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AND BICARBONATE EXTRACTABLE
PHOSPHORUS AND ITS RELATIONSHIP
TO SAMPLE SIZE IN TWO SOILS OF
THE SWAN COASTAL PLAIN,
WESTERN AUSTRALIA**



Department of Conservation and Environment
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SPATIAL VARIABILITY OF TOTAL AND BICARBONATE EXTRACTABLE
PHOSPHORUS AND ITS RELATIONSHIP TO SAMPLE SIZE IN TWO SOILS OF
THE SWAN COASTAL PLAIN, WESTERN AUSTRALIA.

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Bulletin 201 July 1985

ISBN 0 7309 0434 2

ABSTRACT

The spatial variability of bicarbonate extractable phosphorus and total phosphorus in surface samples of two Swan Coastal Plain soils was investigated. In both soils the majority of soil phosphorus variability occurred over small distances of 0.3 m or less, with bicarbonate extractable phosphorus showing greater variability than total phosphorus concentration. The sample number required to measure soil phosphorus within a given confidence interval and over an area of approximately 0.5 ha, was estimated for both soils. Generally it was found that to increase the accuracy of measurement to within $\pm 5\%$ required a greater effort of sample collection than was justified for a practical field programme.

INTRODUCTION

Eutrophication of the Peel Harvey Estuary is principally caused by leaching and runoff of phosphorus from well-drained sandy soils of the coastal plain catchments that surround the estuary (Fig 1). This phosphorus originates from superphosphate which has been applied at rates of approximately 16-20 kg P/ha annually for up to 40 years (Black and Hodgkin, 1984).

In 1982 a research programme was commenced to investigate methods of reducing the transport of phosphorus from farmland into drainage water, and hence alleviate algal growth problems in the estuary. Part of the programme has been to examine the chemistry of soil phosphorus in relation to plant nutrition, leaching and surface runoff. This work involved experimental plot design and extensive soil sampling to determine concentration of soil phosphorus fractions and their temporal-spatial variability. Therefore, it was necessary to know the extent of the intrinsic spatial variability for soil phosphorus to ensure that it is accounted for in any soil sampling programme, which is the aim of this investigation. Specifically we estimated the spatial scale of this intrinsic soil phosphorus variability in the field and determined the sample size required to ascertain the average soil phosphorus concentration within a specified level of confidence for both bicarbonate extractable phosphorus (BIC-P) and total phosphorus (TP).

MATERIALS AND METHODS

Soils

The work was carried out on surface samples (0-10 cm) of two soils (Joel loamy sand, Jls, and Coolup sandy loam, Csl,) which were identified as important sources of leached phosphorus (P) in the coastal plain catchment of the estuary (Fig 1).

The Joel Loamy Sand Site

Jls is one of the soil types found in the interdunal swales of the Bassendean association (McArthur et al., 1959). It is aeolian in origin and generally has an organic hard pan at 0.5-4.5 m below the surface. The site was situated about 40 m from a 0.2 m deep drain and on a slope of approximately 1:40.

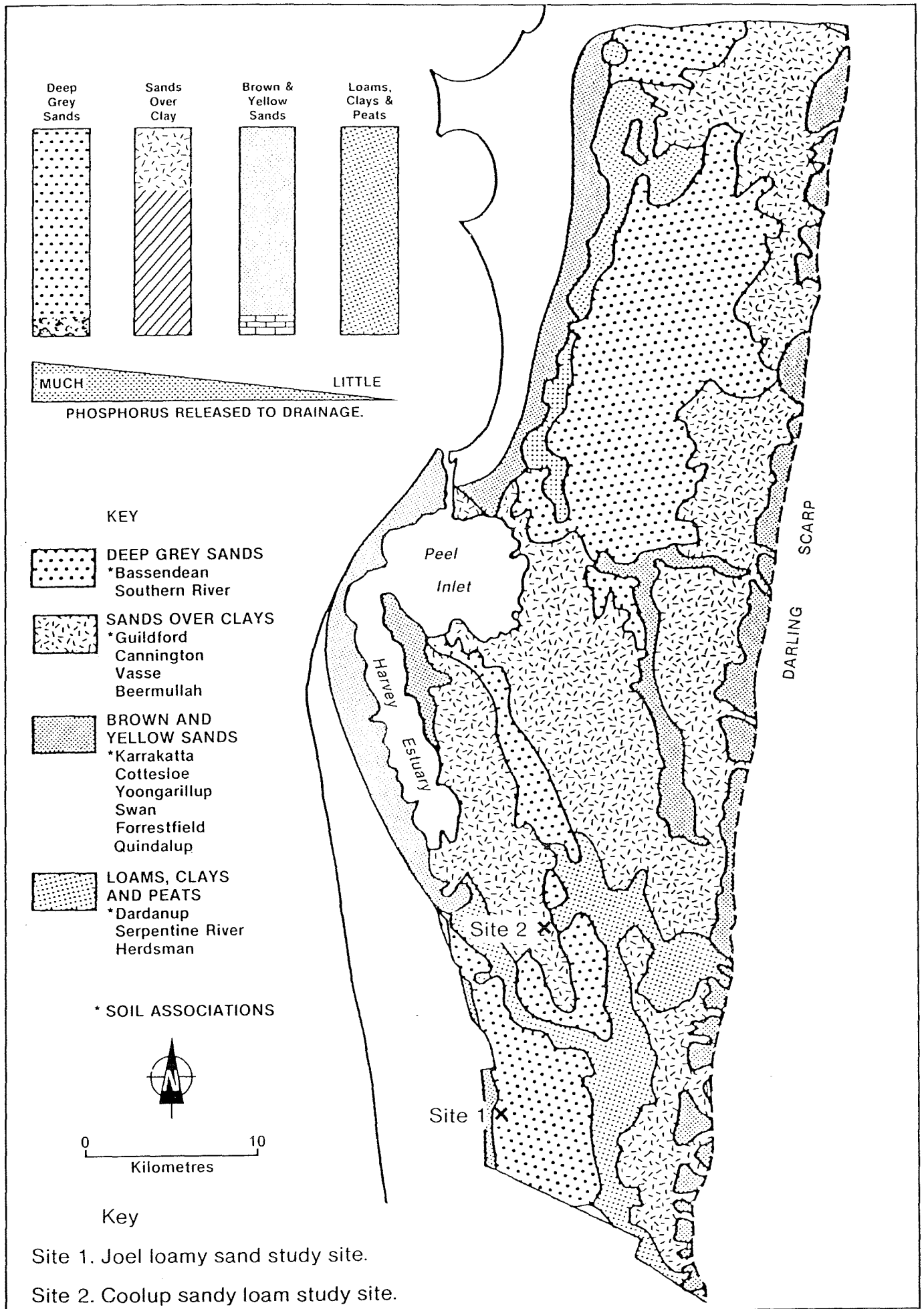


Figure 1. Soil categories on the coastal plain catchment (after Bettenay et al., 1960 and E. Bettenay pers. comm.)

During the hot dry summers experienced in the region, ground water levels drop to about 1 m below the surface. Because of a relatively low elevation, water table levels in the wet winters fluctuate near ground surface, despite influence from the nearby drain.

Sixteen years previously the soil was cleared of virgin bush and seeded with shallow-rooted annual pasture species which die off in early summer because of the lowered water table. Since establishment this clover-based pasture has been grazed by sheep and cattle. Superphosphate fertilizer has also been applied since clearing, at an average rate of 20 kg/ha/yr of P, except the last year when none was applied.

The Coolup Sandy Loam Site

This soil unit is a sandy loam meadow podzol of the Guildford association (Mc Arthur et al, 1959). It is an alluvial soil with a distinct clay B horizon at 0.2 - 0.6 m depth.

The site was situated on flat terrain within approximately 40 m of a 1 m deep drain. The level topography and shallow clay horizon have resulted in poor drainage; consequently surface waterlogging occurs over winter. In summer the water table drops to 1.5 - 2 m depth resulting in pasture death.

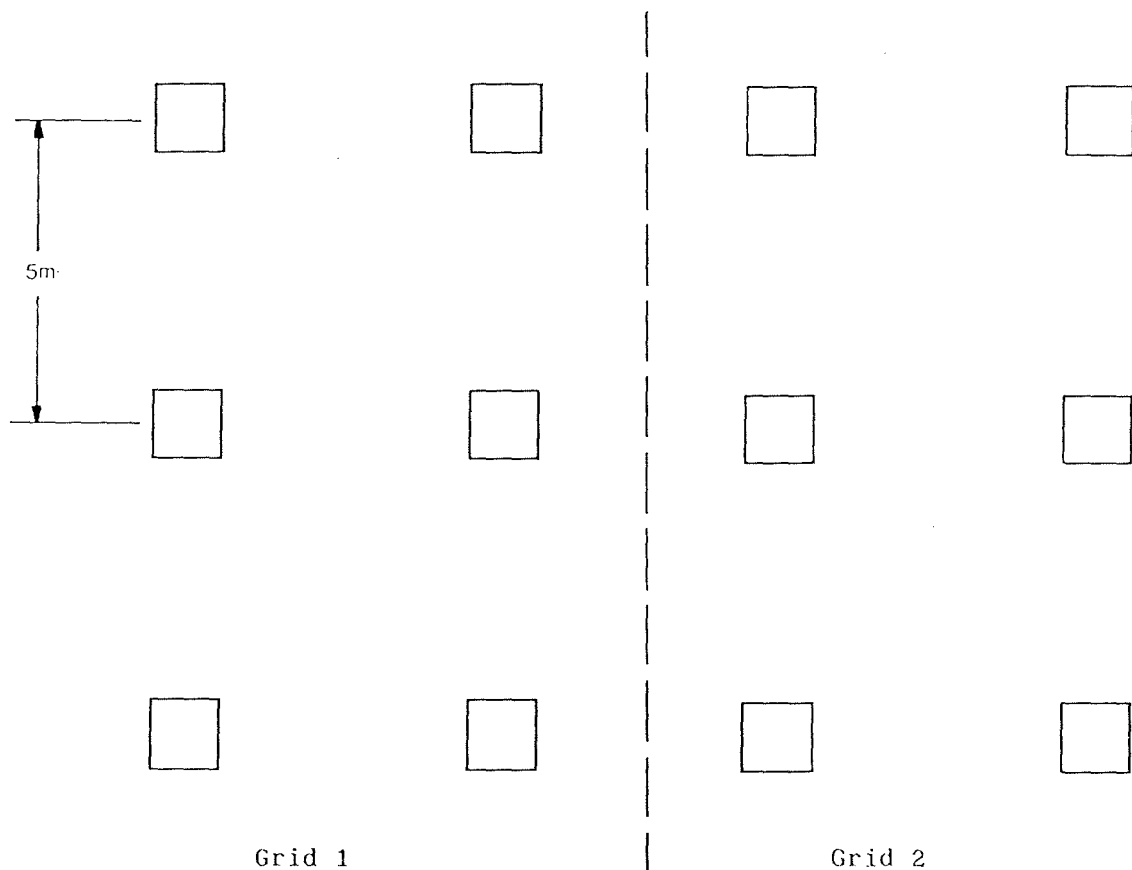
The soil has been covered by shallow-rooted annual pasture species since the 1930's and grazed by stock. Superphosphate fertilizer has been applied most years since clearing at an average rate of 18 kg/ha/yr of P. Clearing was not total and some Marri (Eucalyptus calophylla) trees are present in the vicinity of the site.

Soil Sampling

A sampling system was designed that would test for soil P variability within an area approximately the size of proposed experimental sites to be used in the research programme (approx. 0.1 ha). The sampling system also included enough samples to enable an estimation of the sample number required to reduce the observed variability to within acceptable limits (eg $P(x = \mu \pm 5\%) = 0.95$).

The design at both sites consisted of two 6 m x 11 m grids spaced approximately 40 m apart. Within each grid were marked six, 1 m² quadrates at 5 m intervals to test for within plot variability (Fig 2). Nine soil cores spaced 30 cm apart were taken from each quadrate, ie 54 samples per grid and 108 samples total. Sample cores were taken using a standard Western Australian Department of Agriculture foot soil sampler (pogo), which samples a 19 mm diameter core to a 10 cm depth.

Figure 2 Schematic Diagram of Sampling Design



Sampling was carried out in April before the heavy winter rains; April 1982 for T P analysis on the Csl, April 1984 for both analyses on the Jls and BIC P analysis on the Csl. This ensured that samples were taken before substantial leaching had occurred from soluble P fractions which accumulate in the soil over summer. If leaching had occurred then BIC-P values for the Jls would have been at the limits of analytical detection and too low for variability analysis.

Soil Analyses

All samples were analysed by the West Australian Government Chemical Laboratories using their standard procedures outlined below.

BIC-P Analysis

The samples were dried in an oven at 35°C and put through a 2 mm sieve. Sub-samples of 1 g for BIC-P analysis were then taken and mixed to 100 ml with 0.5 M NaHCO₃, giving a pH of 8.5. The resulting solution was shaken for 16 hours at 23±1°C then centrifuged and neutralized. Dissolved-P was then measured according to Murphy and Riley (1962). The associated analytical error for each analysis was ±1 ppm for samples of approximately 10 ppm or less and 10% for larger concentrations.

T P Analysis

This procedure is a modification of the Kjeldahl method for determination of nitrogen concentration. The sieved soil was finely ground in a TEMA mill and 0.5 g sub-samples were mixed with 3 ml of conc. H₂SO₄. Using CuSO₄ and K₂SO₄ as catalysts the solution was digested for 30 min at 250°C, 30 min at 320°C and finally 100 min at 390°C, ensuring all organic material had been dissolved. The solution was then made up to 50 mL with deionised water and dissolved P measured using the method of Murphy and Riley (1962). The associated analytical error was estimated to be ±10% for analyses greater than 100 ppm and ±10 ppm for smaller measured concentrations.

Replicate analyses were done on each sample for both extractions, thereby reducing the probable analytical error.

Statistical Analysis

Spatial variation was tested by using a one-way analysis of variance (ANOVA) between grids and between quadrates within the grids. If the null hypothesis was rejected at the 0.05 level of significance then significant variation was said to be occurring.

The number of soil samples taken from each site (108) was large relative to the area, thus it was assumed that the sample mean approximated the real mean of the sampled area. This enabled calculation of the sample number required to provide estimates, with varying confidence intervals, of soil P concentration in an experimental plot for both BIC-P and TP.

RESULTS

Joel Loamy Sand (Jls)

Large Scale Variation

The possibility of variation over a 40 m radius was investigated by ANOVA between the two sets of grid data (Table 1). Both BIC-P and TP were found to be significantly different, $P < 0.01$ (Table 2).

Table 1 Grid mean (\bar{x}), standard deviation (s) and coefficient of variation (CV) of BIC-P and TP analyses for Joel loamy sand

	Grid 1		Grid 2	
	BIC-P	TP	BIC-P	TP
\bar{x}	9.7	48.3	7.9	41.0
s	2.9	9.1	2.3	12.8
CV	0.30	0.19	0.29	0.31

Table 2 ANOVA F values for large scale variation (40 m) on Joel loamy sand

Comparison	BIC-P	TP	significance
Between grids	11.5	11.9	$P(F > 3.94) = 0.05$ $P(F > 6.90) = 0.01$

Small Scale Variation

Since both P extractions showed significant difference between grids, ANOVA's between quadrates were done separately for each grid.

Although only 4 m separated the quadrates, observed differences in means and standard deviations (Tables 3,4) were found to be highly significant ($P < 0.01$), (Table 5). Variation appears to be high over these small scale distances.

Table 3 Quadrate means, standard deviations and coefficients of variation for BIC-P analyses on Joel loamy sand

		quad 1	quad 2	quad 3	quad 4	quad 5	quad 6
Grid 1	\bar{x}	11.1	9.9	8.9	9.9	10.3	8.6
	s	3.8	2.8	2.3	3.1	3.4	1.3
	CV	0.34	0.28	0.26	0.31	0.33	0.15
Grid 2	\bar{x}	10.4	8.0	8.1	7.6	6.7	7.7
	s	2.8	1.4	2.5	1.5	1.4	2.3
	CV	0.27	0.18	0.31	0.20	0.21	0.30

Table 4 Quadrate means, standard deviations and coefficients of variation for TP analyses on Joel loamy sand

		quad 1	quad 2	quad 3	quad 4	quad 5	quad 6
Grid 1	\bar{x}	50.6	43.4	38.1	57.1	52.7	47.9
	s	5.7	7.6	3.9	10.2	8.4	4.1
	CV	0.11	0.18	0.10	0.18	0.16	0.09
Grid 2	\bar{x}	42.4	41.7	37.4	42.9	38.7	42.9
	s	7.9	7.2	11.4	13.8	19.1	16.5
	CV	0.19	0.17	0.30	0.32	0.49	0.38

Table 5 ANOVA F values for small scale variation (5 m)

Comparison	BIC-P	TP	significance
Between quadrates within grid 1	57.2	264.6	P(F>2.41)=0.05 P(F>3.42)=0.01
Between quadrates within grid 2	158.6	96.7	P(F>2.41)=0.05 P(F>3.12)=0.01

Microscale Variation

Microscale variation is defined here as changes in soil P concentration that occur within a 0.3 m radius (as sampled) or less.

The variances of the 9 samples taken within each quadrate were compared using coefficients of variation (CV, where $CV = s/\bar{x}$), (Tables 3,4). Although the coefficients do vary they were generally similar within the BIC-P analyses and the TP analyses, differing little from the respective grid coefficients in Table 1. This implied that variability hardly decreased at the quadrate level and that the majority of the observed variability in soil P concentration was occurring within the quadrates at the microscale level.

Sample Size Estimates

Because a relatively large number of samples were taken (108) it was considered reasonable to assume that the phosphorus concentrations were normally distributed (by central limit theorem) and that the sample s approximated the real σ . This enabled the relationship between sample size and confidence interval of the mean to be calculated for P fractions in the experimental soils. The calculations were performed on the data in Table 6 for several levels of confidence using the equation

$$n = \left(\frac{z \cdot s}{\bar{x} - \mu} \right)^2$$

where n = sample size

$(\bar{x} - \mu)$ = error of estimate (confidence interval).

$z = 1.64$ for 90% confidence

$z = 1.96$ for 95% confidence

$z = 2.57$ for 99% confidence

and the results are shown in Table 7.

Table 6 Total sample mean, standard deviation and coefficient of variation of BIC-P and TP for Joel loamy sand

	BIC-P	TP
\bar{x}	8.8	44.7
s	2.8	11.6
CV	0.32	0.26

Table 7 Sample size estimates with varied confidence intervals and confidence levels for Joel loamy sand

confidence interval	<u>BIC-P confidence level</u>			<u>TP confidence level</u>		
	90	95	99	90	95	99
$\mu \pm 5\%$	109*	156	267	72	103	178
$\mu \pm 10\%$	27	39	67	18	26	44
$\mu \pm 15\%$	12	17	30	8	11	20

*ie to be 90% certain that sample mean is within $\pm 5\%$ of the actual soil P concentration, 109 soils cores must be taken.

The larger sample sizes estimated for BIC-P sampling reflected the greater variability observed in the soil BIC-P fraction, as measured by the coefficient of variation (Table 6).

Coolup Sandy Loam (Cs1)

Large Scale Variation

The results in Table 9 of an ANOVA performed on the data in Table 8, show, unlike the Jls, that no significant differences were observed in soil P concentrations over the 40 m distance separating grids.

Table 8 Grid mean (\bar{x}), standard deviation (s) and coefficient of variation (CV) of BIC-P and TP for Coolup Sandy loam

	Grid 1		Grid 2	
	BIC-P	TP	BIC-P	TP
\bar{x}	40.3	210.1	37.3	205.9
s	15.8	59.7	17.0	50.8
CV	0.39	0.28	0.46	0.25

Table 9 ANOVA F values for large scale variation (40m) on Coolup sandy loam

Comparison	BIC-P	TP	Significance
Between grids	0.890	0.155	P(F>3.94)=0.05

Small Scale Variation

Since no significant difference was found between grids, an ANOVA was done between all 12 quadrates (Tables 10,11) to test for soil P variation over the smaller 5 m distances. Both BIC-P and TP were found to show significant variation (P<0.01) over a 5 m radius as shown in Table 12.

Table 10 Quadrate means, standard deviations and coefficients of variation for BIC-P analyses on Coolup sandy loam

		quad 1	quad 2	quad 3	quad 4	quad 5	quad 6
Grid 1	\bar{x}	31.3	45.6	47.9	19.3	43.9	53.6
	s	6.9	15.6	9.4	4.7	9.5	16.3
	CV	0.22	0.34	0.20	0.24	0.22	0.30
Grid 2	\bar{x}	17.9	37.9	31.7	44.1	59.3	32.8
	s	5.0	9.2	8.7	17.1	12.6	14.3
	CV	0.28	0.24	0.27	0.39	0.21	0.44

Table 11 Quadrate means, standard deviations and coefficients of variation for TP analyses on Coolup sandy loam

		quad 1	quad 2	quad 3	quad 4	quad 5	quad 6
Grid 1	\bar{x}	156.7	198.9	256.7	192.2	228.9	202.2
	s	26.0	39.8	59.8	29.1	52.3	34.6
	CV	0.17	0.20	0.23	0.15	0.23	0.17
Grid 2	\bar{x}	167.4	210.0	228.9	208.0	242.2	203.3
	s	34.6	47.4	54.7	87.4	62.4	43.9
	CV	0.21	0.23	0.24	0.42	0.26	0.22

Table 12 ANOVA F values for small scale variation (5 m)

Comparison	BIC-P	TP	Significance
Between quadrates	14.39	3.86	P(F>1.88)=0.05
of both grids			P(F>2.43)=0.01

Microscale Variation

The coefficients of variation in Tables 10,11 showed that variability was generally slightly higher for the BIC-P data at the microscale level (0.3m). A comparison of quadrate coefficients with respective grid coefficients of variation (Table 8), showed a slight decrease in variability at the quadrate level, although still large. This indicated that most of the observed variability for the Csl was found within the 1 m² quadrates at distances of 0.3 m or less.

Sample Size

The relationship between sample size and confidence interval of the mean soil P concentration at various confidence levels for the Csl was calculated by the same method used for the Jls (Tables 13,14).

Table 13 Total sample mean, standard deviation and coefficient of variation for BIC-P and TP on Coolup sandyloam

	BIC-P	TP
\bar{x}	38.8	208.0
s	16.4	55.2
CV	0.42	0.27

Table 14 Sample size estimates with varied confidence intervals and confidence levels for Coolup sandy loam.

confidence interval	BIC-P confidence level			TP confidence level		
	90	95	99	90	95	99
$\mu \pm 5\%$	192	275	472	76	108	186
$\mu \pm 10\%$	48	60	118	19	27	47
$\mu \pm 15\%$	21	31	52	8	12	21

As with the Jls, the larger sample sizes calculated for BIC-P sampling on the Csl reflected a greater variability observed in the soil BIC-P fraction (shown by the higher coefficient of variation in Table 13).

DISCUSSION

Both soils showed similar spatial variability patterns in the BIC-P and T P fraction. The BIC-P fraction was found to be more variable, and the majority of variation encountered in both P fractions appeared to be at the microscale level ($\leq 0.3\text{m}$). The probable causes of this variability are factors such as faecal remnants, clumped pasture distribution, scouring of the soil surface and analytical error.

The difference between sample concentrations was generally found to exceed the maximum possible analytical error between chemical determinations, thus ruling out analytical error as the only cause of observed microscale variation.

For the Jls spatial variability of P concentration was also observed to be high at the larger 5 m and 40 m scales. The larger scale variability (or lack of for Csl) may be a site specific phenomenon and more replications at the 40 m scale would be needed to determine its extent. Changes in soil P would certainly be expected over these larger distances, correlating with landscape features such as swamps and old sand dunes.

The observed variation at the 5 m scale in both soils was most likely real, however, since the majority of variability occurred within 0.3 m distances, more than 9 samples were ideally needed from each quadrat. Sample sizes similar to those calculated in Tables 7 and 14 would be required from each quadrat to give more reliable quadrat means for ANOVA comparisons.

Since most P variability is found at the microscale level, experimental plots of up to 0.5 ha or more, depending on topography, require a sample size of 25-30 cores to be 95% confident of being within $\pm 10\%$ of mean TP concentration for both soils (Tables 7, 14). When sampling for BIC-P concentration a larger sample size is needed, depending on soil type. For a Jls 40 cores, and for a Csl 60 cores are necessary to be 95% confident of being within $\pm 10\%$ of the mean. However, if such a high confidence is not needed, sample size may be reduced by reducing the required confidence, eg to 90% (Tables 7,14).

The required confidence interval also controls sample size (Fig 3a, b). If the confidence interval is reduced to $\pm 5\%$ a very large sample size is necessary (275 for BIC-P on a Csl at the 95% level of confidence). Clearly there are reduced returns for the effort taken. Any decisions made concerning sample size calculations should be made after considering the practicalities of sampling in a field situation and the experimental requirements. As a general guide a composite of 25-30 samples from an experimental plot for T P analysis and 40-50 samples for BIC P analysis would be recommended as a sample size for general work on the soils described.

PREDICTION OF SAMPLE NUMBER

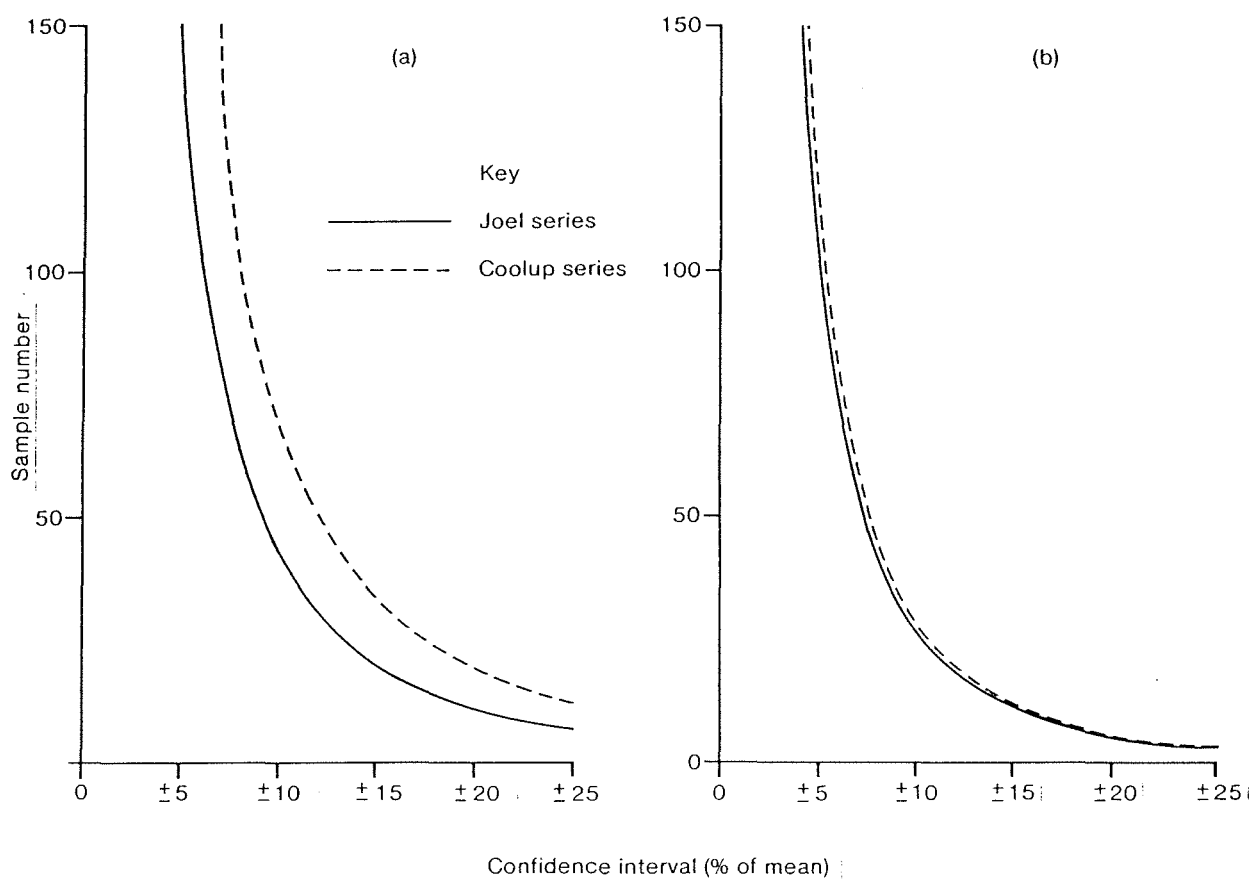


Figure 3. Sample number as determined by the confidence interval at the 95% level of confidence for a) Bic-P, b) TP

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