

## Environmental aspects of soil phosphorus testing

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Soil phosphorus testing in Ireland uses Morgan's reagent from samples taken to 10 cm depth for agronomic recommendations. However, its suitability as an environmental indicator has been questioned in terms of sample depth and extraction solution. Seven grassland sites were sampled to depths of 2, 5 and 10 cm and extracted for Morgan's P, the standard agronomic test, as well as iron-oxide impregnated paper strip P (FeO-P), calcium chloride extractable P (CaCl<sub>2</sub>-P) and water soluble P (WSP), all proposed as environmental soil tests. Extractable soil P decreased with increasing sample depth, as did variances in each test, such that, 2 cm samples had highest concentrations and variances. The current standard sample depth (10 cm) was linearly related to corresponding data from samples taken to 2 and 5 cm, indicating that surface soil P can be consistently estimated from the current standard depth. When soil tests were compared with dissolved reactive P (DRP) in overland flow collected from two field sites, certain soil tests were better indicators of P loss than others. The relative difference in Morgan's P values at the standard sample depth (10 cm) was reflected in the relative difference in P loss between the two sites. Average values of DRP collected from two sites ranged from 0.032 to 0.067 mg/l at the low P site and 0.261 to 0.620 at the high P site. Average DRP values from the high P site and maximum DRP values from the low P site were simulated using water-soluble P extraction at water to soil ratios 5 to 250 l/kg. In this study, Morgan's P to 10 cm gave a good indication of the relative difference in DRP loss between the two sites.

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### Introduction

Phosphorus loss from agriculture is of great concern and has been estimated to account for almost half of river pollution in Ireland (McGarrigle and Clenaghan, 2004). The accumulation of soil P beyond crop requirements is typically a major source of agricultural non-point pollution, and high soil-test P levels have been linked with high losses of P from grassland sites in Ireland (Kurz *et al.*, 2004), and elsewhere (Heckrath *et al.*, 1995; Pote *et al.*, 1996). An obvious indication of soil-P status is the agronomic soil-P test, traditionally used as a guide to optimum fertility levels in soils and a basis recommended for fertilizer-P applications and nutrient management (Sims *et al.*, 2000). More recently, soil-P tests have been used as an environmental indicator of potential losses of P from soil to water at watershed scale (Heathwaite, 1997) and field scale (Lemunyon and Gilbert, 1993). This has led to advancements in soil-P testing with sampling and analytical techniques designed to simulate P loss to water as opposed to P uptake by plants.

Techniques simulating the process of P desorption from soil to solution are favoured over the traditional chemical extraction methods using buffered acid and alkaline solutions that dissolved the plant-available P fractions. Iron-oxide-impregnated paper strip (FeO-P) and anion-exchange methods simulate long-term desorption by using a near-infinite sink for P whilst other extractants such as distilled water and dilute calcium chloride, have been used to represent more readily desorbable forms of P in soil. These methods have been related to agronomic soil-P tests in Ireland (Daly, Jeffrey and Tunney, 2001; Herlihy *et al.*, personal communication) and elsewhere (Sims *et al.*, 2000). In terms of potential losses, these environmental-P tests have been

shown to relate well to the concentration of dissolved reactive P (DRP) in surface runoff at plot-scale using rainfall simulators (McDowell and Sharpley, 2001; Torbert *et al.*, 2002; Pote *et al.*, 1996). Furthermore, P extracted by dilute calcium chloride (CaCl<sub>2</sub>-P) and water-soluble P (WSP) have been used as an approximation of DRP in surface runoff since they represent soil solution concentrations and produce change points when plotted against agronomic or conventional soil-P tests such as Olsen, Mehlich or Morgan's P (Hesketh and Brookes, 2000; McDowell and Sharpley, 2001; Kleinman *et al.*, 2000). Whilst the existence of change points provide a threshold soil-P concentration above which potential release of soil P to water increases, some studies have not found change points as relationships between WSP and CaCl<sub>2</sub>-P and soil-test P remained linear (Torbert *et al.*, 2002; Daly *et al.*, 2001; Herlihy, personal communication).

The effective depth of interaction (EDI) between overland-flow water and surface soil has been shown to occur in the upper few centimeters of soils (Ahuja *et al.*, 1981) and can vary according to rainfall intensity, slope and cover (Sharpley, 1985). Given the stratification of P concentration in grassland soils (Culleton *et al.*, 1999; Humphreys, Tunney and Duggan, 1998) a variety of authors have proposed changing sample depth to enhance environmental interpretation (Mulqueen, Rogers and Scally, 2004; Sharpley, Syers and Williams, 1978). However, Schroeder *et al.* (2004) reported that sample depth (2, 5 and 10 cm) had no effect on the relationship between soil P concentration and P concentration in runoff in a rainfall simulator study. In addition, Torbert *et al.* (2002) found that, whilst soil samples taken to 2.5 cm were highest in P concentration, they showed

more variability when compared to samples taken to 5 or 15 cm.

Environmentally orientated soil-P tests have been designed to simulate the process of desorption of P from soil to solution and modeling P desorption (Sharpley, 1983; Barrow, 1979, 1983) has identified soil-P concentration and water-to-soil ratio as factors controlling amounts of P desorbed from soils. As extract ratios increase, quantities of P desorbed to solution increase (Sharpley, 1983) and wide extract ratios (10,000) have been related to measurements of surface saturation of P in soils (Delgado and Torrent, 2001). Extract ratios have been used to predict desorption of P to water for a wide range of soils (Torrent and Delgado, 2001), however, water-to-soil ratios were shown to affect threshold soil-test P levels (or change points) and linear relationships between soil P and P loss to surface waters, using wide extract ratios, have been reported (Koopmans *et al.*, 2002). In terms of edge-of-field losses, Yli-Halla *et al.* (1995) reported soil-P extracts, obtained at water-to-soil ratios between 250 and 100 l/kg, that corresponded to flow-weighted mean DRP concentrations in overland flow and used the extracted concentrations to describe the range of losses measured.

In Ireland, agronomic soil-P testing uses Morgan's reagent, a buffered acetate-acetic acid (pH 4.8), in a 1:5 soil to solution ratio (Peech and English, 1944; Byrne, 1979), to dissolve plant-available P compounds. Since soil-P testing began in Ireland in 1948, Morgan's reagent has been used, on soil sampled to 10 cm, oven-dried at 40 °C and sieved (2 mm), as an indicator of plant-available P in soils and thus for fertilizer recommendations. In recent times however, its use has extended to include potential losses of P from soil to water and has been found to be highly correlated with

other agronomic (Humphreys, Tunney and Duggan, 2001; Foy *et al.*, 1997) and environmental tests, such as WSP, CaCl<sub>2</sub>-P and FeO-P (Daly *et al.*, 2001; *et al.*, Humphreys *et al.*, 1998).

In this work, aspects of soil-P testing, such as extracting reagent, sample depth, water-to-soil ratio, and sample pretreatment were evaluated at laboratory scale against edge-of-field losses of DRP in overland flow from two field sites. The objective was to place the standard agronomic soil-P test in an environmental context, as an indicator of risk, by evaluating the relevance of sample depth, choice of extractant, water to soil ratio and sample pretreatment.

## Methods

### *Soil sampling and analysis*

Soil samples were taken from seven grassland sites on the Johnstown Castle Estate in south-east Ireland (lat 52°N; mean annual rainfall 1022 mm; mean annual temperature of 9.6 °C). The sites were selected to provide a range of soil-test P levels and were taken from study areas within the Johnstown Castle Estate known locally as Warren (sites 1 and 2), Cowlands (site 3 and 4) and Dairy (sites 5 to 7). The sites were part of on-going beef and dairy systems and were used in this study primarily due to the range of soil-P levels, from below optimum to excessively high. The soil type at the Warren is described as a poorly-drained gley soil with sandy loam topsoil over a layer of sandy loam and loam, with heavy gleying evident at all horizons (Kurz *et al.*, 2004). At the Cowlands, the soil is described as a poorly-drained gley with a sandy loam topsoil overlying a loam with a sandy loam lens imbedded in the loam (Kurz *et al.*, 2004). At the Dairy the soils are moderately well to imperfectly drained with a loam to

sandy loam surface soil over a loam at 50 cm (Ryan, Noonan and Fanning, 1998). Information on the sites is given in Table 1 along with soil-P levels and management regimes. Soil samples were taken on a field-by-field basis to sample depths of 2, 5 and 10 cm, with approximately 30 to 40 soil cores bulked to provide a single composite sample for each depth in each field. The sampling procedure was repeated in triplicate at each site for each sample depth. The agronomic 10 cm sample was taken using the conventional bucket sampler. The shallow samples were taken using a soil corer, slicing cores to provide samples to 2 and 5 cm depths. All soil samples were dried over-night at 40 °C, ground and sieved through a 2 mm mesh and stored in boxes at room temperature prior to analysis.

The samples were analysed for Morgan's P, iron-oxide paper strip P, water-soluble P and calcium chloride P at Johnstown Castle laboratories. Agronomic P, as determined by Morgan's P, was measured on 6.5 ml of soil using a buffered (pH 4.8) acetate-acetic acid reagent in a 1:5 soil-to-solution ratio and extracting for 30 min (Peech and English, 1944; Byrne, 1979). The iron-oxide paper strip P test was used as a 'P sink' method for estimating labile inorganic P in soil according to

the method of Menon, Hammond and Sissingh (1988). One iron-oxide impregnated paper strip (10 cm × 2 cm) was added to a sample of soil (1 g) suspended in 40 ml of 0.01 M CaCl<sub>2</sub> solution and shaken for 16 h on an end-over-end shaker. The paper strips were then eluted with 0.1 M H<sub>2</sub>SO<sub>4</sub> for 1 h and the P concentration in the acidic solution determined colorimetrically. Water-soluble P was measured using the method of Van der Paauw (1971) using 1 g soil suspended in 40 ml of distilled water shaken on an end-over-end shaker for 1 h. Extracts of soil were filtered (Whatman no. 2 filter paper) before colorimetric analysis. Readily extractable P was determined using 0.01 M CaCl<sub>2</sub> in a 1:10 soil-to-solution ratio, extracted for 1 h (Houba, Novozamsky and Temminghoff, 1994) on an end-over-end shaker. Extracts of soil were filtered (Whatman no. 2 filter paper) before colorimetric analysis. Phosphorus concentrations in filtered extracts was determined using the modified ascorbic acid method for soils (John, 1970).

Mehlich-3 extraction was carried out at the Department of Plant and Soil Science at the University of Delaware using the modified Mehlich test (Mehlich, 1984) to extract P, Al and Fe at a 1:10 soil-to-solution ratio using Mehlich-3 reagent (0.2 M

**Table 1. Site details, fertiliser P and management system for the seven sites located at Johnstown Castle**

Site no.	Location	Area (ha)	Average slope	Morgan's P (mg/kg)	Fertiliser P applied (kg ha <sup>-1</sup> year <sup>-1</sup> )	Management system
1	Warren	1.54	3°	3.1 (2.8)	None	Grazed by beef cattle
2	Warren	1.09	4°	6.9 (6.0)	None	Cut for silage
3	Cowlands	0.46	3°	20.6 (17.6)	30	Grazed by beef cattle
4	Cowlands	0.50	3°	21.0 (18.1)	30	Grazed by beef cattle
5	Dairy	0.50	3°	4.1 (3.9)	17.5	Grazed by dairy cows
6	Dairy	0.50	3°	8.3 (7.8)	17.5	Grazed by dairy cows
7	Dairy	0.51	3°	6.8 (6.5)	17.5	Grazed by dairy cows

<sup>-1</sup> ( ) = mg/l.

$\text{CH}_3\text{COOH} + 0.25 \text{ M NH}_4\text{NO}_3 + 0.015 \text{ M NH}_4\text{F} + 0.13 \text{ M HNO}_3 + 0.001 \text{ M EDTA}$ ). A subsample of each triplicate soil sample was composited for each depth to represent soil at that depth and samples were shipped to the US for analysis. The P saturation in soils ( $P_{\text{sat}}$ ) was calculated using the molar ratio of Mehlich-3 P to Mehlich-3 Al expressed as a percentage. Organic matter (OM) in soil was measured by loss on ignition and the bulk density was estimated from OM by the method of Jeffrey (1970).

The effect of water-to-soil ratio on P desorption was investigated using the method of Yli-Halla *et al.* (1995). Weighed samples of soil were used to give dry-soil concentrations of 200, 20, 10, 6.25, 5 and 4 g/l in deionised water, representing water-to-soil ratios 5 to 250 l/kg. The soil extracts were filtered and P concentration in solution determined colorimetrically (John, 1970). The effect of sample pretreatment on water-soluble P was examined by carrying out extractions on field-moist and oven-dried (40 °C) samples. Soil sampled to 10 cm was collected from each site and the composite divided into two samples. Oven-dried samples were ground and sieved as previously described, whilst field-moist samples were hand sieved and percentage moisture determined. Both oven-dried and field-moist samples were weighed out to give equivalent dry soil concentrations and extracted at water-to-soil ratios in the range of 5 to 250 l/kg by the above method with water-to-soil ratios adjusted in moist samples to correspond to water-to-soil ratio for dry soil.

#### *Overland-flow analysis*

Overland-flow samples were collected from two of the seven sites used for soil sampling. These were a low P site (3.1 mg/kg; Site 1) and a high P site (20.6

mg/kg Site 3). These sites had been instrumented with discrete automatic samplers with integral data loggers (SIGMA 900). Overland flow was sampled from pipes before entering large tanks fitted with v-notch weirs and water level recorders. Both sites vary in size but have similar slope (Table 1) and were hydrologically isolated from surrounding sites by backfilling trenches, about 25 cm deep, with impermeable soil and building earth banks above to 20 cm, which were re-seeded with grass.

Overland-flow samples were collected from runoff events between December 2001 and March 2002. Samples were analysed for dissolved reactive P concentration after filtering through 0.45  $\mu\text{m}$  Sartorius filters using the ascorbic acid method (Murphy and Riley, 1962). Suspended solids concentration (SS) was measured on each runoff sample during the same period using standard methods (Anon, 1992).

In addition to collection of overland flow, soil samples were taken across sites 1 and 3 on 13 dates, from 4<sup>th</sup> December 2001 to 1<sup>st</sup> March 2002, to sample depths of 5 and 10 cm to correspond to overland flow events that occurred at these sites. Soil samples were oven-dried (40 °C) and sieved (2 mm mesh) prior to analysis of water-soluble P using water-to-soil ratios in the range of 5 to 250 l/kg as outlined above.

#### *Statistical methods*

All chemical analyses were carried out in duplicate and the average value was taken. Factorial analysis was carried out in Genstat and variance comparisons were made using Bartlett's test. Morgan's P results at Johnstown Castle laboratories are quoted in mg/kg with mg/l in parentheses since the test is carried out on a volume basis and is compared with methods

carried out on a weight basis. Water-soluble P is quoted in mg/kg to describe quantity of P desorbed to solution while the term extract-P concentration (using the same method) refers to the concentration of water-soluble P in the soil-water extract in mg/l.

## Results

### *Effect of sample depth and extracting solution*

Values of Morgan's P, CaCl<sub>2</sub>-P, FeO-P, WSP and Mehlich-3 P at each sample

depth are presented in Table 2. At similar depths, Mehlich-3 extracted the highest amount of P, followed by FeO-P, WSP, Morgan's P and CaCl<sub>2</sub>-P and the fertilizer application rate was reflected in soil-P level. Initially results from Morgan's P, CaCl<sub>2</sub>-P, FeO-P and WSP were analysed as a factorial by sample depth, across seven sites and three sample depths. Mehlich-3 was not included in this analysis because it represented a composite without replication. A non-significant interaction between sample depth and method was obtained indicating that each

**Table 2. Phosphorus concentration (mg/kg) in soil samples taken to 2, 5 and 10 cm from seven sites**

Site	Samples taken to a depth	Method <sup>1</sup> for P concentration (mg/kg)					
		Morgan's P	CaCl <sub>2</sub> -P	FeO-P	WSP	M3-P	P <sub>Sat</sub> (%) <sup>2</sup>
1	2 cm	6.9 (5.3) <sup>3</sup>	2.1	26.3	13.5	63.0	8.6
	5 cm	4.2 (3.5)	1.0	18.8	7.6	30.0	3.8
	10 cm	3.1 (2.8)	0.8	18.2	6.7	38.0	4.3
2	2 cm	8.0 (6.8)	1.4	43.8	15.7	67.0	9.8
	5 cm	8.7 (7.5)	1.2	37.1	15.0	82.0	9.5
	10 cm	6.9 (6.0)	0.9	37.8	11.7	75.0	8.7
3	2 cm	35.7 (26.7)	8.8	101.7	47.7	129.0	20.0
	5 cm	27.2 (21.6)	6.0	98.7	43.7	121.0	17.1
	10 cm	20.6 (17.6)	4.7	82.0	36.0	124.0	16.3
4	2 cm	29.4 (23.1)	10.0	100.4	53.2	157.0	20.8
	5 cm	24.5 (19.8)	5.9	78.3	38.8	140.0	18.5
	10 cm	21.0 (18.1)	5.1	76.6	36.6	135.0	17.4
5	2 cm	7.8 (7)	2.8	33.1	10.1	50.0	6.6
	5 cm	5.7 (5.0)	2.5	23.7	8.3	40.0	5.4
	10 cm	4.1 (3.9)	1.7	21.9	6.9	29.0	3.9
6	2 cm	11.5 (10.3)	3.0	47.5	16.6	95.0	10.5
	5 cm	9.7 (8.9)	2.5	44.0	14.5	84.0	9.6
	10 cm	8.3 (7.8)	2.3	41.1	15.9	87.0	9.4
7	2 cm	10.0 (8.8)	3.3	37.1	20.3	64.0	8.1
	5 cm	8.3 (7.4)	2.3	37.0	17.4	59.0	6.9
	10 cm	6.8 (6.5)	1.2	32.4	14.1	64.0	7.3

<sup>1</sup>Extraction method: Morgan's = Morgan's solution; CaCl<sub>2</sub>-P = diluted CaCl<sub>2</sub> solution; FeO-P = iron-oxide paper; WSP = water soluble; M3 = Mehlich-3 solution.

<sup>2</sup>P<sub>Sat</sub> = Molar ratio of Mehlich-3 P to Mehlich-3 Al, expressed as a percentage.

<sup>3</sup>Morgan's P as mg/l.

method responded the same with sample depth. The amount of extractable P differed greatly from method to method and the overall variability in the data was large (CV 21%). The large variability between methods masked any differences in extractable P between sample depth and the analysis yielded a non-significant effect of sample depth. Thus, the factorial analysis was then carried out across all sites, for each method separately, and a significant difference with sample depth was found for Morgan's P ( $P < 0.01$ ),  $\text{CaCl}_2$ -P ( $P < 0.01$ ), FeO-P ( $P < 0.01$ ) and WSP ( $P < 0.01$ ). In general soil-P level was highest at the surface to 2 cm, with highest values recorded at the high-P sites (3 and 4) receiving high fertilizer P application.

The variances at each sample depth were compared for each method. While there was no significant difference in the variances of individual soil test values between depths, the trend in variances

suggested that as sample depth increased the variance in the data decreased (Table 3). Extractable-P values at each sample depth were significantly correlated and the relationship between the standard agronomic sample depth to 10 cm and shallow sampling depths to 2 and 5 cm is presented for each of the relevant methods in Figure 1.

#### *Effect of water-to-soil ratio*

A factorial analysis of the data across the sites, for sample depth by water-to-soil ratio, showed a significant effect of both sample depth ( $P < 0.001$ ) and water-to-soil ratio ( $P < 0.001$ ) on WSP with no interaction between these factors. The effect of varying extract ratios on WSP, at each of the seven sites indicated that as water-to-soil ratios were widened, from 5 to 250 l/kg, the amount of extractable P (mg/kg) increased. These relationships are presented in Figure 2.

**Table 3. The effect of sampling depth on mean and variance of soil P level (mg/kg) using four extractants across all seven sites**

P test <sup>1</sup>	Sampling depth (cm)			s.e.d. (df=12)	Chi-square (df=2)
	2	5	10		
Morgan's P					
Mean	15.6	12.6	10.1	1.28	
Variance	138.8	85.8	56.6		1.32
$\text{CaCl}_2$ -P					
Mean	4.5	3.1	2.4	0.51	
Variance	11.8	4.3	3.2		3.29
FeO-P					
Mean	55.7	48.2	44.3	2.59	
Variance	1006.4	863.9	640.4		0.34
WSP					
Mean	25.3	20.7	18.3	1.67	
Variance	306.6	210.5	164.7		0.65

<sup>1</sup> Morgan's P = Morgan's P;  $\text{CaCl}_2$ -P = dilute  $\text{CaCl}_2$  extractable P; FeO-P = iron-oxide paper strip P; WSP = water soluble P.

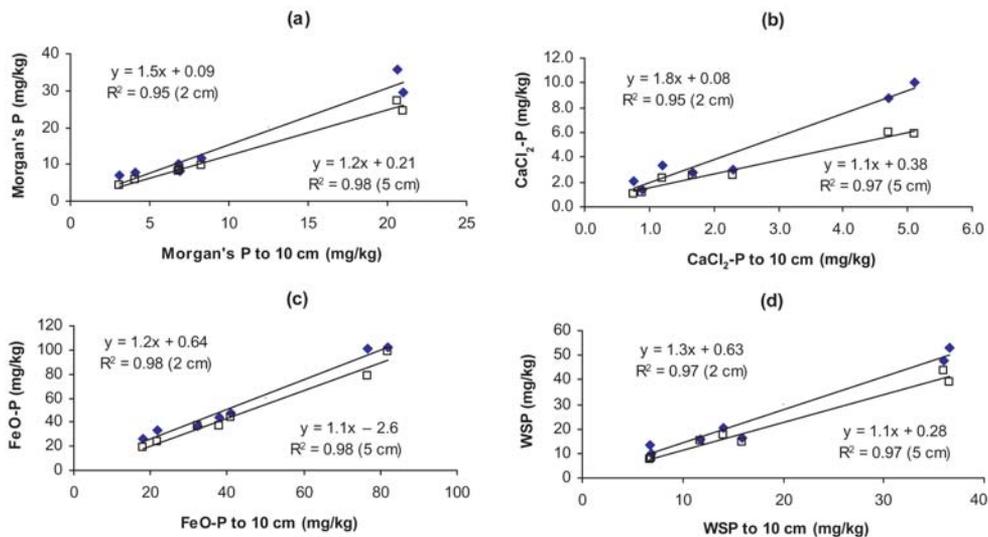


Figure 1: The relationship between soil-P concentration at the standard sample depth (10 cm) and those from samples taken to 2  $\blacklozenge$  and 5 cm  $\square$  at seven sites based on (a) Morgan's P test, (b) extraction using dilute  $CaCl_2$  solution ( $CaCl_2$ -P), (c) extraction using iron-oxide paper (FeO-P), (d) extraction using diluted water (WSP).

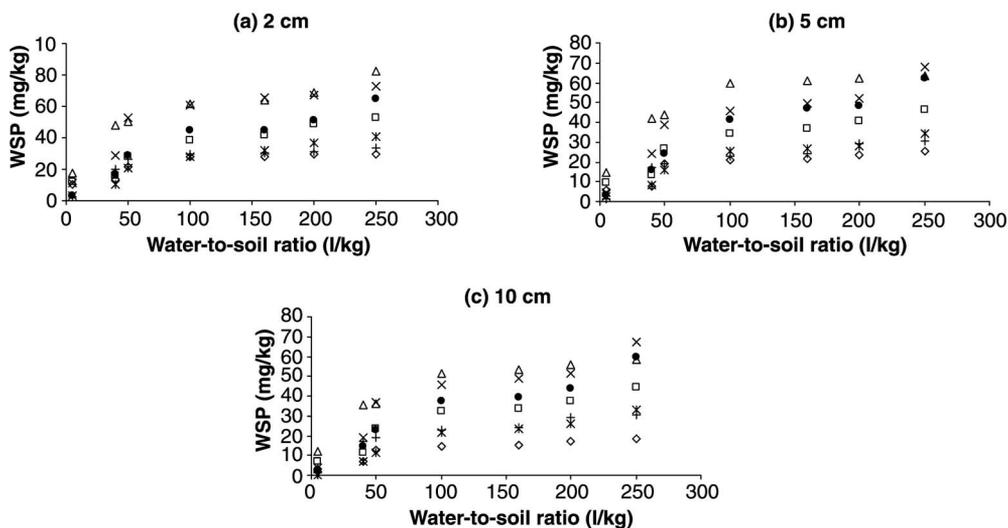


Figure 2: Water-soluble P (WSP) desorbed at water-to-soil ratios ranging from 5 to 250 l/kg at each site ( $\diamond$  Site 1,  $\square$  Site 2,  $\triangle$  Site 3,  $\times$  Site 4,  $\ast$  Site 5,  $\bullet$  Site 6,  $+$  Site 7) from samples taken to (a) 2 cm, (b) 5 cm and (c) 10 cm.

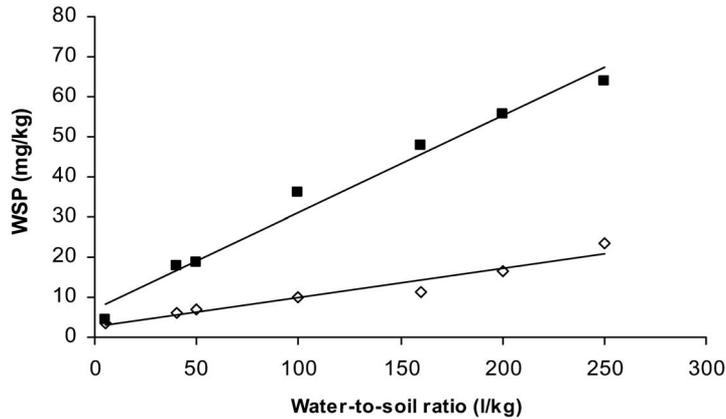


Figure 3: The relationship between water-soluble P (WSP) and water-to-soil ratio for field-moist ( $y = 0.07x + 2.5$ ;  $R^2 0.94$ ) ◇ and oven-dried ( $y = 0.24x + 7.1$ ;  $R^2 0.98$ ) ■ soil from samples taken to 10 cm at seven sites, averaged across the sites.

#### Effect of sample pretreatment

Values of WSP from oven-dry samples were significantly higher than corresponding values from field-moist samples ( $P < 0.001$ ); oven-drying significantly increased WSP by 27 to 175% over the range of water-to-soil ratios, compared to field-moist samples ( $P < 0.001$ ). There was an interaction ( $P < 0.001$ ) between extract ratio and sample pretreatment with the oven-dried samples showing a higher rate of increase in WSP than the field moist-samples as the extract ratio widened. The data were averaged over the seven sites and WSP from oven-dry soil at the 5 and 250 l/kg ratios were 4.2 and 64 mg/kg, respectively, while values measured from field-moist soil at corresponding extract ratios were 3.3 and 23.3 mg/kg, respectively. Taking average values across the seven sites, the relationships between WSP and water-to-soil ratio for field-moist and oven-dry samples are plotted in Figure 3.

#### Dissolved reactive P in overland flow and soil P data

Overland-flow data were classified into discrete events and summary statistics on

flow volume, DRP, P loss and SS are presented in Table 4 for Sites 1 and 3. The average DRP concentration ranged from 0.032 to 0.067 mg/l at Site 1 compared with 0.261 to 0.620 mg/l at Site 3. The total volume of overland flow collected from the sites was calculated for each event and used to calculate P losses based on DRP. The P loss ranged from <0.005 to 0.05 kg/ha from the low P site, and from 0.002 to 0.37 kg/ha for the high P site. The total P loss from the sites during the sampling period (December 2001 to March 2002) were calculated as 0.065 and 0.457 kg/ha from Sites 1 and 3, respectively; a seven-fold difference between the sites. Overland flow samples from event 3 at both sites represented 66 and 67% of samples collected from Site 1 and 3, respectively, between December 2001 and March 2002, and the DRP and flow values recorded during this event are plotted in the event profiles in Figure 4. At Site 3 (Figure 4a) DRP values fell initially from an average of 0.558 mg/l to 0.208 mg/l before increasing again to a 'plateau' (average concentration of 0.427 mg/l) for

Table 4. Event date and summary statistics for dissolved reactive P and suspended solids recorded at Sites 1 and 3

Event dates	Event no.	No. of samples	Total flow volume (l)	Dissolved reactive P (mg/l)		P loss (kg/ha)	Suspended solids (mg/l)	
				Range	Mean		Range	Mean
<i>Site 1</i>								
3 Dec-5 Dec 2001	1	16	180,394	0.025-0.085	0.059	0.008	2-85	20
6 Dec-13 Dec 2001	2	3	42,949	0.027-0.069	0.046	0.001	4-14	9
13 Jan-13 Feb 2002	3	77	1179,019	<0.005-0.314	0.067	0.050	1-125	28
24 Feb-2 Mar 2002	4	20	281,189	<0.005-0.084	0.032	0.006	2-73	17
<i>Site 3</i>								
3 Dec-6 Dec 2001	1	47	26,259	0.356-0.730	0.620	0.035	1-393	33
13 Jan-12 Feb 2002	3	151	413,376	0.037-0.695	0.411	0.37	0-184	29
25 Feb-25 Feb 2002	4	6	3,832	0.207-0.312	0.261	0.002	32-50	43
28 Feb-2 Mar 2002	5	23	56,064	0.334-0.506	0.417	0.05	10-107	39

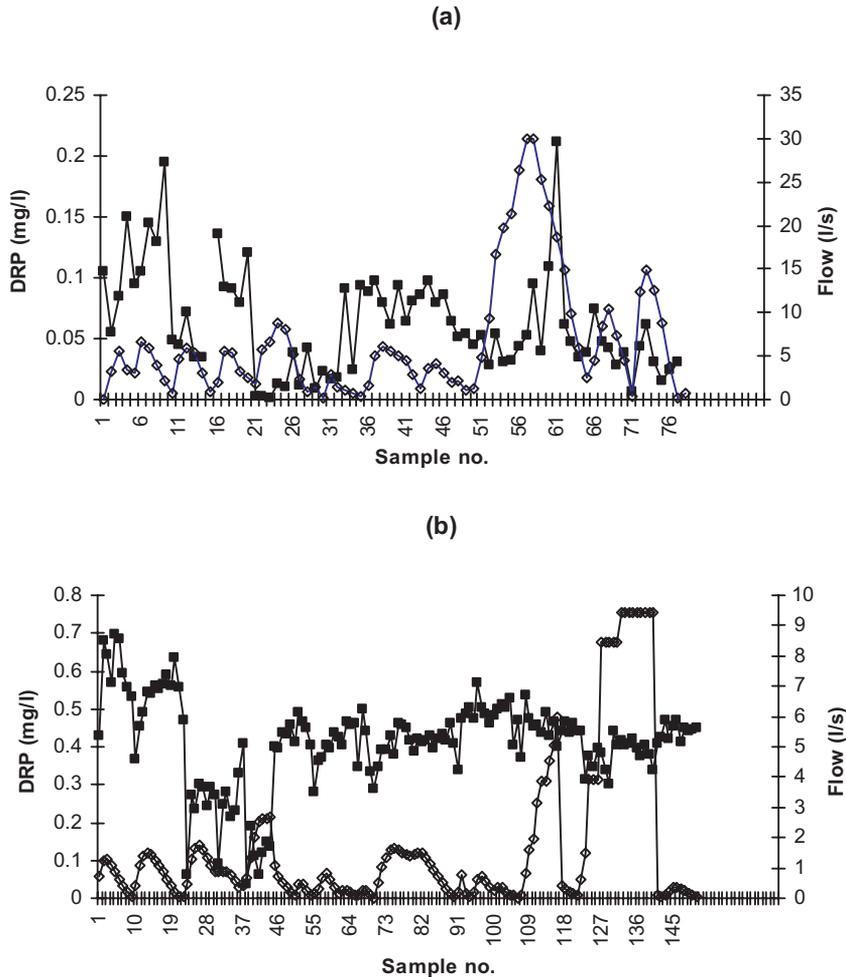


Figure 4: Event profiles for concentration of dissolved reactive P (DRP —■—) and flow rate (—◇—) recorded during event 3 at (a) Site 1 and (b) Site 3.

the remainder of the event. However, no such plateau concentrations were observed in the event profile plotted at Site 1 for the corresponding event (Figure 4a). The SS values measured in overland flow during event 3 at the two sites were similar with values ranging from 1 to 125 mg/l and 0 to 184 mg/l for Sites 1 and 3, respectively. The relationship between DRP and SS is presented in Figure 5 for each site. Although less than 20% of the

variation in DRP can be explained by SS, the correlation between these two variables was significant at each site, but was positive at Site 3 and negative at Site 1.

Soil samples taken to 5 and 10 cm during overland flow events were extracted with distilled water at a range of water-to-soil ratios. In this instance, P concentration in the water extracts (mg/l) at each water-to-soil ratio is presented (referred to as extract-P concentration), where previously

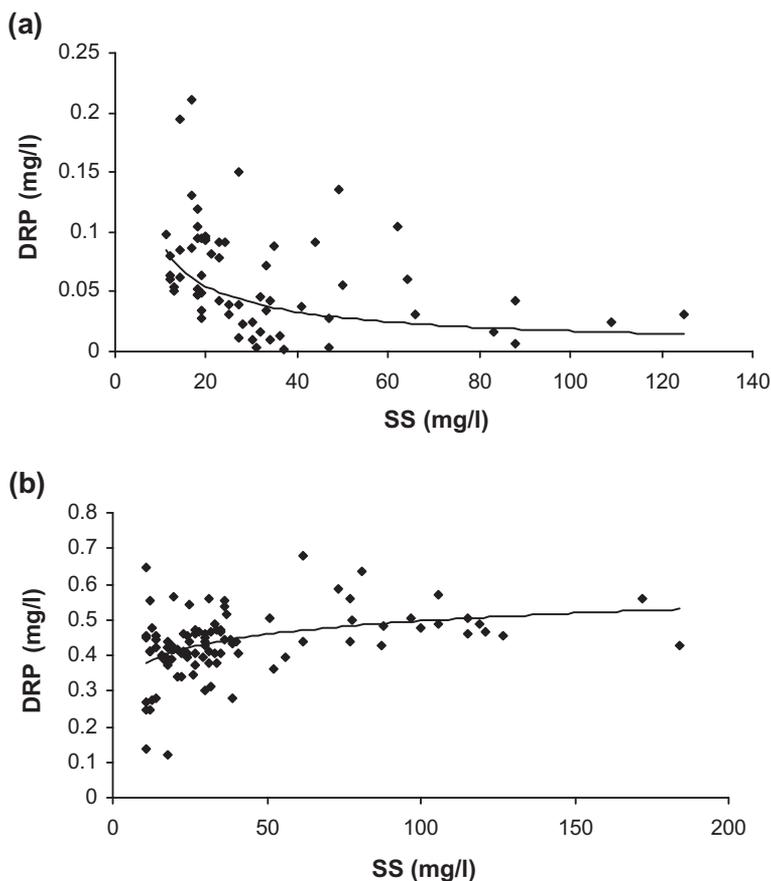


Figure 5: The relationship between dissolved reactive P (DRP) and suspended solids (SS) in overland flow during event 3 at (a) Site 1 ( $y = 0.48x^{-0.72}$ ;  $R^2 0.17$ ,  $P < 0.001$ ) and (b) Site 3 ( $y = 0.26x^{0.14}$ ;  $R^2 0.16$ ,  $P < 0.001$ ).

quantities of P desorbed (mg/kg) were used. No temporal trend was apparent in extract-P concentration at any extract ratio, and values measured over the sampling period were averaged for each water-to-soil ratio. Average values of extract P ranged from 0.13 to 0.63 mg/l and 0.27 to 1.01 mg/l for soil sampled to 10 cm and from 0.17 to 0.64 mg/l and 0.27 to 1.66 mg/l for soil sampled to 5 cm, at Sites 1 and 3, respectively, reflecting the difference in soil-test P between the sites. These average values are plotted in Figure 6 and

show the decrease in extract-P concentration as water-to-soil ratio widens. The effect of sample depth (5 cm > 10 cm) was evident at all water-to-soil ratios at Site 1. At Site 3, however, the difference between sample depths was more evident at the lowest extract ratio (5 l/kg) while at other extract ratios the values measured to 5 cm sample depth were either greater than or equal to values measured to 10 cm.

Dissolved reactive P concentrations in overland flow from Sites 1 and 3 were compared with extract-P concentrations

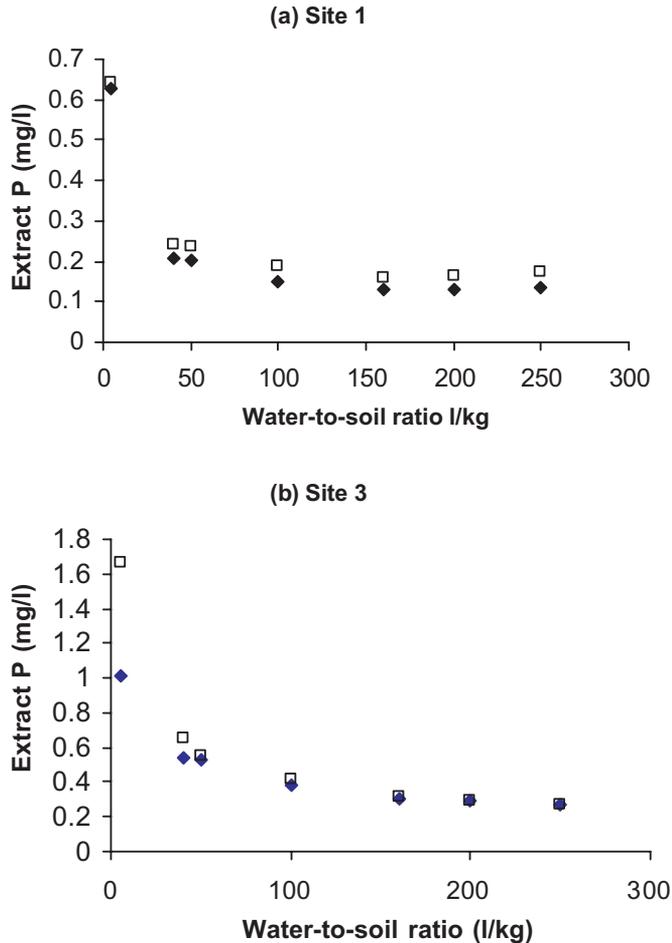


Figure 6: Soil P measured from soil extracts at varying water-to-soil ratios from samples taken to 5(□) and 10(◆) cm during overland flow events at (a) Site 1 and (b) Site 3.

from soil sampled to 10 and 5 cm, from the same sites, extracted using water-to-soil ratios ranging from 5 to 250 l/kg. At Site 1, extract-P concentrations ranged from 0.13 to 0.63 mg/l for samples taken to 10 cm and from 0.17 to 0.64 mg/l for samples taken to 5 cm, which exceeded the average DRP values (0.032 to 0.067 mg/l) measured in overland flow. Only maximum values of DRP were comparable to extract-P concentrations at this site. At Site 3, extract P concentrations from sam-

ples taken to 10 (0.27 to 1.01 mg/l) and 5 cm (0.27 to 1.66 mg/l) at water-to-soil ratios of 250 to 100 l/kg captured the average DRP values (0.261 to 0.411 mg/l) recorded during the events at this site. Maximum values of DRP were reflected in extract-P concentrations using water-to-soil ratios from 5 to 250 l/kg. Soil-test P data were compared with P loss in overland flow from the two sites monitored in this study (Table 5). The relative rates of P loss from Sites 1 and 3 were compared

**Table 5. Phosphorus loss (kg/ha) from site 1 and 3 and soil-test P results (mg/kg), and loss ratios based on actual P loss and soil-test P values**

	Method for P concentration						
	Estimated P loss kg P/ha	Morgan's P	CaCl <sub>2</sub> -P	FeO-P	WSP	M3-P	P <sub>Sat</sub> (%)
Site 1	0.065	3.1 (2.8) <sup>1</sup>	0.8 (0.8)	18.2 (16.5)	6.1 (5.6)	38.2 (35)	4.3
Site 3	0.457	20.6 (17.6)	4.7 (4)	82 (70.1)	29.5 (25.1)	124 (106)	16.3
Ratios <sup>2</sup>	7.0	6.6 (6.3)	5.9 (5)	4.5 (4.2)	4.8 (4.5)	3.2 (3)	3.8

<sup>1</sup> ( ) Values on volume bases (mg/l).

<sup>2</sup> Value for Site 3 relates and corresponding value for Site 1.

with the relative difference in soil-test P using Morgan's P, CaCl<sub>2</sub>-P, FeO-P, WSP, M3-P and P<sub>Sat</sub> at both sites. Since Morgan's P is carried out on a routine basis using a volume of soil while the other tests were done on a weight basis, test results are given for both bases using bulk density to convert weight basis to volume basis. There was a seven-fold difference in P loss between the two sites; the difference in Morgan's P value (6.6) was closest to the difference in P loss.

## Discussion

### *Effect of sample depth and extracting solution*

Soil-test P values decreased as sampling depth increased and while the test method had a large effect, the absence of an interaction between test method and sample depth in the factorial analysis indicated that all of the methods used responded similarly to changes in sample depth. These findings concur with Torbert *et al.* (2002) in that high P values were associated with shallow sampling depths when depths to 2.5, 5 and 15 cm were compared using Mehlich-3 extractable P. However, Schroeder *et al.* (2004) compared exactly

the same range of sample depths as in the present studies (2, 5 and 10 cm) and extracted soil using Mehlich-3, WSP, FeO-P and degree of P saturation (DPS) across six fields with varying soil-test P levels. The authors reported no difference in P levels among sample depths with Mehlich-3 and DPS but statistically significant differences with sample depth for WSP and FeO-P. In that study, significant differences were observed with sample depth using FeO-P but not using WSP (2 cm *v.* 5 cm, and, 5 cm *v.* 10 cm were not significantly different, but 2 *v.* 10 cm was significantly different). In the present study, no overall significance in P tests was detected with depth, similar to Schroeder *et al.* (2004); but, when a factorial analysis was performed using data from all sites testing for interactions, significant differences with depth were found when each P test was evaluated.

The trend of decreasing soil P with increasing sample depth from 2 to 5 to 10 cm in each of the four P tests used here was also demonstrated in the correlations between sample depth for each test, indicating that the standard agronomic sample depth to 10 cm was correlated with results from shallow sample depths (2 and

5 cm) using Morgan's P,  $\text{CaCl}_2\text{-P}$ , WSP and FeO-P. Similar relationships between the standard agronomic sample depth and samples taken to 2 cm and 7.5 cm have been demonstrated in a range of Irish soil types (Humphreys *et al.*, 1998; Daly, unpublished) across a similar set of P tests. Recommendations to adopt shallow sample depths (to 2 cm) have been made (Mulqueen *et al.*, 2004) for situations in Ireland where soil sampling is used for environmental purposes. However, in the present study, data from samples taken to 2 cm had higher variance than corresponding values from samples taken to 5 and 10 cm. This finding concurs with Torbert *et al.* (2002) who surmised that the greater variability in 2.5 cm samples was due to dung patches and problems in obtaining a consistent sample at these depths. Furthermore, the authors opined that whilst 5 cm samples gave the 'best fit' to DRP in rainfall simulated runoff, shallow sampling depths would be easier to falsify. This may be an important consideration if shallow sample depths are used for setting limits or environmental threshold values of soil P for agriculture. Therefore, it would be difficult to justify a change in sample depth when the standard agronomic depth has been shown to be well correlated with shallow depths in this and other studies. In addition, the higher variances for the 2 and 5 cm samples compared with the standard sample depth, would indicate that adopting a 2 cm sample may lead to more variability in soil P data, and perhaps a less reliable or reproducible indicator of environmental loss.

*Effect of water-to-soil ratio and sample pre-treatment on water soluble P*

At each of the sites WSP in soil increased as water-to-soil ratio was increased from 5 to 250 l/kg, which agrees with other findings that extract ratios have an important

effect on P desorbed from soil to solution (Sharpley, 1983; Barrow, 1979, 1983). When the effect of sample pre-treatment was introduced, field-moist samples desorbed significantly less P to solution than oven-dried samples. This difference was evident at each site and could be attributed to effects of drying and rewetting of soils that have been cited in the literature, namely, release of P from soil microbial biomass following cell rupture (Turner and Haygarth, 2001), increased solubility of organic matter and soil P (Olsen and Court, 1982) and changes in soil structure and reactive cation solubility (Bartlett and James, 1980). The large difference between oven-dry and field-moist WSP values suggests that some physico-chemical effect occurred in addition to the contribution of extractable P from microbial cell rupture. Drying soil has been reported to affect concentrations of extractable Al and Fe (Bartlett and James, 1980), which may account for increased P solubility in dried soils. Physical changes that have been reported include shrinking and swelling during the drying and rewetting process which exposes some previously unexposed soil organic matter and P sorption surfaces, resulting in an increase in organic matter solubility and P desorption (Chepkwony, Haynes and Harrison, 2001). In the present study, the interaction between sample pre-treatment and water-to-soil ratio may have been due to increased extractable P in dried soils across a range of increasing water-to-soil ratios.

Styles, Donohue and Coxon (2004) observed significant differences in desorption between air-dried and field-moist samples and suggested that the standard practice of simply air-drying soil prior to desorption analysis may obscure important effects such as differences in soil type. Whilst oven-drying soil samples prior to

analysis affects P desorption, the standard agronomic soil P test in Ireland uses oven-dried (40 °C) soil prior to extracting with Morgan's reagent. However, Olsen and Court (1981) reported that since acid and alkaline reagents alter the components and structure of soil, the effects of drying are less noticeable. Furthermore, several northeastern states in the US use Morgan's reagent, or a modified Morgan's reagent, for fertilizer recommendations and P runoff risk indices from soil samples that have been oven-dried to 55 °C prior to analysis (Ketterings *et al.*, 2002). Conversion equations have been derived to convert Morgan's P to Mehlich-3 extractable P and Olsen P (Ketterings *et al.*, 2002; Foy *et al.*, 1997) and correlations have been established between Morgan's P and P desorption tests such as WSP and CaCl<sub>2</sub>-P in Ireland (Daly *et al.*, 2001) and the US (Kleinman *et al.*, 2000; Jokela, Magdoff and Durieux, 1998) using air-dried and oven-dried samples. Whilst oven-drying soils affects desorption values, the literature cited here on Morgan's P, would suggest that it does not seem to affect the performance of the test as an agronomic or environmental indicator.

#### *Relating soil P to DRP concentration in overland flow*

The difference in P levels between the sites was reflected in the range of DRP concentrations in overland flow. Kurz *et al.* (2004) recorded winter DRP values from the same sites over a 493-day period that were comparable to values measured in the present study. For example, the range of maximum values from Sites 1 and 3 recorded by Kurz *et al.* (2004) were 0.011 to 0.511 and 0.173 to 0.889 mg/l, respectively, whilst maximum values measured in the present study ranged from 0.069 to 0.314 and 0.312 to 0.730 mg/l, from Sites 1 and 3, respectively. The range of minimum

values recorded by Kurz *et al.* (2004) were also similar to the range of minimum values measured in the present study. Given that both studies on the same sites were carried out independently the similarity in the range of values measured indicates that DRP concentration in overland flow can be relatively stable for sites under consistent P management regimes. Suspended solids accounted for less than 20% of the variation in DRP from each site suggesting that the major source of DRP is the soil remaining in the field. Yhi-Halla *et al.* (1995) calculated that SS contributed no more than 16 to 38% of DRP in surface runoff from edge-of-field measurements and concluded that soil-P status was more significant than eroded soil in determining DRP concentration in runoff.

A direct comparison of DRP in overland flow and extract-P concentration was made to see if DRP values could be simulated by water-extractable P, using soil samples taken to 5 and 10 cm from the sites at the time of each runoff event. At Site 1, extract-P concentrations over the range of extract ratios exceeded the average DRP values measured during events. Only maximum DRP values from event 3 (0.314 mg/l) were compatible with extract P concentrations measured at extract ratios of 50 to 5 l/kg from 10 cm (0.20 to 0.63 mg/l) and 5 cm samples (0.24 to 0.64 mg/l). Since soil samples were oven-dried according to the standard procedure prior to analysis, the increased available P due to drying may be more noticeable at the low P site and caused the discrepancy between DRP and extract-P concentration. At Site 3, the extract-P concentrations were more compatible with the DRP concentrations in overland flow and the average DRP from events (0.261 to 0.411 mg/l) corresponded with extract-P concentrations at extract ratios of 250 to 50 l/kg

(0.27 to 0.53 mg/l) for samples taken to 10 cm, and at extract ratios of 250 to 100 l/kg (0.27 to 0.41 mg/l) samples taken to 5 cm. Yli-Halla *et al.* (1995) measured water-extractable P from samples taken to 10 cm, extracted with ratios in the range 250 to 100 l/kg that gave a rough estimate for average concentrations of DRP in field runoff. Although, in the present study, soil samples from Site 3 were also oven-dried prior to analysis the results were more comparable with DRP concentrations, than those measured at Site 1. Perhaps the effects of drying soil, discussed previously, had a greater impact on water-extractable P values at the low soil-P site.

With only two field sites monitored in this study a statistical relationship between DRP loss and soil P data was not possible, and the relative difference in P loss and extractable soil P between the sites was considered in order to compare P loss in overland flow with soil test P data. Losses of P from Sites 1 and 3 were 0.065 and 0.457 kg/ha, a seven-fold difference between the sites for the monitoring period. Kurz *et al.* (2004) monitored DRP concentrations from the same sites and computed annual P losses of 0.698 and 4.764 kg/ha from Sites 1 and 3, respectively, showing a similar seven-fold difference between the sites. The total monitoring period reported by Kurz *et al.* (2004) was 493 days and total P loss measured over that time was 0.778 and 5.299 kg/ha from Sites 1 and 3, respectively, with significant losses in overland flow measured in summer months from May to September. Whilst we can only discuss the relative difference in DRP loss and soil P levels between these sites it is important to note that the relative difference in P loss is reflected in the relative difference in soil-test P levels using the standard agronomic test (Morgan's P) to 10 cm. This

observation concurs with Kurz *et al.* (2004) who monitored DRP losses from a total of three field sites at low (Site 1 in this study), medium and high (Site 3 in this study) soil-test P levels, and found a pattern of increasing minimum, maximum and flow-weighted average DRP concentrations with a clear increase in P loss from sites at low to medium to high soil P levels, as measured by Morgan's P to 10 cm. Whilst absolute concentrations of DRP in overland flow were not easily simulated by laboratory extraction in this work, the standard agronomic soil P to 10 cm gave a good indication of the relative losses of P at edge-of-field scale. This concurs with other studies that have used Morgan's P as an indicator of P loss from soil to water.

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