# A Comparison of Sampling Methods for Soil-test Phosphorus

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### ABSTRACT

Banded fertilizers and injected manure can increase the variability of soil-test phosphorus (STP) measurements, leading to difficulties in obtaining representative soil samples. A frameexcavation sampling method was compared with a number of different coring methods to determine if the STP variability could be affected by method. Phosphorus was banded as injected hog manure, inorganic fertilizer, and biosolids at four small-plot sites in Alberta. Soils were sampled using a steel frame, which was 11-cm wide by two times the distance between bands of applied phosphorus. The coring methods included 5-, 10-, and 20-core composite samples using a 2-cm diameter hand probe. Directed-core (a proportional ratio of in-band to between-band samples) and 10-core/4-cm diameter core methods were also used. Soils were sampled at incremental layers of 0 to 2.5 cm, 2.5 to 5 cm, and 5 to 15 cm. Dried and ground subsamples were removed by weight for laboratory analysis of STP and levels in the 0- to 2.5-cm, 0- to 5cm, and 0- to 15-cm layers were calculated. Mean STP was significantly different among a few of the sampling methods at two sites, but results were not consistent. Standard deviations did not differ significantly among the methods at any of the sites, regardless of soil layer, except for the 0- to 2.5-cm and 0- to 5-cm layers at the biosolids site. The standard deviations for the frameexcavation method tended to be among the lowest measured where high levels of phosphorus  $(>176 \text{ mg kg}^{-1})$  had been applied. In the soil layers where STP increases were greatest, the number of samples needed to measure STP within 10% of the mean was reduced using the frame-excavation method for three of the four study sites. The time requirement for the frameexcavation method was similar to taking a 10-core composite sample, and about half that of taking a 20-core composite sample. For soils with low levels and low inputs of phosphorus, a composite of at least 5-cores is adequate for determining STP. For soils with high phosphorus contents, the frame-excavation method may provide a sample that is equally representative as the 10- and 20-coring methods, with a tendency towards lower standard deviations, reduced requirements for number of replicates needed to measure an accurate mean, and reduced sampling time.

The frame-excavation and 10-core methods were also compared at seven field-scale sites throughout Alberta. Five sites were non-manured and two sites had received manure for several years. Soil samples from the 0- to 15-cm layer were collected from a total of 50 sampling points. A 4-g subsample was removed from the frame-excavation samples and a 4-cm<sup>3</sup> subsample was removed from the 10-core samples for STP analysis. There was a significant positive relationship  $(r^2 = 0.93)$  between the two methods; although, the 10-core method STP values were about 28% lower than the frame-excavation method STP values. The difference was similar for the non-manured and manured sites. Although the study design did not allow for separation of the sampling method (frame and core) and analytical method effects (weight versus volume subsample analysis), or their interactions, we suggest that a more precise measure of STP would result from weighing subsamples for laboratory analysis, rather than removing subsamples by volume.

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#### INTRODUCTION

The distribution of soil-test phosphorus (STP) within farm fields is variable due to natural variation, soil redistribution by erosion and tillage, and management practices such as non-uniform manure application. As such, the assessment of STP status for determining the risk of phosphorus loss is uncertain and problematic (Page et al. 2005). In addition to the field-scale variability of STP, the practice of banding inorganic fertilizer and injecting manure can increase variability at point scales. Morton et al. (2000) found that 38% of the total variance of STP was measured within 1 m of a fixed point, 27% within 100 m, and 35% between fields within a farm block. The variability of STP measurements has been identified as a weakness for the soil threshold approach to assessing levels of phosphorus in farm fields (Feagley and Lory 2005). It is important for producers, researchers, and for environmental protection that STP levels are measured accurately in agricultural soils at field and point scales.

Phosphorus forms strong chemical bonds in soil and is not nearly as mobile as other nutrients such as nitrate nitrogen (Vadas et al. 2005). Banded applications of inorganic fertilizer and other sources of phosphorus can result in a residual buildup of STP levels. Kitchen et al. (1990) showed that after 2 yr, fertilizer bands remained within 5 cm of the original position of application and that STP levels declined exponentially to near-background levels 5 cm away from the middle of the band. Measurements of STP ranged from 0 to 80 mg kg<sup>-1</sup> with banded fertilizer, depending on where the soil was sampled (Westfall et al. 1991). Widths between bands typically vary from 15 to 30 cm, but can be as much as 50 cm apart. Band depths vary from 1 to 6 cm, depending on crop type and moisture conditions. Soil sampling must take into account high concentrations of phosphorus in the bands and low concentrations between bands, plus any residual bands from previous years. Kitchen et al. (1990) reported that the highest deviation from a true level of STP occurs when the residual fertilizer band is over-represented by soil sampling, leading to underestimations of fertilizer recommendations. When high rates of phosphorus are applied in bands, such as when liquid manure or biosolids are injected into agricultural fields, these questions of accuracy and variability are amplified.

The recommended method of soil phosphorus sampling is from 0 to 15 cm, using 10 to 30 soil cores per composite sample, at a minimum density of one sampling point per 10 ha (Coale 2000). However, this method can result in misrepresentation of STP levels if the composite soil sample contains a disproportionate amount of cores from either the fertilizer band or from between bands (Ashworth et al. 1994). The alternative of increasing the required number of cores is time-consuming and difficult to accomplish in busy fall sampling periods. Taking a smaller number of core samples may result in a greater chance of not obtaining a proportional sample within bands and between bands.

Various soil core-sampling strategies to collect representative proportions of soil in the fertilizer band and between bands for the composite sample have been tested. Some researchers reported that random sampling is the best way to obtain an accurate representation of STP (Tyler and Howard 1991). Kitchen et al. (1990) developed a systematic soil sampling formula to predict the ratio of between-band cores to within-band cores needed to represent the whole soil. Zebarth et al. (2002) found that the best strategy for sampling soils where phosphorus had been banded was to collect core samples from the crop row, the fertilizer band, the centre of the inter-row, and

midway between the fertilizer band and the centre of the inter-row. Mahler (1990) concluded that although a systematic soil sampling method was probably more accurate than the random method for non-mobile nutrients (phosphorus and potassium), soil-test correlation data bases may not allow accurate interpretation of the higher levels of nutrients that were measured. However, Westfall et al. (1991) concluded that it is not possible to take enough cores to obtain an accurate measure of STP levels if high rates of phosphorus fertilizer are applied in wide (76 cm) bands.

Soil sampling techniques that improve the representation of the proportion of banded phosphorus in soil relative to the coring techniques have also been explored. The slot-sampling method outlined by Ashworth et al. (1994) involves the use of a modified chainsaw to cut a 15cm long section of soil across the fertilizer bands. This method was shown to significantly reduce the variability of STP levels in soil samples compared with a composite of 12 core samples. James and Hurst (1995) also recommended taking a slice of soil perpendicular to fertilizer bands after calculating the prohibitive number of random soil samples required to represent a field with banded fertilizer. Although many strategies for obtaining representative proportions of soils banded with fertilizer have been offered, there is no accepted method at present.

One of the challenges of identifying an appropriate sampling method is the difficulty in assessing the accuracy of different methods. Researchers have used a variety of mathematical approaches to estimate true soil-test levels. Zebarth et al. (2002) used the results of an unbiased, random, sampling strategy where all inter-row locations were sampled equally to represent true values. James and Hurst (1995) tried to simulate a phosphorus-enriched band thoroughly mixed throughout the bulk soil by averaging STP values of soil cores sampled in a study where inorganic phosphorus fertilizer was banded in a corn field. Zerkoune et al. (1994) and Kitchen et al. (1990) divided the area of an integrated decay model of STP distribution by the band spacing to represent the true value of STP. Although it appears that a true STP level can only be approximated, the accuracy of different soil sampling methods can be assessed based on whether or not variability is reduced.

Although soil samples for assessing the nutrient sufficiency for crops are usually taken from 0 to 15 cm, Sharpley et al. (1978) and Sharpley (1985) have shown that the top few centimeters of soil are the primary zone of interaction where runoff can carry dissolved and particulate phosphorus from fields to riparian areas. A pattern of increased levels of phosphorus in soil surface horizons has resulted from gradual build-up of fertilizers where accumulations have built up at the 10-cm depth under all tillage systems (Crozier et al. 1999), but in particular where phosphorus fertilizer was banded (Grant and Bailey 1994; Malhi et al. 2003). In reduced tillage and pasture systems, phosphorus fertilizers do not have an opportunity to become evenly distributed throughout the topsoil (Crozier et al. 1999). Instead, the fertilizers become vertically stratified, with increased levels in the surface 5 to 7 cm and decreases with depth (Andraski et al. 2003). After 12 yr of no-till, continuous wheat in western Canadian cropping systems, Selles et al. (1999) measured an accumulation of plant-available phosphorus in the surface 5 cm of soil. Guertal et al. (1991) measured up to three times more STP in the 0- to 2-cm layer than in the 0to 8-cm layer in long-term, no-tillage plots. Banding phosphorus fertilizer can also result in little or no fertilizer in the surface 5 cm of soil, but increased concentrations in the 8- to 16-cm layers or lower, depending on where fertilizer bands were placed (Janssen et al. 1998).

Since the concentration of phosphorus in runoff water increases as concentration of phosphorus in soils increases (Sharpley et al. 2001), accurate measures of STP concentrations in the surface few centimetres, which interact with rainfall or snowmelt and contribute to runoff losses, are needed. However, Vadas et al. (2005) pointed out that the variation among samples may be reduced for deep agronomic samples compared to shallow samples, due to less relative interference from thatch layers or crop residues, which increase the difficulty of determining STP levels at the soil surface. To assess the effects of possible increases in concentrations of STP on phosphorus concentrations in runoff, a soil sampling method is needed to obtain a representative sample of phosphorus in soil at shallower depths than those used for assessing crop nutrient requirements.

A frame-excavation method of soil sampling was developed to obtain representative soil samples across bands of applied phosphorus. The objective of this study was to determine whether the frame-excavation method of sampling decreased STP variability, and thus improved the accuracy of STP measurements compared to a variety of coring methods. A comparison of the time requirements for each sampling method was also included.

#### **MATERIALS AND METHODS**

#### **General Description of Sampling Methods**

An 11- by 60-cm steel frame was used to outline an excavation area (Fig. 1). A 5-cm deep frame was used for untilled soils and a 10-cm deep frame was used for tilled soils to prevent the collapse of side-walls in loose soil conditions. The length of the sampling area was adjusted to two times the distance between bands of applied phosphorus by bolting an adjustable divider inside the frame. All plant material at the soil surface was removed before sampling. The frame was placed perpendicular to the direction of fertilizer banding or manure injection and driven into the ground until the top of the frame was even with the soil surface. Where the soil surface was ridged, the frame was driven into the ground so that the top of the frame was about halfway between the ridges and the valleys. The frame was secured in place with four 15-cm long steel pins, two along each side of the frame.

Soil was excavated from the 0- to 2.5-cm, 2.5- to 5-cm, and 5- to 15-cm layers using a 2.5cm deep scoop, a 5-cm deep scoop, and a shovel, respectively (Fig. 1). The excavated soil from each of the top two layers was well mixed in a bucket. The larger volume of sample removed from the 5- to 15-cm layer was mixed with a shovel and then with a trowel on a tarp. Randomly selected subsamples were then removed to create a composite sample of about 500 g. Samples were bagged, packed in coolers with ice packs, and transported to the laboratory within 24 h. Soils were dried, ground (< 2 mm), and remixed in the lab prior to analysis.

Sets of 5, 10, and 20 soil core composite samples were taken equal distance along the circumference of a circle 2 to 3 m in radius from the frame, depending on the site (Figs. 2 and 3). Core sampling locations were determined by looking upwards while placing the core on the soil surface to avoid biased sampling either between crop rows or in the row. Soil cores were taken with a core tube and then sliced into 0- to 2.5-cm, 2.5- to 5-cm, and 5- to 15-cm layers (Fig. 2).



**Fig. 1.** Frame-excavation sampling technique showing (a) tools, (b) frame installation, (c) removal of the top two layers (0 to 2.5 cm and 2.5 to 5 cm) with scoops, and (d) removal of the third layer (5 to 15 cm) with a shovel.

At three of the study sites, a directed-core method was used by taking one core from within a band of applied phosphorus and a prescribed number of cores between bands, according to the recommendation of Kitchen et al. (1990) for sampling when phosphorus-band locations are known (Equation 1). A composite sample was then prepared by combining the within-band and between-band core samples.

(1)

$$S = 8 [BS/(30)]$$

Where: S = the number of core samples between bands BS = band spacing (cm)

Composite samples were prepared by mixing the soil from each layer for each core set (i.e. 5, 10, or 20 cores). The composite samples were well mixed and about 100 to 500 g of soil were removed, depending on the amount of total sample. After being transported in coolers to the lab, samples were dried, ground (< 2 mm), and remixed prior to removing a 4-g subsample for analysis. Additional details about soil sampling methods used at each study site are described below.

### **Small-plot Sites**

Wainwright manure site. A site was established in a field about 22 km north of Wainwright, Alberta, on a Thin Black Chernozemic loam textured soil. The field was used to grow a cereal crop during the 2003 growing season. Soils were sampled on October 31, 2003. Approximately 1 mo prior to soil sampling, liquid hog manure was injected at a depth of 10 to 15 cm below the surface on a 30-cm spacing at a rate of 60 kg ha<sup>-1</sup> total phosphorus. This rate was comparable to what may be applied based on land requirements for liquid hog manure (farrow to finish) in the Black Soil Zone, and is typical of manure production of hogs, and of



Fig. 2. Core sampling method.

the total phosphorus content in hog manure as outlined in the Agricultural Operation Practices Act (Province of Alberta 2001, 2004). This rate is higher than the Alberta Fertilizer Guide application rates of phosphorus that range from 5 to 24 kg ha<sup>-1</sup> phosphorus in the Thin Black Soil Zone (AAFRD 2004). The manure storage tank had been agitated for approximately 24 h prior to injecting the manure, and the injection bands were clearly visible at the time of sampling.

Samples were collected in the centre of a 2-m radius circle using the frame-excavation method and around the circumference of the circle using the 5-, 10-, and 20-core sampling methods with a 2-cm diameter core tube (Fig. 3). In addition, soil samples were collected to characterize STP levels within and between bands using a composite sample of 10 cores from the 0- to 15-cm layer taken in the bands and 10 cores from the 0- to 15-cm layer taken between the bands. All soil-sampling methods were repeated three times (Appendix Fig. A1.1a). Directed-core sampling, using Equation 1, was not carried out at this site.

**Lethbridge manure site.** A site was established about 3 km southeast of Lethbridge, Alberta, on an Orthic Dark Brown Chernozemic loam to silt loam textured soil with standing cereal stubble. Liquid hog manure was injected with a knife-chisel-blade implement at a rate of 90,000 L ha<sup>-1</sup> (Fig. 4). The injector spacing was 30 cm and the injection depth was 12 to 15 cm. The hog manure contained 96% moisture, 590 mg L<sup>-1</sup> total phosphorus, and 54 mg L<sup>-1</sup> orthophosphate phosphorus. The application rate delivered 53 kg ha<sup>-1</sup> of total phosphorus. This rate was comparable to or slightly higher than what may be applied based on land requirements for liquid hog manure (farrow to finish) in the Dark Brown Soil Zone, and is typical of the manure production of hogs, and the total phosphorus content in hog manure, as outlined in the



Phosphorús bands on 23 to 30 cm spacing

Fig. 3. Layout of frame-excavation and core sampling points.

Agricultural Operation Practices Act (Province of Alberta 2001, 2004). This rate of applied phosphorus was also higher than the Alberta Fertilizer Guide value as the typical range of 5 to 20 kg ha<sup>-1</sup> phosphorus required in the Dark Brown Soil Zone (AAFRD 2004). The hog manure was injected on October 8, 2004, and time was allowed for equilibration with the soil before soil sampling was carried out on May 4, 2005.

The design of the experiment included two treatments (with and without added phosphorus), replicated four times in a randomized complete block arrangement (Appendix Fig. A1.2a). The experiment consisted of eight plots, each 7.5 by 10 m in size. Soil sampling included the frame-excavation, 5-core, 10-core, and 20-core methods (Fig. 3). The core samples were taken in a 3-m radius from the frame sampling point. Directed-core samples were also taken and consisted of one core from within a phosphorus band and eight cores between bands for the 30-cm spacing (Equation 1). A 2-cm diameter soil hand probe was used for all coring, except the 10-core method was repeated using a 4-cm diameter core tube to represent a truck-mounted core sampler. The time required to take each sample set was also recorded.

**Lethbridge fertilizer site.** This site was established beside the Lethbridge Manure site. The experimental design was essentially the same as for the liquid hog manure site, except that inorganic fertilizer (11-52-0) was used as the phosphorus source (Appendix Fig. A1.2b). The inorganic fertilizer was banded with a disc-opener applicator with packers at a rate of 176 kg ha<sup>-1</sup> elemental phosphorus (i.e., 400 kg ha<sup>-1</sup> P<sub>2</sub>O<sub>5</sub>). Although this rate was much higher than would normally be applied for agronomic purposes, high concentrations of applied phosphorus were used to ensure that differences in phosphorus levels would be apparent. The width between the bands was 23 cm and the depth of phosphorus placement was about 9 cm. The phosphorus



**Fig. 4.** Equipment used to inject hog manure at the Lethbridge Manure site.

fertilizer was banded on October 29, 2004, and was allowed to equilibrate with the soil before the samples were removed on May 3, 2005. The soil sampling methods described for the Lethbridge Manure site experiment were also used at this site, although the directed-core method consisted of one core within a phosphorus band and six cores between bands, as prescribed for the 23-cm band spacing (Equation 1). The time required to take each sample set was also recorded.

**Calgary biosolids site.** Soil samples were collected from a biosolids injection site on a Black Chernozemic loam-textured soil on the eastern edge of Calgary, Alberta. Bands of biosolid material were injected with a Terra-Gator applicator at a rate of 10 Mg ha<sup>-1</sup> (City of Calgary 2005) to a 10-cm depth on a 23-cm spacing, approximately 2 wk prior to sampling on September 14, 2004. The soil sampling of the two treatments (with and without added biosolids) was replicated three times for a total of six plots (Appendix Fig. A1.1b). The plots were sampled using the frame-excavation, 5-core, 10-core, and 20-core methods. A 2-cm diameter core sampler was used, and the cores were taken in a 3-m radius around the frame sample. A directed-core sample, consisting of one core from within the phosphorus band and six cores between bands was also collected, based on the 23-cm spacing (Equation 1). A set of 10-core samples was taken using a 4-cm diameter core tube to represent a truck-mounted core sampler. The time required to take each sample set was also recorded.

#### **Microwatershed Study Sites**

The frame-excavation method was also compared with a commonly used agronomic method of sampling and analyzing for STP. This study was conducted in cultivated fields within seven small watersheds (26 to 248 ha) as part of a 3-yr study to determine the relationship between STP and phosphorus in runoff, referred to as the Microwatershed Study (Little et al. 2006). The seven microwatersheds used to compare the two soil sampling methods were Crowfoot Creek (CFT), Grand Prairie Creek (GPC), Renwick Creek (REN), Three Hills Creek (THC), Wabash Creek (WAB), Lower Little Bow (LLB), and Ponoka (PON) (Appendix Fig. A1.3). Five of the sites were non-manured (CFT, GPC, REN, THC, WAB) and two sites were manured (LLB, PON). Solid cattle manure had been applied at both manured sites for several years. Manure had been applied at least 1 yr prior to sampling at both sites and had been incorporated by discing followed by seeding and tillage operations. Soils within all sites were medium textured, and levels of organic matter in the soil surface ranged from 4.3 to 10.0% (Table 1).

The frame-excavation method was used to sample 0- to 2.5-cm, 2.5- to 5-cm, 5- to 15-cm depths along a transect of upper, mid, and lower landform positions within the microwatersheds. A set of 10-core composite samples from the 0- to 15-cm layer was also collected in a radius about 3 m from the frame-excavation sampling points for comparison (Fig. 3). Since different soil conditions were encountered in the microwatershed sites at the time of sampling, either 2-cm hand-probe cores, or 5-cm diameter hand augers were used to remove the core samples. A truck-mounted 4-cm diameter core tube was used in 2003 to sample at the LLB site. All soil samples used in this study were collected in the fall of 2003 and 2004. The total number of sampling points used was 22 in 2003 (1 to 7 per site) and 28 in 2004 (2 to 6 per site). Composite samples were well mixed in the field and approximately 500 g were removed and sent to a private laboratory for analysis. Samples were air-dried and ground to pass through a 2-mm sieve.

#### **Soil Analysis**

Soil samples were analyzed for STP using the modified Kelowna method (Qian et al. 1991). Results were expressed for the 0- to 2.5-cm, 0- to 5-cm, and 0- to 15-cm layers, except for the Microwatershed Study sites where results were expressed for the 0- to 15-cm layer. The 0- to 5-cm and 0- to 15-cm values were calculated using results from the 0- to 2.5-cm, 2.5- to 5-cm and 5- to 15-cm layers.

The method of handling subsamples in the laboratory for extraction analysis of STP differs between samples used for environmental purposes and those used for agronomic purposes. All frame samples and all core samples, except for the core samples from the seven Microwatershed Study sites, were treated as environmental samples. The core samples from the Microwatershed Study were treated as agronomic samples.

For environmental samples, a 4-g subsample was removed by weight and extracted in 40 mL of the modified Kelowna extract solution. The phosphorus concentration of the filtered extract was reported in mg L<sup>-1</sup> and then converted to mg kg<sup>-1</sup> by multiplying by the extraction ratio (i.e., 10). Samples for agronomic purposes were analyzed by removing a 4-cm<sup>3</sup> subsample for

SILCS.							
			Added				Organic
	Area		phosphorus	Slope		Clay <sup>x</sup>	matter <sup>x</sup>
Site	(ha)	Management <sup>z</sup>	$(kg ha^{-1}yr^{-1})$	(%)	Texture <sup>y,x</sup>	(%)	(%)
CFT <sup>w</sup>	248	NT	17 - 22	1 - 4	L/SiL	21	5.3
GPC	62	СТ	10 - 21	1 - 4	CL/C	29	7.5
REN	26	RT	22-28	1 - 8	L/SL	15	6.6
THC	51	NT	15 - 25	0 - 6	L / L	23	10.0
WAB	33	СТ	15 - 17	1 - 4	L/CL	20	4.3
LLB <sup>v</sup>	88	СТ	Moderate <sup>u</sup>	1 - 2	L/CL	26	4.5
PON	30	СТ	High <sup>t</sup>	0 - 5	L / CL	12	9.6

**Table 1.** Characteristics and management information for the seven Microwatershed Study sites.

<sup>z</sup> CT = conventional tillage, RT = reduced tillage, NT = no tillage before seeding.

<sup>y</sup> Surface/subsurface.

<sup>x</sup> At midslope landform positions.

<sup>w</sup>Some grazing.

<sup>v</sup> Irrigated.

<sup>u</sup> Moderate - manured once every 2 to 3 yr.

<sup>t</sup>High - manured one to two times per year.

extraction with 40 mL of extract solution. The resulting mg  $L^{-1}$  values measured in the soil extract obtained from the volume-based samples were converted to mg kg<sup>-1</sup> of soil, using a mean bulk density of 1.18 Mg m<sup>-3</sup> for dried and ground mineral soil samples, where 4 cm<sup>3</sup> soil x 1.18 g cm<sup>-3</sup> = 4.7 g soil. The 4.7-g soil and 40-mL extractant solution were equal to a soil-to-extractant ratio of 8.5. The mg kg<sup>-1</sup>value of STP was obtained by multiplying the mg L<sup>-1</sup> value by the extraction ratio of 8.5 (Brandon Green, Senior Agronomist, Enviro-Test Laboratories, personal communication).

## **Statistical Analysis**

The Statistical Analysis System program (SAS Institute Inc. 2000) was used to calculate means and standard deviations using Proc Summary. Differences among means were tested using mixed-model analysis (Proc Mixed) and the Tukey-Kramer test with a significance level of P < 0.05. Differences among standard deviations were tested using a Folded F-test with a Bonferroni adjustment to identify significance levels that were appropriate for the number of comparisons. Because the Bonferroni adjustment is conservative, some observed trends were discussed without applying the Bonferroni factor. The number of replicates needed to accurately measure various layer means was estimated using Stein's two-stage sampling formula (Stein 1945). The concordance correlation coefficient analysis (Lin 1989) was used to evaluate the two sampling methods used at the Microwatershed Study sites.

#### **RESULTS AND DISCUSSION**

### **Small-plot Sites**

**Wainwright manure site.** Mean STP values and standard deviations were not significantly different among sampling methods for each soil layer (Table 2). The between-band value can be assumed to represent the STP status in the 0- to 15-cm layer prior to manure injection and it was expected that STP content would increase in the within-band zone. However, the within-band value of STP was only 9 mg kg<sup>-1</sup> more than the between-band STP value at this site and the

**Table 2.** Mean soil-test phosphorus (STP), standard deviation (SD), standard error (SE), and coefficients of variation (CV) for different soil sampling methods at the Wainwright Manure site<sup>z</sup>.

Sampling		STP mean	SD	SE	CV
method	n		$- (mg kg^{-1})$		(%)
		0- to 2.5-cn	ı layer		
5 cores	3	31 a <sup>y</sup>	7 a	4	22
10 cores	3	35 a	6a	4	18
20 cores	3	23 a	3 a	2	12
Frame	3	25 a	10 a	6	40
Layer mean		28	6	4	23
		0- to 5-cm la	iver		
5 cores	3	30 a	6a	4	21
10 cores	3	32 a	7 a	4	20
20 cores	3	23 a	3 a	2	12
Frame	3	26 a	7 a	4	27
Layer mean		28	6	3	20
		0- to 15-cm l	aver		
5 cores	3	25 a	6a	3	24
10 cores	3	28 a	5 a	3	18
20 cores	3	20 a	2 a	1	12
Frame	3	23 a	2 a	1	9
Layer mean		24	4	2	16
Within band	3	31 a			
Between band	3	22 a			
Difference		9			

<sup>z</sup> Hog manure was injected between 10 and 15 cm deep.

<sup>y</sup> Values within the same soil layer followed by the same letter are not significantly different at P < 0.05.

means were not significantly different (Table 2). If all of the applied rate of 60 kg ha<sup>-1</sup> total phosphorus was evenly distributed throughout the top 15-cm layer, and the soil bulk density was 1.3 Mg m<sup>-3</sup>, then the STP would have increased by about 31 mg kg<sup>-1</sup>. Since the manure was banded, it was expected that the increase in STP concentration in the within-band zone compared to the between-band zone would have been greater than 31 mg kg<sup>-1</sup>. However, it is estimated that only 70% of the applied phosphorus from manure is in an extractable form in the first year after application (Eghball et al. 2002). Since, only 1 mo elapsed between manure application and soil sampling, which was carried out in cool, fall conditions, it was likely that less than 50% of the total phosphorus was present in an extractable form. As well, the injected manure may have been distributed laterally and reduced the concentration effect in the within-band zone, reducing the difference between the two zones.

A banding effect from the applied manure phosphorus was not expected in the two top layers since the target depth of injection was 10 to 15 cm. Differences among sampling methods were not observed in the 0- to 15-cm soil layer, since the banding effect of applied manure phosphorus from a single application only 1 mo after application in cool, fall conditions was essentially undetectable.

**Lethbridge manure site.** The higher STP (26 mg kg<sup>-1</sup>) in the 0- to 2.5-cm layer and lower STP (14 mg kg<sup>-1</sup>) in the 0- to 15-cm layer of the control plots were consistent with levels expected in non-manured soils (Table 3). Researchers have described a similar pattern of increased levels of phosphorus in the soil surface resulting from a variety of fertilizer management scenarios (Crozier et al. 1999). The mean STP and the standard deviations among sampling methods were not significantly different for each of the three soil layers in the control plots, indicating relatively uniform initial conditions. The coefficients of variation (CV) calculated from the average values of the mean and standard deviations of all sampling methods for each soil layer in the control samples ranged from 17 to 26%.

The application rate of the liquid hog manure was 53 kg ha<sup>-1</sup> total phosphorus. If all of the applied phosphorus was evenly distributed throughout the top 15-cm layer, and the soil bulk density was 1.3 Mg m<sup>3</sup>, the STP would have increased by about 28 mg kg<sup>-1</sup>. An 8-mo period of equilibration occurred between the time manure was injected on October 8 and soils were sampled on May 4. It is estimated that about 70% of the applied phosphorus may be present in an extractable form in the first year after application (Eghball et al. 2002). A portion of the applied total phosphorus would have already been in extractable form at the time of application plus a portion of the organic phosphorus would have mineralized to extractable phosphorus in the few months of unfrozen, moist conditions in the early spring. This would give an expected increase of about 20 mg kg<sup>-1</sup>. A larger increase would be expected where phosphorus was concentrated in bands at the targeted injection depth of 12 to 15 cm.

The injected hog manure increased STP content to close to expected levels in all soil layers (Table 3). The mean increase was 16 mg kg<sup>-1</sup> in the 0- to 2.5-cm layer, 19 mg kg<sup>-1</sup> in the 0- to 5- cm layer, and 12 mg kg<sup>-1</sup> in the 0- to 15-cm layer. Increases in STP in all soil layers suggest that the liquid manure was applied throughout the 0- to 15-cm layer rather than just at the targeted injection depth of 10 to 15 cm. There were no significant differences among the STP means of

Table 3. Mean so	oil-tes	t phosphorus	(STP),	standar	d devia	tion (SD), sta	ndard er	ror (SE),	and
coefficient of var	iation	(CV) for dif	ferent so	oil sam	pling m	ethods at the	Lethbrid	ge Manu	re site <sup>z</sup> .
Control Hog							Hog ma	anure	
Sampling		STP mean	SD	SE	CV	STP mean	SD	SE	CV
method	n	(mg	g kg <sup>-1</sup> )		(%)	(1	$mg kg^{-1}$ )		(%)
			0- te	o 2.5-cr	n layer				
5 cores	4	27 a <sup>y</sup>	6 a	3	21	57 a	40 a	20	70
Directed cores <sup>x</sup>	-	ns	-	-	-	35 a	9 a	5	27
10 cores	4	26 a	6 a	3	24	42 a	6 a	3	15
10 cores/4 cm	4	26 a	4 a	2	16	30 a	13 a	7	44
20 cores	4	25 a	3 a	2	12	44 a	21 a	10	46
Frame	4	27 a	3 a	2	11	47 a	25 a	12	53
Layer mean		26	4	2	17	42	19	9	43
			<i>0- i</i>	to 5-cm	layer				
5 cores	4	23 a	7 a	4	30	53 a	35 a	18	66
Directed cores	-	ns	-	-	-	34 a	9 a	5	27
10 cores	4	22 a	6 a	3	25	46 a	14 a	7	29
10 cores/4 cm	4	24 a	6 a	3	27	34 a	10 a	5	28
20 cores	4	23 a	7 a	3	28	45 a	19 a	10	43
Frame	4	24 a	3 a	2	12	42 a	14 a	7	33
Layer mean		23	6	3	24	42	17	8	38
			0_ t	o 15-cm	n lavar				
5 cores	1	13 a	1 a	ווס-כוו ר	32	26 ah	13 9	6	18
Directed cores	4	15 a	4 a	2	52	20 a0 17 b	13 a 5 a	2	40
10 cores	-	12 0	-	-	-	170	5 a	2	20
10  cores/4  cm	4	12 a	5 a 1 o	2	23	24 ab	5 a 4 o	2	21 15
10  cores/4 cm	4	15 a 12 -	4 a		28 22	20 a	4 a 7 -	<u>ک</u>	15
20 cores	4	15 a	4 a	<u>ل</u>	33	23 ab	/ a 25	4	51
Frame	4	16 a	2 a	1	9	39 ab	25 a	13	65
Layer mean		14	3	2	26	26	10	5	35

the sampling methods in any of the soil layers, except for the10-core/4-cm method, which was significantly higher than the directed method in the 0- to 15-cm soil layer.

<sup>z</sup> Hog manure was injected between 12 and 15 cm deep.

<sup>y</sup> Values within each column per soil layer followed by same letter are not significantly different at P < 0.05.

<sup>x</sup> Directed cores: one within-band and eight between-band cores. Control plots were not sampled (ns).

Standard deviations increased with increased STP levels following manure application (Table 3). Daniels et al. (2001) also reported higher standard deviations in STP data after manure application. Since the hog manure was applied for the first time at the Lethbridge Manure site, the average increase in standard deviations compared to the control plots was similar to the

average increase in mean STP levels (15 mg kg<sup>-1</sup> in the 0- to 2.5-cm layer, 11 mg kg<sup>-1</sup> in the 0- to 5-cm layer, and 7 mg kg<sup>-1</sup> in the 0- to 15-cm layer). Standard deviations in the 0- to 2.5-cm and 0- to 5-cm layers were 1.7 to 1.9 times higher than in the 0- to 15-cm layer. The increased variability in these two layers suggest that there were difficulties in applying hog manure at the targeted injection depth. The nutrient content of manure can be variable (Dou et al. 2001), and this may have had some influence. The higher variability in the 0- to 2.5-cm layer indicates that the measure of STP in the 0- to 15-cm layer may be more precise than in the two shallower layers, although STP levels in the 0- to 15-cm layer were lower than in the other two layers.

The application of 53 kg ha<sup>-1</sup> of phosphorus injected as hog manure resulted in highly variable site conditions where the coefficient of variation averaged between 43% in the 0- to 2.5-cm layer to 35% in the 0- to 15-cm layer, and was as high as 70% for the 5-core method in the 0- to 2.5-cm layer (Table 3). The CV calculated from the mean STP and standard deviation for each soil layer were similar among the layers. In these conditions, there were no significant differences among the standard deviations of the different methods for each soil layer.

**Lethbridge fertilizer site.** The control samples had low levels of STP that were typical of nonmanured soil and STP levels decreased with depth of soil layer (Table 4). Soil-test phosphorus and standard deviations of the control samples were not significantly different among the sampling methods for any of the soil layers.

Levels of STP in the fertilized plots were similar to the control plots in the 0- to 2.5-cm layer and were slightly higher than the control plots in the 0- to 5-cm layer (Table 4). Mean STP in the 0- to 15-cm layer for the fertilized treatments averaged 42 mg kg<sup>-1</sup> higher than the control treatment across all sampling methods, and this was attributed to the fertilizer banded at 9 cm below the surface. Janssen et al. (1998) also reported increased STP of 42 mg kg<sup>-1</sup> at banded depths between 8 and 16 cm after 4 yr of banding fertilizer phosphorus, although amounts applied were not specified. There were no significant differences among STP means and standard deviations for the methods tested in each soil layer.

If it is assumed that all of the elemental phosphorus applied at a rate of 176 kg ha<sup>-1</sup> was evenly distributed throughout the top 15-cm layer of soil, all remained in extractable form, and the soil bulk density was 1.3 Mg m<sup>-3</sup>, the STP would have increased by about 90 mg kg<sup>-1</sup>. Considering the 6-mo time interval between application and sampling, the actual observed increase of about half this amount is probably realistic. This value is within the range of banded phosphorus fertilizer concentrations of 0 to 80 mg kg<sup>-1</sup> measured by Westfall et al. (1991).

Standard deviations for the 0- to 2.5-cm soil layer in the fertilized plots were similar to those measured in the control plots, reflecting the precision of the knife-chisel implement in applying the fertilizer at deeper depths. The mean standard deviations in the fertilizer plots for the 0- to 5-cm and the 0- to 15-cm layers were greater than the control plots. The standard deviation of the 0- to 15-cm layer was about five times greater than the mean for the control treatment, reflecting increased STP variability at the depth where the fertilizer was targeted. These results contrast with those from the Lethbridge Manure site, where there was nearly a five-time increase in standard deviation in the 0- to 2.5-cm layer, but only a three-time increase in the targeted injection zone. The average CV in the fertilized plots was only 9% in the 0- to 2.5-cm layer, but

Control					Pho	sphorus	fertilized	1	
Sampling		STP mean	SD	SE	CV	STP mean	SD	SE	CV
method	n	(m	g kg <sup>-1</sup> ) -		(%)	(n	$\log kg^{-1}$ )		(%)
			0- i	to 2.5-ci	n layer				
5 cores	4	32 a <sup>y</sup>	12 a	6	39	38 a	1 a	1	3
Directed cores <sup>x</sup>	4	ns	-	-	-	37 a	5 a	3	14
10 cores	4	31 a	6 a	3	18	36 a	4 a	2	12
10 cores/4 cm	4	32 a	4 a	2	13	38 a	4 a	2	11
20 cores	4	34 a	4 a	2	12	37 a	3 a	2	9
Frame	4	37 a	2 a	1	6	37 a	2 a	1	6
Layer mean		33	6	3	18	37	3	2	9
			0-	to 5-cm	ı layer				
5 cores	4	31 a	10 a	5	32	41 a	13 a	6	31
Directed cores	4	ns	-	-	-	45 a	20 a	10	44
10 cores	4	27 a	5 a	2	17	41 a	13 a	7	33
10 cores/4 cm	4	29 a	4 a	2	14	34 a	5 a	2	14
20 cores	4	30 a	5 a	3	17	40 a	14 a	7	34
Frame	4	36 a	8 a	4	21	34 a	3 a	1	8
Layer mean		30	6	3	20	39	11	6	27
			0-	to 15-cn	n laver				
5 cores	4	20 a	7 a	4	37	79 a	26 a	13	33
Directed cores	4	ns	-	-	-	50 a	18 a	9	37
10 cores	4	16 a	3 a	2	19	62 a	19 a	9	30
10 cores/4 cm	4	20 a	3 a	1	13	63 a	42 a	21	66
20 cores	4	18 a	2 a	1	12	55 a	17 a	8	30
Frame	4	23 a	7 a	4	31	54 a	11 a	6	21
Layer mean		19	4	2	22	61	22	11	36

**Table 4.** Mean soil-test phosphorus (STP), standard deviation (SD), standard error (SE), and coefficient of variation (CV) for different soil sampling methods at the Lethbridge Fertilizer site<sup>z</sup>.

<sup>z</sup> Inorganic fertilizer was banded 9 cm deep.

<sup>y</sup> Values within each column per soil layer followed by same letter are not significantly different at P < 0.05.

<sup>x</sup> Directed cores: one within-band and six between-band cores. Control plots were not sampled (ns).

increased to 27% in the 0- to 5-cm layer and to 36% in the 0- to 15-cm layer where the fertilizer was targeted. The increased variability in the deeper banded layer relative to the shallower layers at the fertilized site suggests, in this case, that STP in the shallower layers was a more precise measure of STP than STP in the deeper layer, although a higher concentration of STP was measured in the 0- to 15-cm layer than in the other two layers.

The variability of the frame-excavation method tended to be the lowest in each soil layer, but there were no significant differences between the standard deviations of the frame excavation method and any of the other methods. However, when the Bonferroni adjustment was not applied, a P value of 0.03 indicated a significantly lower standard deviation for the frame-excavation method relative to the 10-core/4-cm method in the 0- to 15-cm layer.

These results suggest that in conditions where high levels of phosphorus were precisely applied in bands, the variability of the frame-excavation method was similar to the variability of other sampling methods used in this study, with a slight tendency toward reduced variability.

**Calgary biosolids site.** Preliminary analysis of the data from six plots measured at the Calgary Biosolids site showed that two plots (one without biosolids and one with biosolids) had very low phosphorus levels, while the remaining four plots had very high phosphorus levels. As a result, data from the two plots with very low levels were omitted from the final analysis (Appendix Table A2.4). As the following results are based on only two replicates, they should be interpreted with caution.

Levels of STP were very high in the control plots, reflecting a previous history of phosphorus application (Table 5). There were no significant differences in mean STP values among the sampling methods for the control plots in any of the soil layers. The standard deviations were not significantly different for the 0- to 2.5-cm and 0- to 5-cm layers in the control plots, but the 10- core/4-cm method in the 0- to 15-cm soil layer had a significantly lower standard deviation than the other methods measured in the control plots, indicating some background variability in this layer.

The application of biosolids increased the mean STP levels by about 85 mg kg<sup>-1</sup> in the 0- to 2.5-cm and 0- to 5-cm layers compared to the control plots, reflecting variations in actual injection depths (Table 5). There was a large increase of 130 mg kg<sup>-1</sup> in the deepest layer that included the 10-cm injection depth where the biosolids were targeted. These increases were measured 2 wk after application and probably represent only a fraction of the phosphorus that could become available in the following year. Significantly lower STP levels were measured with the 5-core method relative to other methods in the 0- to 2.5-cm layer, with the 5-core and frame methods relative to the 20-core method in the 0- to 15-cm layer.

Variability also increased with application of biosolids. When compared to the control plots, the average standard deviation among the sampling methods for the biosolids plots increased by 29 mg kg<sup>-1</sup> in the 0- to 2.5-cm layer, possibly due to the increased roughness of the soil surface, 17 mg kg<sup>-1</sup> in the 0- to 5-cm layer, and 12 mg kg<sup>-1</sup> in the 0- to 15-cm layer. Although the variability increased with the application of biosolids, the average CV for each layer was quite low (21% for the 0- to 2.5-cm layer, 14% for the 0- to 5-cm layer, and 11% for the 0- to 15-cm layer). As mean STP levels were similar among the soil layers, the decreased variability in the deepest layer relative to the 0- to 2.5-cm layer indicates that the 0- to 15-cm layer would be the most accurate measure of STP.

Control						Biosolids			
Sampling		STP mean	SD	SE	CV	STP mean	SD	SE	CV
method	n	(m	ıg kg <sup>-1</sup> ) -		(%)		$(mg kg^{-1})$		(%)
			(	0- to 2.5-	cm layer				
5 cores	2	116 a <sup>y</sup>	11 a	8	10	179 b	20 abc	14	11
Directed cores <sup>x</sup>	2	ns	-	-	-	260 a	12 bc	9	5
10 cores	2	139 a	23 a	16	16	214 a	66 ab	47	31
10 cores/4 cm	2	140 a	5 a	4	4	195 a	124 a	88	64
20 cores	2	138 a	19 a	14	14	268 a	4 c	3	2
Frame	2	150 a	7 a	5	5	212 a	24 abc	17	11
Layer mean		137	13	9	10	221	42	29	21
				0- to 5-c	m layer				
5 cores	2	128 a	13 a	9	10	181 c	5 cd	4	3
Directed cores	2	ns	-	-	-	287 a	1 d	1	0
10 cores	2	154 a	15 a	10	9	172 abc	44 ab	31	26
10 cores/4 cm	2	140 a	5 a	3	3	189 ab	79 a	56	42
20 cores	2	122 a	8 a	6	6	313 a	24 abc	17	8
Frame	2	151 a	11 a	8	7	200 bc	8 bc	6	4
Layer mean		139	10	7	7	224	27	19	14
				0- to 15-a	cm layer				
5 cores	2	101 a	12 a	9	12	234 ab	18 a	13	8
Directed cores	2	ns	-	-	-	306 ab	34 a	24	11
10 cores	2	145 a	29 a	20	20	250 ab	42 a	30	17
10 cores/4 cm	2	118 a	1 b	1	1	210 ab	44 a	31	21
20 cores	2	107 a	12 a	9	11	334 a	13 a	9	4
Frame	2	146 a	17 a	12	12	185 b	7 a	5	4
Layer mean		123	14	10	11	253	26	19	11

**Table 5.** Mean soil-test phosphorus (STP), standard deviation (SD), standard error (SE), and coefficient of variation (CV) for different soil sampling methods at the Calgary Biosolids site<sup>z</sup>.

<sup>z</sup> Biosolids were injected 10 cm deep.

<sup>y</sup> Values within each column per soil layer followed by same letter are not significantly different at P < 0.05.

<sup>x</sup> Directed cores: one within-band and six between-band cores. Control plots were not sampled (ns).

The standard deviation of the 10-core/4-cm method was significantly greater than some of the other methods in the 0- to 2.5-cm and 0- to 5-cm layers, notably the frame method in the 0- to 5-cm layer and the 20-core method in the 0- to 2.5-cm layer. Though the differences were not significant in the 0- to 15-cm layer, the standard deviation of the frame-excavation method was the lowest.

The standard deviations measured by the frame-excavation method were among the lowest measured in the 0- to 5-cm and 0- to 15-cm layers that were influenced by the injected biosolids, representing about 30% of the mean standard deviation for each of these soil layers, similar to the Lethbridge Fertilizer site. The other methods accounted for up to 1.6 times the mean standard deviation in the 0- to 15-cm layer where the biosolids were most heavily applied. Since these results were based on only two replicates, further investigation is required to determine if there are differences in means and standard deviations among methods for soil with very high STP.

**Timing.** The time required to take soil samples using the various sampling methods was recorded at the Lethbridge Manure, Lethbridge Fertilizer, and Calgary Biosolids sites. More time was required to sample a greater number of cores, although the sampling times among sites and among replicates at each site varied due to different soil conditions (Table 6). The time required for the frame-excavation method was similar to the directed-core and 10-core composite sampling methods, and the 5-core method required the least amount of time. The 20-core method required about twice as much time as the frame-excavation method.

<b>Table 6.</b> Time required for the different methods of soil sampling.								
	Lethbridge	Lethbridge	Calgary					
	Manure site	Fertilizer site	<b>Biosolids site</b>					
Sampling method		(min)						
5 cores	11	9	10					
Directed cores <sup>z</sup>	15	11	15					
10 cores	15	13	20					
20 cores	29	23	30					
Frame	15	13	15					

<sup>z</sup> One within-band to eight between-bands cores at the manure site (30-cm spacing), and one within-band core to six between-band cores at the fertilizer and biosolids sites (23-cm spacing).

Additional discussion. There were no significant differences among the mean STP values of samples with the frame-excavation method and the other sampling methods at the Wainwright Manure and Lethbridge Fertilizer sites. Ashworth et al. (1994) also reported no difference in mean STP values among samples taken in 15-cm deep cuts across bands of fertilizer with a modified chainsaw and composite samples of 12, 5-cm cores. However, Mahler (1990) reported higher mean STP levels using a systematic method of sampling eight cores equally spaced across a fertilizer band (similar in principle to the frame-excavation method) compared with random sampling.

Significant differences among mean STP values were observed at the more variable Lethbridge Manure and Calgary Biosolids sites, although the results were not consistent. For example, significantly lower levels of STP were measured using the directed-core method compared with the 10-core/4-cm method in the 0- to 15-cm layer at the Lethbridge Manure site, while in the 0- to 5-cm layer at the Calgary Biosolids site, STP measured using the directed-core method was not different from the 10-core/4-cm method, but was significantly higher than the 5core and the frame-excavation methods.

There were no differences among the standard deviations of the frame-excavation method compared with the other sampling methods at any of the sites, except in the 0- to 5-cm soil layer at the Calgary Biosolids site. The standard deviations for the frame-excavation samples were generally the lowest measured in the application zones at the Lethbridge Fertilizer and Calgary Biosolids sites where high levels of phosphorus were applied. When the Bonferroni adjustment was not used at the Lethbridge Fertilizer site, standard deviations with the frame-excavation

method were significantly lower than the other sampling methods in the soil layer that included the zone where phosphorus had been applied. Ashworth et al. (1994) measured significantly lower coefficients of variation in slot-sampled banded soils than in 12-core composite samples taken in the 0- to 15-cm layer at low levels of applied phosphorus fertilizer, although Fisher's LSD test at P < 0.10 had been used.

Researchers have measured wide variations in STP. Cameron et al. (1971) reported coefficients of variation of 40 to 60% for STP measured using core samples in 0- to 15-cm depths in three representative locations in Alberta. In a study of soil sampling techniques for band-fertilized, no-till fields, James and Hurst (1995) concluded that the number of randomly sampled soil cores needed to represent band-fertilized fields is prohibitive. Westfall et al. (1991) also concluded that it is not possible to take enough cores to obtain an accurate measure of STP levels when high rates of phosphorus fertilizer are applied in wide (76 cm) bands. Therefore, it is possible that the two to four replicates used at our small-plot sites were too few to obtain an accurate measure of STP.

Stein's two-stage test (Stein 1945) was used to estimate the number of sampling points, or replicates, required to accurately measure STP levels for each of the soil layers where STP increases were largest, using the different sampling methods. The criteria suggested by Cameron et al. (1971) as adequate and realistic for soil testing purposes in Alberta is that in 8 out of 10 sampling occasions, or at an 80% probability level, results should be within 10% of the mean. In this study, the layer mean was considered to be a better representation of a "true" mean than the mean of each sampling method. At the Wainwright Manure site, where STP in the applied bands was not significantly greater than STP between bands, the 5-core method would require the most number of replicates (22) and the frame-excavation method would require the least number of replicates (2) (Table 7). At the Lethbridge Manure site, where moderate levels of phosphorus (53  $mg kg^{-1}$ ) had been applied at depths shallower than the target depths, increases in STP were similar to increases in standard deviations, which resulted in high CVs. In these variable conditions, the 5-core method again required the largest number of replicates (187) and the directed-core method required the least number of replicates (12). Where high levels of phosphorus (> 176 mg kg<sup>-1</sup>) had been applied, the frame-excavation method required the least number of replicates, only 9 compared with 125 for the 10-core/4-cm method at the Lethbridge Fertilizer site, and only 1 compared with 29 for the 10-core/4-cm method at the Calgary Biosolids site.

The variability of STP among the soil layers was also considered in the comparison of soil sampling methods, although results were difficult to evaluate. The lower CV in the 0- to 15-cm layer at the Lethbridge Manure and Calgary Biosolids sites compared with the 0- to 2.5-cm layers suggest that the deepest layer may give a more accurate measure, although the opposite result was measured at the Lethbridge Fertilizer site.

While these results from the small-plot sites do not indicate a clear answer to the question of which sampling method, or sampling depth is best for reducing the variability of STP in soils where sources of phosphorus have been applied in bands, the frame-excavation method offers the advantages of a lower number of samples for three of four study sites and similar sampling times compared to the 10-core composite sampling methods, particularly in conditions where levels of

applied phosphorus are high (> 176 mg kg<sup>-1</sup>). This is likely since the larger volumes of soil obtained with the frame-excavation method should result in a more proportionate distribution of within and between phosphorus bands. However, it is important that the excavated sample is well mixed in the field and again in the laboratory after drying and grinding so that subsamples used for analysis are representative of the proportion of phosphorus band to the soil. Since the frame-excavation method requires knowledge of the band location, direction, and spacing, its effective use may be limited when this information is not known.

**Table 7.** Results of Stein's two-stage approach for estimation of the number of replicates required to estimate the layer mean within 10%, 8 times out of 10, in the layer of greatest STP increase after phosphorus application.

1	1 11			
	Wainwright	Lethbridge	Lethbridge	Calgary
	Manure site	Manure site	Fertilizer site	<b>Biosolids site</b>
	0 to 15 cm	0 to 5 cm	0 to 15 cm	0 to 15 cm
Treatment	(df = 2)	(df = 3)	(df = 3)	(df = 1)
5 cores	22	187	47	5
Directed <sup>z</sup>	-	12	24	17
10 cores	16	30	25	26
10 cores/4 cm	-	15	125	29
20 cores	4	55	20	3
Frame	2	30	9	1

<sup>z</sup> One within-band to eight between-bands cores at manure site (30-cm spacing), and one within-band core to six between-band cores at fertilizer and biosolids sites (23-cm spacing).

### **Microwatershed Study Sites**

Mean STP values were about 28% lower when measured by the 10-core agronomic method compared to the frame-excavation environmental method when all sites were combined (Table 8). This was also true when the five non-manured and two manured sites were separated (Fig. 5). Even though slopes of the relationships for the non-manured sites (Fig. 5b) and the manured sites (Fig. 5c) were nearly 1, the lines have positive y-axis intercepts. If the two methods were able to reproduce the same results, the relationships should fall on a one-to-one line and pass through the origin. The concordance correlation coefficient measures how far the best-fit line deviates from the one-to-one line (Lin 1989). Results of the concordance correlation analysis, along with the Pearson correlation and the concordance correlation coefficients are not close, particularly for the non-manured and manured sites. The concordance correlation coefficients are quite different from 1, particularly when the data were separated into non-manured and manured sites. In all three data sets, the Pearson correlation coefficient was outside the lower and upper 95% conference intervals. The location shift values were not near zero (1:1 line is 0) and the scale shift values were not near one (1:1 line is 1).

analysis methods in the Microwatershed Study.									
		Standard							
		mean	Min.	Max.	deviation				
Sites <sup>z</sup>	Method	$(mg kg^{-1})$	$(mg kg^{-1})$	$(mg kg^{-1})$	$(mg kg^{-1})$	n	Core:frame		
All	core	81	7	367	106	50	0.72		
All	frame	112	10	456	144	50	0.72		
			_		_				
Non-manured	core	17	7	40	7	34	0.71		
Non-manured	frame	24	10	55	8	34	0.71		
		• 1 0		o - =	- <b>-</b>				
Manured	core	219	55	367	85	16	0.73		
Manured	frame	298	84	456	115	16	0.75		

**Table 8.** Soil-test phosphorus (STP) data from the frame-excavation and 10-core sampling and analysis methods in the Microwatershed Study.

 $^{\mathbf{z}}$  All included data from all seven watershed sites, non-manured included data from five sites, and manured included data from two sites.

Results obtained by the two sampling methods in this study were unexpected since STP measured by the core and the frame-excavation methods, where all subsamples were removed by weight for laboratory analysis, were not significantly different for most of the small-plot sites discussed previously. As well, Ashworth et al. (1994) found no difference among the mean STP values of samples collected using a slot-sampling technique and a composite of 12 cores. Subsamples used by the lab for STP analysis were removed by weight (4-g environmental method) from the frame-excavation samples and by volume (4-cm<sup>3</sup> agronomic method) from the 10-core method samples. An average bulk density (1.18 Mg m<sup>-3</sup>), which was previously determined based on a range of dried and ground mineral soil samples, was used to calculate the 10-core sample results on a weight basis (mg kg<sup>-1</sup>). However, variation in the actual bulk density of the dried and ground soil samples may have resulted in some error. Therefore, there are two factors that may have caused differences between the two methods: field sampling (frame versus core) and analytical procedure (weighed subsamples versus volume subsamples). A more controlled study would be required to better determine the influence of these factors on the variation between the two methods (frame and subsample by weight versus 10-core and subsample by volume) used in the Microwatershed Study.

#### CONCLUSIONS

Comparison of the frame-excavation method with several methods of core sampling showed that all of the sampling methods provided similar STP values at the Wainwright Manure and the Lethbridge Fertilizer sites. At the Calgary Biosolids site, where only two replicates were used, significantly higher STP levels were measured using the 20-core method relative to the 5-core or the frame-excavation methods in all soil layers. Standard deviations did not differ significantly among the sampling methods at any of the sites, regardless of soil layer, except in the 0- to 2.5-



**Fig. 5.** Comparison of soil-test phosphorus (STP) measured using the frame-excavation and 10core composite methods for (a) all sample sites, (b) non-manured sites, and (c) manured sites from the Microwatershed Study. The dashed lines are the one-to-one lines passing through the origin.

cm and 0- to 5-cm layers at the Calgary Biosolids site. However, where high levels of phosphorus (>176 mg kg<sup>-1</sup>) were applied in targeted layers, the standard deviations for the frame-excavation samples tended to be the lowest measured. The time required to use the frame-excavation method was comparable to the 10-core composite sample method. The number of samples needed to measure STP within 10% of the mean in the layer where STP increases were greatest was reduced with the frame-excavation method for three of the four study sites. We conclude that a minimum of five core composite samples per sampling point would be adequate

Doromotor	All sites $(n = 50)$	Non-manured sites $(n - 34)$	Manured sites $(n - 16)$
Faranieter	(II - J0)	(11 - 34)	(II = 10)
Pearson correlation (r)	0.963	0.833	0.814
Concordance correlation (CC)	0.895	0.593	0.596
Lower 95% confidence interval	0.846	0.413	0.302
Upper 95% confidence interval	0.929	0.728	0.786
Location shift (1:1 line is 0)	0.244	0.887	0.802
Scale shift (1:1 line is 1)	0.738	0.858	0.741

**Table 9.** Pearson correlation and concordance correlation coefficient analysis results of the soiltest phosphorus data for the frame-excavation and 10-core sampling methods used in the Microwatershed Study.

for determination of STP in soils with low levels of banded phosphorus. For soils with higher phosphorus content, the frame-type method of sampling was comparable to other coring methods for determination of STP, reduced requirements for sampling time, and reduced requirements for the number of replicates needed to measure an accurate mean.

The STP in the 0- to 15-cm soil layer measured using the 10-core (subsampled by volume) method was well correlated with STP measured using the frame-excavation (subsampled by weight) method over a range of site conditions ( $r^2 = 0.93$ ) in the Microwatershed study. On average, the 10-core method STP values were about 28% less than STP values using the frame-excavation method. The same difference in STP values between the two methods was observed when the non-manured and manured site data were separated. The study design for the Microwatershed Study did not allow for separation of the sampling method (frame and core) and analytical method effects (weight versus volume subsamples for analysis), or their interactions, and additional work is needed to clarify these effects. However, we suggest that a more precise measure of STP would be obtained by weighing subsamples for laboratory analysis because of the uncertainty of the bulk density of subsamples removed by volume.

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### Appendix 1. Field study sites.



Treatment M: 90,000 L ha<sup>-1</sup> hog manure Implement: Knife (shank with chisel blade) Injection band spacing: 30 cm Injection depth: 10 to 15 cm Five shanks: Five passes to give a plot width of about 7.5 m

Treatment C: Control Treatment M: Municipal sludge Implement: Terra-Gator applicator Band spacing: 23 cm Band depth: 10 cm Machine width: About 1.5 m, five passes per plot

**Fig. A1.1.** Layout of soil sampling locations at (a) Wainwright Manure and (b) Calgary Biosolids sites.



Treatment C: Control Treatment H: 90,000 L ha<sup>-1</sup> hog manure Implement: Knife (shank with chisel blade) Injection band spacing: 30 cm Injection depth: 12 to 15 cm Five shanks: Five passes to give a plot width of about 7.5 m

Treatment C: Control Treatment F: 176 kg ha<sup>-1</sup> phosphorus Implement: Disc-opener with packers Band spacing: 23 cm Band depth: 9 cm Machine width: 1.83 m with four passes per plot

**Fig. A1.2.** Layout of sampling locations at (a) Lethbridge Manure and (b) Lethbridge Fertilizer sites.



Fig. A1.3. Location of the Microwatershed Study sites in Alberta.

# Appendix 2. Soil-test phosphorus data.

	Rep 1 STP	Rep 2 STP	Rep 3 STP	
Sampling method		(mg kg <sup>-1</sup> )		
	0- to 2.5-	cm layer		
5 cores	37	23	32	
10 cores	42	30	33	
20 cores	21	22	26	
Frame	14	33	28	
	0- to 5-c	m layer		
5 cores	36	24	29	
10 cores	40	29	28	
20 cores	20	23	25	
Frame	19	32	28	
	0- to 15-0	cm layer		
5 cores	31	19	24	
10 cores	32	22	29	
20 cores	17	21	21	
Frame	21	25	21	
Within band	21	44	27	
Between band	29	16	22	

# Table A2.1. Soil-test phosphorus (STP) results from the Wainwright Manure site.

<b>Table A2.2.</b> Soil-test phosphorus (STP) results from the Lethbridge Manure site.									
Control plots STP					Manured	plots STP			
Sampling	Rep 1	Rep 2	Rep 3	Rep 4	Rep 1	Rep 2	Rep 3	Rep 4	
method		(mg	kg <sup>-1</sup> )			(mg	kg <sup>-1</sup> )		
			0- ta	o 2.5-cm la	yer				
5 cores	31	23	32	21	54	33	26	113	
10 cores	24	29	32	18	50	35	42	40	
10 core/4 cm	25	29	30	21	39	39	11	30	
20 cores	24	26	29	22	61	63	25	28	
Directed cores	$ns^{z}$	ns	ns	ns	47	26	29	37	
Frame	25	30	29	24	84	38	38	29	
			0- t	to 5-cm lay	er				
5 cores	25	18	32	17	67	28	23	97	
10 cores	21	25	28	15	47	30	46	63	
10 core/4 cm	23	24	33	17	44	35	21	37	
20 cores	20	22	33	19	69	51	29	30	
Directed cores	ns	ns	ns	ns	46	24	30	37	
Frame	23	24	29	22	62	38	34	33	
0- to 15-cm layer									
5 cores	14	9	18	9	34	16	15	40	
10 cores	14	13	15	8	26	17	24	29	
10 core/4 cm	14	18	17	9	33	24	29	24	
20 cores	11	12	18	9	32	23	20	16	
Directed cores	ns	ns	ns	ns	23	12	16	18	
Frame	16	14	18	16	77	29	24	25	

Table A2 2 Soil-test ph (STP) ilte fr Lethbridg Ma th ٦ĥ

<sup>z</sup> ns = not sampled.

<b>Table A2.3.</b> Soll-test phosphorus (STP) results from the Lethbridge Fertilizer site.								
		Control p	plots STP	•	Fertilized plots STP			2
Sampling	Rep 1	Rep 2	Rep 3	Rep 4	Rep 1	Rep 2	Rep 3	Rep 4
method		(mg	kg <sup>-1</sup> )			(n	ng kg <sup>-1</sup> )	
			0- to	2.5-cm lay	ver			
5 cores	24	43	18	41	39	37	37	39
10 cores	26	36	27	36	41	31	36	35
10 core/4 cm	27	35	30	36	41	32	38	41
20 cores	28	34	34	38	38	32	36	40
Directed cores	ns <sup>z</sup>	ns	ns	ns	42	30	40	37
Frame	35	37	40	36	40	35	36	37
			0 +	o 5 am law				
5	20	40	0- li 25	29 <i>3-cm iay</i> e	er c0	25	22	26
	20	40	25	30 21	60	23 27	33 24	20 22
10  cores	21	31 22	20	31 22	01	37 29	54 24	23 27
$10 \operatorname{core}/4 \operatorname{cm}$	24	32 20	28	33 25	38 (1)	28	54 24	37 29
20 cores	23	30	33	35	60	28	34	38
Directed cores	ns	ns	ns	ns	74	32	36	37
Frame	31	33	47	34	38	32	34	34
			0- to	15-cm lay	er			
5 cores	13	25	14	27	99	95	78	43
10 cores	13	20	15	17	80	37	70	61
10 core/4 cm	18	22	17	22	49	40	125	39
20 cores	15	18	18	19	59	53	75	34
Directed cores	ns	ns	ns	ns	38	70	31	59
Frame	18	22	33	19	62	60	38	58

Table A2.3. Soil-test phosphorus (STP) results from the Lethbridge Fertilizer sit

<sup>z</sup> ns = not sampled.

Table A2.4. Soil-te	est phosphorus (	STP) results from the	Calgary Biosolids site	2.
	Control p	olots STP	Biosolids	plots STP
Sampling	Rep 1	Rep 2	Rep 1	Rep 2
method -	(mg	kg <sup>-1</sup> )	(mg	kg <sup>-1</sup> )
		0- to 2.5-cm laye	r	
5 cores	124	108	165	193
10 cores	123	155	167	260
10 core/4 cm	136	143	107	282
20 cores	124	151	265	271
Directed cores	ns <sup>z</sup>	ns	268	251
Frame	155	145	229	195
		0- to 5-cm layer		
05 cores	137	119	178	185
10 cores	144	165	141	203
10 core/4 cm	137	143	133	244
20 cores	116	127	296	330
Directed cores	ns	ns	288	287
Frame	158	143	195	206
		0- to 15-cm layer	r	
5 cores	92	109	222	247
10 cores	125	165	220	279
10 core/4 cm	118	117	179	241
20 cores	98	116	324	343
Directed cores	ns	ns	330	282
Frame	133	158	180	190

<sup> $\mathbf{z}$ </sup> ns = not sampled.

		Landform	Frame <sup>y</sup>	Core				
Site <sup>z</sup>	Sample	position	(n	$\log kg^{-1}$ )				
Fall 2003								
CFT	12	depression	24	19				
CFT	14	mid	15	17				
CFT	18	mid	27	25				
CFT	34	mid	55	40				
CFT	42	upper	15	8				
CFT	45	mid	48	30				
GPC	27	lower	33	27				
GPC	33	upper	24	18				
LLB	19	mid	84	116				
LLB	31	mid	447	295				
LLB	32	mid	259	172				
LLB	33	mid	364	169				
LLB	35	lower	202	124				
LLB	42	lower	225	204				
LLB	17	upper	177	55				
PON	11	mid	413	310				
PON	19	lower	295	205				
REN	13	mid	20	18				
REN	19	upper	25	16				
THC	15	lower	23	18				
THC	23	lower	17	9				
WAB	26	lower	29	28				
WAB	17	mid	27	67 <sup>x</sup>				

**Table A2.5.** Soil-test phosphorus (STP) results for the 0- to 15-cm soil layer using the frameexcavation and 10-core sampling methods at seven Microwatershed Study sites in the fall of 2003.

<sup>z</sup> CFT = Crowfoot Creek, GPC = Grande Prairie Creek, LLB = Lower Little Bow, PON = Ponoka, REN = Renwick Creek, THC = Three Hills Creek, WAB = Wabash Creek.

<sup>y</sup> Calculated using the weighted sum of STP values for the 0- to 2.5-cm, 2.5- to 5-cm, and 5- to 15-cm soil layers. <sup>x</sup> Outlier removed from results.

		Landform	Frame <sup>y</sup>	Core				
Site <sup>z</sup>	Sample	position -	(mg kg <sup>-1</sup> )					
Fall 2004								
CFT	5	depression	28	17				
CFT	9	mid	28	17				
CFT	12	mid	19	14				
CFT	14	mid	16	12				
CFT	17	upper	24	19				
CFT	18	mid	30	24				
GPC	9	lower	10	10				
GPC	24	upper	27	24				
GPC	27	mid	27	27				
LLB	31	mid	456	273				
LLB	32	mid	168	146				
LLB	33	mid	421	293				
PON	2	lower	283	298				
PON	3	lower	448	367				
PON	10	upper	261	235				
PON	11	mid	274	247				
REN	4	lower	21	9				
REN	13	mid	16	7				
REN	14	upper	22	9				
REN	25	lower	27	10				
THC	3	lower	18	14				
THC	6	lower	26	17				
THC	15	mid	27	15				
THC	16	upper	17	10				
WAB	18	upper	20	14				
WAB	25	lower	19	13				
WAB	5	lower	23	15				
WAB	17	mid	23	11				

**Table A2.6.** Soil-test phosphorus (STP) results for the 0- to 15-cm soil layer using the frameexcavation and 10-core sampling methods at seven Microwatershed Study sites in the fall of 2004.

<sup>z</sup> CFT = Crowfoot Creek, GPC = Grande Prairie Creek, LLB = Lower Little Bow, PON = Ponoka, REN = Renwick Creek, THC = Three Hills Creek, WAB = Wabash Creek.

<sup>y</sup> Calculated using the weighted sum of STP values for the 0- to 2.5-cm, 2.5- to 5-cm, and 5- to 15-cm soil layers.