assess the residual value of fertilizer P to sorghum in the field. Agronomic assessments of the P residual value will be reported separately.

In order to compare fresh and residual P directly in each season, a split-plot design was adopted in which diammonium phosphate was applied at 4 rates (0, 10, 20 and 40 kg P ha^{-1}) in May 1987. In May 1988, each plot was split into 4 sub-plots and the 4 rates of fresh P applied in the sub-plots. Finally, in May 1989, the sub-plots were themselves further split to give sub-sub-plots (each 4 m $\times$ 4 m) for the application of more fresh P at the same 4 rates. One crop of sorghum (variety CV.CSH6) was grown each year in the rainy season from June to September.

In April each year, soil samples were taken from the surface (0-150 mm) horizon of each sub-sub-plot in each of the four replicate blocks, air-dried, ground and passed through a 2 mm sieve for chemical analysis. In this work, soil samples taken in April 1989 were analyzed, so they contained residues of P applied in 1987 and 1988. The soil chemical analyses were then compared with sorghum yield and P uptake in

Fig. 1. Example of the relationship between cumulative P extracted by CAER (P_t) and extraction time (T), for soil of replicate block 1 with 1987-P = 0 kg ha^{-1} and 1988-P = 10 kg ha^{-1}.

Asymptote = 14.72

P_t = 14.72 (1 - exp(-0.01225 T))

% Variance accounted for = 90.2

the 1989 sub-sub-plots without fresh fertilizer that year.

Olsen-P

0.5 M NaHCO$_3$ solution was adjusted to pH 8.5, and 100 ml of solution was added to 5 g soil and shaken for 30 minutes at 25 °C. Phosphate extracted was measured colorimetrically by the ascorbic acid/molybdenum blue method [15].

Isotopically exchangeable and KCl extractable-P

Soil (1.5 g) was weighed into glass centrifuge tubes in duplicate and 0.03 M KCl containing 0.05% formaldehyde (29 ml) added. The suspension was shaken end-over-end (1 rpm) for 22 h, carrier free H$_3$^{32}PO$_4$ solution (1 ml) was added to each tube, and the tubes were shaken for a further 22 h. After centrifuging the suspensions for 5 minutes at 2500 rpm, the supernatant liquid was decanted and filtered through Whatman No. 1 paper. The concentration of unlabelled phosphate in the supernatant liquid was measured colorimetrically [14] and the concentration of ^{32}P by Cerenkov counting in a liquid scintillation counter. Unlabelled phosphate desorbed (^{31}P$_1$) was calculated from the concentration of unlabelled P. The total isotopically labile P, which equals the sum of isotopically exchangeable P on the soil and P desorbed was calculated from the following equation [17]:

Isotopically labile P = ^{31}P$_1$/f

where f equals the ratio of ^{32}P added to ^{32}P remaining in solution, (^{32}P$_a$/^{32}P$_1$). In a preliminary experiment, the more common background electrolyte of 0.01 M CaCl$_2$ was used, but the amounts of P desorbed and exchangeable P were negligible. This was attributed to Ca phosphates remaining precipitated after the addition of excess Ca in a highly calcareous soil; it may be noted in a calcareous soil of the USA, P extracted by anion-exchange resin was reduced, sometimes greatly so, if Ca salts were used in the extracting solution, compared to water alone or Na salts [2]. When 0.01 M CaCl$_2$ was substituted by KCl of the same ionic strength (0.03 M), significant desorption of P was found. In addition to the exchangeable P data, amounts of unlabelled P extracted by KCl were also examined.

P extracted by cation-anion exchange resin

To contain the resin, porous bags were sewn [20] using 400 μm polyester mesh (Nybolt brand, type 40-420 ASTM, Schweizerische Seidengazefabrik AG, Zürich). Amberlite MB-1 resin was sieved and 5 ml of resin beads, >600 μm, were trapped in each bag. The resin was then converted to the recommended bicarbonate-chloride form [21]. The bags were treated in succession with 0.5 M HCl first, then twice with 0.5 M NaHCO$_3$, for 1 h with occasional stirring in each solution, and were finally rinsed with water.

A successive extraction method [11] was used, in order to obtain information on the amount and rate of P desorption. Soil samples (4 g) in triplicate were weighed into wide mouth plastic screw top bottles (100 ml nominal capacity), water (100 ml) and one resin bag were added, and the bottles shaken end-over-end (1 rpm) overnight (22 h) at 30 °C. Next, each bag was lifted from its bottle and held there briefly to allow the suspension to drain back to the bottle, rinsed with water (retaining these washings), excess water blotted with tissue paper, and the bag put in another bottle. A freshly regenerated (bicarbonate-chloride form) resin bag was placed in the soil suspension, and these suspensions returned to the shaker for another overnight period. The retained washings were dried overnight at 100 °C, and to find the amount of soil, approximately 0.1 g, lost from each bottle with the resin bag. A total of 8 extractions were made from each soil sample. Little additional P was extractable by resin after 8 extractions and this procedure gave a convenient work schedule.

To recover phosphate from the resin, 0.5 M HCl (50 ml) was added to the bottle with the bag, the bottle was swirled at frequent intervals for 20 minutes to release CO$_2$ gas, and then capped and shaken for 2 h. The acid extract was decanted and filtered (Whatman No 5 paper) into a 100 ml flask. The bag was shaken with a second aliquot (50 ml) of HCl to complete the recovery of P and the two aliquots of acid were combined. Phosphate in the extract was measured colorimetrically [14]. In calculating the P desorbed during each 22 h period, allowance was made for the loss of soil from each bottle. Data for cumulative extracted P (P$_t$) and cumulative

extraction time (T) were fitted to the following simple exponential equation:

$$P_t = a(1 - \exp(-kT))$$

The fit of this equation was always highly significant, and in 79% of the soil samples, >80% of the variance was accounted for; an example of the extraction curve is given in Fig. 1. Fitted parameter a equals the asymptote of the extraction curve, and is considered to be a measure of the ultimate amount of P extractable by resin, while parameter k describes the rate of desorption; an exponential equation may be derived from first principles if it is assumed that desorption follows first order kinetics.

The amounts of soil P measured by each method were designated as follows: Olsen-P ($NaHCO_3$); KCl-P ($0.03\,M$ KCl); Exch-P (Isotopically labile); Resin1-P (first extraction by CAER); Resin8-P (cumulative P extracted up to the eight extraction by CAER). The following abbreviations are also used: Resin-a (asymptote of the fitted equation); Resin-k (constant k of the fitted equation); 1987-P (fertilizer P applied in the field in 1987); 1988-P (fertilizer P applied in the field in 1988). Olsen-P was measured at the ICRISAT Center and all other measurements were made at Reading.

Results

Response of extractable P to 1987-P and 1988-P
The amounts of extractable P differed greatly between the methods, the overall mean values (in $mg\,kg^{-1}$) being 31.88 (Resin-a), 29.01 (Resin8-P), 8.66 (Resin1-P), 8.83 (Exch-P), 1.15 (Olsen-P) and 0.102 (KCl-P). In general, there were significant positive effects of 1988-P on extractable P, but no significant effects of 1987-P (Tables 1 and 2). For Resin1-P there appeared to be a significant effect of 1987-P, but this was the result of particularly high values for

Table 1. Extractable P and exchangeable P in soil sampled in 1989 and containing residues of fertilizer P applied in 1987 and 1988

1987-P ($kg\,ha^{-1}$)	1988-P ($kg\,ha^{-1}$)				
	0	10	20	40	mean
Olsen-P ($mg\,kg^{-1}$)					
0	0.67	1.00	1.23	1.81	1.18
10	0.63	0.75	1.18	1.89	1.11
20	0.81	0.94	1.21	1.61	1.14
40	0.75	0.90	1.17	1.86	1.17
mean	0.71	0.90	1.20	1.79	
Values of F ratios: (a) 0.97, (b) 72.67***, (c) 0.84					
KCl-P ($mg\,kg^{-1}$)					
0	0.078	0.082	0.107	0.128	0.099
10	0.088	0.080	0.098	0.130	0.099
20	0.098	0.090	0.090	0.132	0.103
40	0.083	0.109	0.117	0.127	0.109
mean	0.087	0.090	0.103	0.129	
Values of F ratios: (a) 1.42, (b) 11.69***, (c) 0.77					
Exch-P ($mg\,kg^{-1}$)					
0	7.29	7.20	8.94	10.15	8.39
10	9.11	8.02	8.59	9.88	8.90
20	9.09	8.14	7.92	10.53	8.92
40	7.82	9.20	9.47	9.92	9.10
mean	8.33	8.14	8.73	10.12	
Values of F ratios: (a) 0.29, (b) 2.86*, (c) 0.49					

(a) Effect of P applied in 1987
(b) Effect of P applied in 1988
(c) Interaction 1987-P × 1988-P
*,***; denote significance at the 5 and 0.1% levels respectively.

Table 2. Amounts of extractable P and parameters for P desorption by CAER, in soil sampled in 1989 and containing residues of fertilizer P applied in 1987 and 1988

1987-P (kg ha^{-1})	1988-P (kg ha^{-1})				
	0	10	20	40	mean
	Resin1-P (mg kg^{-1})				
0	5.64	6.11	8.57	11.62	7.98
10	7.22	6.74	10.37	14.52	9.71
20	5.59	5.85	8.95	10.63	7.76
40	7.54	8.10	8.45	12.58	9.17
mean	6.50	6.70	9.09	12.34	
Values of F ratios: (a) 4.52*, (b) 24.46***, (c) 0.57					
	Resin8-P (mg kg^{-1})				
0	21.83	20.68	28.83	39.39	27.68
10	23.22	22.26	31.28	39.62	29.10
20	22.75	23.18	29.27	36.58	27.94
40	27.83	26.42	32.10	38.95	31.32
mean	23.91	23.13	30.37	38.64	
Values of F ratios: (a) 2.25, (b) 24.78***, (c) 0.26					
	Resin-a (mg kg^{-1})				
0	25.12	22.55	31.30	41.63	30.15
10	24.33	24.22	34.94	40.16	30.91
20	28.78	26.43	31.30	40.29	31.70
40	31.74	27.82	39.82	41.24	35.16
mean	27.49	25.25	34.34	40.83	
Values of F ratios: (a) 1.38, (b) 12.92***, (c) 0.31					
	Resin-k				
0	0.0126	0.0137	0.0154	0.0162	0.0145
10	0.0154	0.0133	0.0161	0.0185	0.0158
20	0.0122	0.0141	0.0155	0.0158	0.0144
40	0.0133	0.0160	0.0135	0.0192	0.0155
mean	0.0134	0.0143	0.0151	0.0174	
Values of F ratios: (a) 0.41, (b) 2.74, (c) 0.40					

(a) Effect of P applied in 1987
(b) Effect of P applied in 1988
(c) Interaction 1987-P × 1988-P
*, ***; denote significance at the 5 and 0.1% levels respectively.

10 kg ha^{-1} of 1987-P in the presence of 20 or 40 kg ha^{-1} of 1988-P (Table 2) and as these values were higher than the corresponding values for 40 kg ha^{-1} of 1987-P, the result was regarded as unreliable. Olsen-P showed the clearest response to 1988-P, as judged by size of the F-ratio (Table 1). The assessments by Resin1-P, Resin8-P, Resin-*a* and KCl-P did not respond so strongly to 1988-P, while Exch-P and Resin-*k* were insensitive to 1988-P. The highly significant effect of 1988-P and lack of clear effect of 1987-P seen in analyses of variance for most chemical measurements correctly reflect the relative importance of 1988-P and the small effect of 1987-P to the sorghum (Table 3).

Relationships between yield or P uptake and fertilizer or measured soil P

Linear relationships were a satisfactory description for all the relationships between yield or P uptake (PU) and measured soil P, and the simple correlation coefficients are given in Table 4. For the chemical methods, grain yield and P uptake were most closely related to Olsen-P (Fig. 2), followed by Resin8-P. Stalk yield was most closely correlated to Resin8-P, followed by Olsen-P. The other assessments of soil P were less closely related to available P. In particular, it was observed that measurements of Exch-P showed great variability, leading to a low correlation coefficient. Resin-*k* was significantly corre-

Table 3. Sorghum grain yield and total P uptake by grain stalk in 1989, in response to residues of fertilizer P applied in 1987 and 1988

1987-P (kg ha^{-1})	1988-P (kg ha^{-1})				
	0	10	20	40	mean
	Grain yield (kg ha^{-1})				
0	314	385	1043	2299	1010
10	484	714	1090	2261	1137
20	322	945	1382	1987	1159
40	820	972	1788	2679	1565
mean	485	754	1326	2307	
Values of F ratios: (a) 1.68, (b) 79.08***, (c) 2.20*					
	P uptake (kg ha^{-1})				
0	1.46	1.77	3.92	4.40	3.39
10	1.97	2.40	3.02	6.57	3.49
20	1.75	3.22	4.20	5.17	3.59
40	2.60	2.94	5.25	7.50	4.57
mean	1.94	2.58	4.10	6.41	
Values of F ratios (a) 1.72, (b) 72.01***, (c) 1.83					

(a) Effect of P applied in 1987
(b) Effect of P applied in 1988
(c) Interaction 1987-P × 1988-P
*, ***; denote significance at the 5 and 0.1% levels respectively.

Table 4. Simple correlation coefficients (r values) of 1989 sorghum yield and P uptake, with fertilizer P, extractable P and P desorption parameters

Soil P assessment	Grain	Stalk	Total yield	P uptake
1987-P	0.251	0.206	0.233	0.221
1988-P	0.824***	0.659***	0.755***	0.807***
Olsen-P	0.799***	0.663***	0.747***	0.808***
Exch-P	0.374**	0.344**	0.372**	0.362**
KCl-P	0.684***	0.621***	0.675***	0.667***
Resin1-P	0.710***	0.552***	0.640***	0.676***
Resin8-P	0.778***	0.702***	0.764***	0.744***
Resin-*a*	0.652***	0.656***	0.685***	0.620***
Resin-*k*	−0.262*	−0.082	−0.156	−0.266*

*, **, ***; denote coefficients significant at the 5, 1, 0.1% levels respectively.

lated with grain yield and PU, but only at low levels, with 6.4 and 6.5% of the variance accounted for, respectively. In stepwise multiple regression analysis, neither it nor KCl-P significantly improved the fit of yield or PU to Olsen-P or Resin8-P.

1988-P gave a higher correlation coefficient with grain yield than any chemical assessment of soil P (Table 3), while the correlations between 1988-P and stalk yield or PU were the same as between Olsen-P and stalk yield or PU. 1987-P alone was not significantly correlated with yield or PU, but it gave a significant improvement in fit in multiple regressions of grain, stalk or PU

with 1988-P. The regression equations were as follows:

Grain Yield = 46.8 × 1988-P + 13.6 × 1987-P + 167
(SE:)   (±3.83)***   (±3.83)**   (±111)
(72.0% variance accounted for)

PU = 0.116 × 1988-P + 0.031 × 1987-P + 1.21
(SE:)(±0.0106)***   (±0.0106)***   (±0.308)
(67.7% variance accounted for)

The ratio of the regression coefficients (1988-P ÷ 1987-P) was 3.4 for grain yield and 3.7 for PU, suggesting that residues of 1988-P were about 3.5
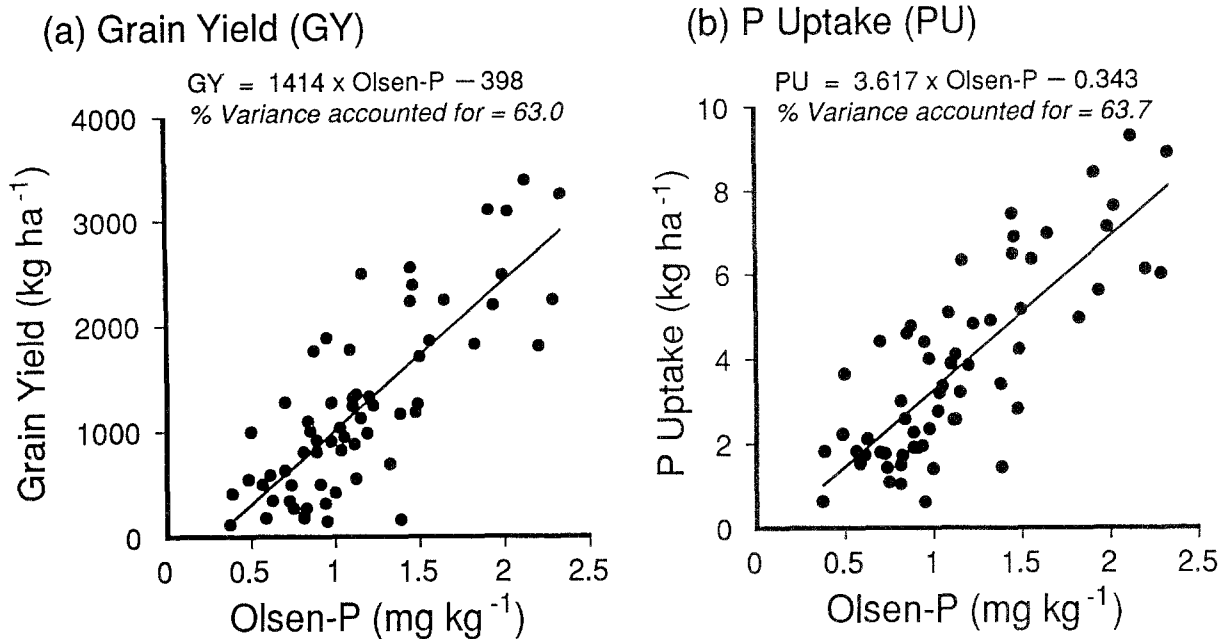
(a) Grain Yield (GY)

$$GY = 1414 \times \text{Olsen-P} - 398$$
% Variance accounted for = 63.0

(b) P Uptake (PU)

$$PU = 3.617 \times \text{Olsen-P} - 0.343$$
% Variance accounted for = 63.7

Fig. 2. Relationships between (a) sorghum grain yield and (b) P uptake by sorghum and Olsen-P in a Vertisol with varying amounts of P fertilizer residues.

about 3.5 times as effective as residues of 1987-P. Although this interpretation indicates that 1987-P had some effect on sorghum yields, the analyses of variance showed showed that it was not enough to increase yield significantly.

Discussion and conclusions

Sequential extraction with resin
On average, Resin8-P, the cumulative P desorbed after 8 extractions with CAER was over three times greater than Resin1-P, showing that a large pool of phosphate is quite easily desorbed, provided that the soil solution P concentration is maintained at a low concentration. This agrees with the results of electroultrafiltration (EUF) extractions on some Indian Vertisols [19]. The additional P desorbed in the last of the CAER extractions was small in most samples and it had been thought that Resin-*a* might predict available P better than Resin8-P because it may be viewed as the maximum amount of CAER extractable P. A similar asymptotic estimate of resin extractable P was the best chemical assessment of available P in about 500 tropical soils [16], although most soils were acid. But in this

work, Resin-*a* did not correlate with yield and P uptake as well as Resin8-P, or even Resin1-P for grain or PU. Resin-*k* was changed little by P residues in the soil suggesting that the amounts of fertilizer used did not alter the soil characteristics which control desorption rate. Nor did Resin-*k* improve prediction of available P in conjunction with P quantity measurements. If different soils were to be compared, adjustment for Resin-*k* might have a larger effect, by analogy with the improvement in prediction of available P given by P buffer capacity, which was greater in a heterogeneous group of soils than a homogeneous group [9]. Thus the work of making the additional extractions with resin did not seem justified for this soil.

Comparisons of different extractants
Resin1-P was of similar magnitude to Exch-P, in agreement with other work [2,3,20], provided an equilibration time of around 24 h is used for both resin and ^{32}P methods. Much less P was extractable as Olsen-P (about 15% of Resin1-P). Although available P was adequate for good yields with 40 kg ha^{-1} of 1988-P (Table 3), Olsen-P was less than 2 mg kg^{-1} in every treatment (Table 1), a value normally associated with P deficiency [7].

This suggested that Olsen-P underestimated the amounts of potentially available P. The numerically small values and differences in Olsen-P mean that calibration of the method for practical assessment of P in fields is difficult because an individual result is unlikely to be sufficiently precise. Nevertheless, the percentage differences in Olsen-P caused by the residual P treatments were larger than for the other methods, so variations in Olsen-P predicted better the crop yield. The choice of the best method lies between (i) Olsen-P, which gives the most accurate representation of differences between available residual fertilizer P and (ii) Resin8-P, for which the larger amount of P extracted makes it easier to observe the differences and to calibrate the method. Normally, for practical reasons, Olsen-P would be recommended since it is much quicker and easier to measure.

The very small values for KCl-P would make reliable calibration a problem with this reagent also, although KCl-P reflected the highly significant crop responses due to 1988-P. However, P extracted by water or dilute salts is generally used to represent the soil phosphate intensity i.e. concentration of P in soil solution [8,13,15]. KCl-P was not as well correlated with yield and crop P uptake as Olsen-P or Resin-P, suggesting that the size of the labile P pool was more important than the P intensity. Variations in P requirement have been shown to be related to measurements of P intensity in comparisons between different soils [13], but the application of up to $80 \, \text{kg} \, \text{ha}^{-1}$ fertilizer P over 2 years would not be expected to cause large differences in the quantity-intensity relationships, such as can be found between completely different soils. Thus, it is reasonable that the intensity measurement was not influential in this work.

Prediction through modelling of residual value
The best prediction of available P was obtained from multiple regression with the amounts of fertilizer P applied each year. This suggests that for prediction of available fertilizer residues, modelling of the residual effects from records of fertilization is likely to be more effective than direct assessment by chemical extractants. Without the need for practical work, model calculations would also be much cheaper to use. The

equations given above are based on a relatively short period. Under a longer period of fertilization, the balance of P inputs and offtakes for the soil may be different, as the amounts of residual P increase. Therefore the development of an appropriate mathematical model to describe the residual availability of P in relation to the soil P turnover is seen as the best way to assess residual value. Information on the most appropriate chemical extractant is still desirable though, to cater for situations where the history of fertilization is lacking.

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