Effect of Methodology in Estimating and Interpreting Water-Extractable Phosphorus in Animal Manures

P. A. Vadas* and P. J. A. Kleinman

ABSTRACT

Manure water-extractable phosphorus (WEP) data are used in indices and models to assess P transport in runoff. Methods to measure WEP vary widely, often without understanding the effect on how much P is extracted. We conducted water extractions on five dairy, swine, and poultry manures to assess single and sequential extractions, drying manures, solution to solid ($cm^3\ g^{-1}$) extraction ratios, and P determination method. We found little difference in WEP of single or sequential extractions. Increasing extraction ratio from 10:1 to 250:1 resulted in more WEP recovered, but in a diminishing fashion so that ratios of 200:1 and 250:1 were not significantly different. Patterns of increased WEP with extraction ratio varied with manure type, presence of bedding material, and drying treatment. Fresh and air-dried manures had similar patterns, but differed substantially from oven-dried (90°C) manures. The differential effect of oven-drying on WEP was greatest for dairy and poultry manure, and less for swine manure. We analyzed water extracts colorimetrically before and after digestion, to examine the potential effect of P determination by inductively coupled plasma (ICP) spectroscopy. Digested extracts always contained more P. For manures with bedding, drying decreased the difference in P measured before and after digestion. The opposite was true for manures without bedding. Results highlight the influence of methodology on manure WEP measurement and caution needed when comparing data across studies using different WEP methods. Overall, our results point to a need for a standard manure water extraction method.

Nonpoint-source pollution of fresh surface waters by P is a water quality concern because it contributes to accelerated eutrophication and subsequently limited water use for drinking, recreation, and industry (Carpenter et al., 1998). Transfer of P from agricultural soils in surface runoff is a dominant transport mechanism in many situations (Sharpley et al., 2003). Soil and plant material contribute P to runoff, but their effect can be overwhelmed by P release from recently surface-applied manures (Eghball and Gilley, 1999; Kleinman and Sharpley, 2003; Moore et al., 2000). The quantity of water-extractable phosphorus (WEP) in surface-applied manures is an important factor controlling dissolved P concentrations in runoff (DeLaune et al., 2004a; Haggard et al., 2005; Kleinman et al., 2002b).

Most studies have demonstrated this effect through regressions of WEP in surface-applied manure, typically expressed as a concentration ($g\ kg^{-1}$) or mass ($kg\ ha^{-1}$), and concentrations ($mg\ L^{-1}$) of dissolved P in runoff (typically P in a runoff sample passing through a 0.45-μm filter) (Haggard et al., 2005; Vadas et al., 2004b). Therefore, WEP is seen as a key indicator of the potential for surface-applied manure to supply dissolved P to runoff (Kleinman et al., 2006), and manure WEP data are increasingly being used to guide management decisions. For example, the Arkansas P Index for pastures relies heavily on manure WEP surface-applied to fields to determine potential P transport in runoff (DeLaune et al., 2004b). Other P site assessment indices similarly propose to use manure WEP data, although to a lesser extent (Coale and Elliott, 2004). Manure WEP also plays a key role in improving the ability of fate-and-transport models to estimate P in runoff from surface-applied manures (Vadas et al., 2005).

Manure WEP is generally estimated by shaking a small, representative quantity of manure with water and measuring P released into the solution. The concept is simple, but many variations have been used, often without acknowledgment or thorough understanding of how a specific method may affect the ultimate estimate of WEP or interpretation of results. Methods vary mostly in whether or not manures are dried or fresh (see references in Table 1), the use of single or sequential extractions (Dou et al., 2000, 2002), the solution to solids extraction ratio and times of shaking used during extraction (Kleinman et al., 2002b), and how P in the extracts is analyzed (Wolf et al., 2005). Because these variables may affect the quantity of manure P extracted and thus deemed WEP, methodology is critical to applications such as the Arkansas Pasture P Index, which is based on a specific method of estimating manure WEP (Self-Davis and Moore, 2000). Variations to this method that change WEP estimates will be a source of error in P Index results.

Given the growing importance of WEP data in environmental interpretation of manure management, our objective in this study was to investigate the effects of variations in methodology on estimating WEP in animal manures. Key variables that we examined include the use of single or sequential extractions, method of drying manures before analysis, the solids to solution ratio used during extraction, and how P in extracts is analyzed.

MATERIALS AND METHODS

Manure Collection and Water-Extractable Phosphorus Analysis

We collected five manures for P analysis that represented a range of livestock species, dry matter, and bedding contents

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P.A. Vadas, USDA-ARS, U.S. Dairy Forage Research Center, 1925 Linden Drive West, Madison, WI 53706. P.J.A. Kleinman, USDA-ARS, Pasture Systems and Watershed Management Research Unit, Building 3702, Curtin Road, University Park, PA 16802-3702. Received 1 Sept. 2005. *Corresponding author (vadas@wisc.edu).

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677 S. Segoe Rd., Madison, WI 53711 USA

Abbreviations: ICP, phosphorus analysis by inductively coupled plasma spectroscopy; TP, total phosphorus in manures; WEP, manure water-extractable phosphorus; WEP_I, inorganic manure water-extractable phosphorus measured colorimetrically without digestion; WEP_T, total manure water-extractable phosphorus measured colorimetrically with digestion.

Table 1. Summary of literature review showing variations in methods to measure manure water-extractable phosphorus (WEP).

Source	Manure type	Manure condition	Water to manure ratio	Time of extraction	P analysis method	Filtration	Solution additives	Extract additives
			$cm^3\ g^{-1}$	min				
Penn et al. (2004)	turkey manure	fresh	10:1	60	ICP†	0.45 μm	–	–
Maguire et al. (2005b)	turkey litter	dried at 40°C	10:1; 200:1	60	ICP	0.45 μm	–	–
Maguire et al. (2003)	turkey litter	dried at 55°C	10:1	60	ICP	0.45 μm	–	–
Maguire et al. (2004)	turkey, poultry litter	dried at 40°C	200:1	60	ICP	0.45 μm	–	–
Toor et al. (2005)	turkey, poultry litter	dried at 55°C	10:1; 200:1	60	ICP	0.45 μm	–	–
McGrath et al. (2005)	poultry litter	fresh, dried at 60°C	10:1; 200:1	60	ICP	0.45 μm	–	–
Applegate et al. (2003)	poultry litter	lyophilized	10:1	60	Murphy and Riley (1962)	0.45 μm	–	–
Miles et al. (2003)	poultry litter	dried at 80°C	15:1	30	ICP	2V Whatman	–	–
Smith et al. (2004b)	poultry litter	fresh	10:1	60	Murphy and Riley (1962)	0.45 μm	–	HCl to pH 2
Vadas et al. (2004a)	poultry manure	fresh	10:1	60	Murphy and Riley (1962)	0.45 μm	–	–
Baxter et al. (2003)	swine slurry	lyophilized	100:1	1440	Murphy and Riley (1962)	centrifuged	chloroform	–
Smith et al. (2004a)	swine slurry	fresh	approximately 50:1	–	Murphy and Riley (1962)	0.45 μm	–	HCl to pH 2

† Inductively coupled plasma.

(Table 2). They included two dairy manures from lactating Friesian cows (*Bos taurus*) with and without bedding material, a swine slurry from finishing sows (*Sus scrofa domestica* L.) that had been washed into a holding tank and agitated before sampling, and two poultry litters from broiler chickens (*Gallus gallus domesticus* L.) in Texas and Pennsylvania that were a mixture of manure and sawdust–wood chip bedding material. We measured manure dry matter content gravimetrically after drying at 90°C for 48 h and total phosphorus (TP) by a modified semimicro-Kjeldahl procedure (Bremner, 1996). Swine slurry contained the most total P, followed by poultry and then dairy manure. Total P concentrations were in a range typically reported for such manures (Kleinman and Sharpley, 2003; Kleinman et al., 2002a, 2002b, 2005; Pote et al., 2001; Sharpley and Moyer, 2000).

We measured WEP in fresh manures as initially collected, after air-drying at room temperature in an exhaust hood, and after oven-drying at 90°C for 48 h. We chose 90°C because it represented a significant variation from air-drying and because such high temperatures have been used in other manure P studies (Ajiboye et al., 2004; Sistani et al., 2001) and are recommended for some routine manure analyses (Peters et al., 2006). We ground all dried manures with a mortar and pestle. Kleinman et al. (2002b) and Dou et al. (2000) observed that manure WEP increases with the shaking time of water extractions, that WEP will reach a maximum at 16 to 24 h of shaking, and that 70 to 90% of P is extracted within 1 h. Because these data show that shaking times beyond 1 h have a limited effect on increasing manure WEP, we conducted all extractions for 1 h. We performed all extractions in duplicate.

We measured WEP with single batch extractions by shaking all fresh and dried manures with deionized water at solution to solids ($cm^3\ g^{-1}$) extraction ratios of 10:1, 50:1, 100:1, 150:1, 200:1, and 250:1. All ratios were on an oven-dry weight equivalent basis and accounted for any water in fresh manures. The swine slurry already had a solution to solids ratio of about 16:1, so we filtered the swine slurry directly through a 0.45-μm filter and analyzed the filtered solution for P to approximate the 10:1 extraction ratio.

Table 2. Selected properties of the five manures used in the experiments.

Manure	Dry matter	Total P
	%	$mg\ kg^{-1}$
Dairy with bedding	57.5	1908
Dairy no bedding	25.0	2556
Pennsylvania poultry	32.8	34325
Texas poultry	28.0	24599
Swine	6.3	47374

Researchers have also measured manure WEP with sequential extractions (Dou et al., 2000, 2002), where manure is shaken with water, the sample is centrifuged and the water decanted, and new water is added to repeat the extraction. Adding fresh water will help keep solution P concentrations relatively low, thereby possibly enhancing P desorption or dissolution from manure particles. This in turn may promote greater overall extraction of manure P with sequential extractions than with single batch extractions. To better explore this possibility, we conducted sequential batch extractions of manure as a comparison to single batch extractions. We shook all fresh and dried manures with deionized water at an extraction ratio of 50:1. We centrifuged the samples for 10 min at 1500 × *g* to separate solids and liquids and decanted the liquids. We then added enough deionized water to bring the extraction ratio back up to 50:1 and repeated the extraction. We performed five sequential extractions to compare manure P extracted with each single batch extraction to the cumulative amount extracted over sequential extractions. For example, we compared manure P extracted with a single extraction at a ratio of 150:1 with cumulative manure P extracted over three sequential extractions, each at a ratio of 50:1. One confounding variable in this comparison is that sequential extractions necessarily increased the time with which manure interacted with water. The maximum difference was 5 h of interaction for five sequential extractions compared to 1 h of interaction for a single extraction at a 250:1 ratio. Kleinman et al. (2002b) observed that increasing extraction times from 1 to 4 h increased the amount of P extracted by about 20% when using 200:1 extraction ratios, Dou et al. (2000) observed that increasing extraction times from 1 to 4 h increased P extracted by about 6% when using 100:1 extraction ratios, and Tasistro et al. (2004) rarely saw a difference in P extracted from poultry litter when increasing shaking times from 1 to 4 h at an extraction ratio of 200:1. Given these small potential increases in manure

WEP with extraction time, we felt it was justified to compare single and sequential extractions using all 1-h extraction times, especially at our 50:1 sequential extraction ratio.

We filtered all manure WEP solutions through 0.45-μm filters, and analyzed them for P by a modified colorimetric procedure of Murphy and Riley (1962), with $\lambda = 712$ nm. We also digested the filtered extracts by an alkaline persulfate method (Patton and Kryskalla, 2003), and analyzed digested samples colorimetrically. Analysis after digestion measures total P in the original solution, whereas analysis without digestion measures only inorganic P, with minor measurement of organic or colloidal P (McDowell and Sharpley, 2001). We intended the digestion analysis to be a comparison to P measured by inductively coupled plasma (ICP), which is often used to analyze P in manure extracts. Financial and logistical constraints prevented us from conducting actual ICP analyses. We refer to P measured without digestion as inorganic water-extractable phosphorus (WEP_I), and P measured after digestion as total water-extractable phosphorus (WEP_T).

RESULTS AND DISCUSSION

Single versus Sequential Water Extractions

Manure WEP_I or WEP_T measured by single extractions was not significantly different from WEP_I or WEP_T in sequential extractions for 70% of all 150 comparisons. For the remaining 30%, WEP_I or WEP_T was always greater after sequential extractions. Table 3 shows ratios of manure WEP_I or WEP_T measured by single extractions to cumulative WEP_I or WEP_T measured through comparable sequential extractions (e.g., 150:1 single extraction compared to three consecutive 50:1 extractions). Because there was no consistent effect of extraction ratio on whether or not sequential extractions recovered more P than a single extraction, we averaged data over all extraction ratios. Data reveal that even though sequential extractions occasionally recover more WEP than single extractions, the difference is neither consistent enough nor great enough to justify the extra laboratory work involved for sequential extractions or evaluating the two extraction methods separately. Thus, the remaining discussion of our manure WEP extractions uses data averaged over single and sequential water extractions.

Table 3. Average ratios of manure inorganic water-extractable phosphorus measured colorimetrically without digestion (WEP_I) (or total water-extractable phosphorus measured colorimetrically with digestion, WEP_T) estimated by single extractions at five water to manure ratios ranging from 50:1 to 250:1 to cumulative WEP_I estimated by five sequential extractions at water to manure ratios of 50:1. Values are averages over five extractions.

	Ratio of single to sequential extractions		
Manure	Oven-dried	Air-dried	Fresh
		WEP_I	
Dairy with bedding	0.89	0.87	0.87
Dairy no bedding	0.76	0.83	0.98
Pennsylvania poultry	0.87	0.93	0.90
Texas poultry	0.96	0.97	0.83
Swine	0.88	1.12	0.84
		WEP_T	
Dairy with bedding	0.84	0.88	0.99
Dairy no bedding	0.91	0.91	0.74
Pennsylvania poultry	0.87	0.87	0.84
Texas poultry	0.89	0.99	0.78
Swine	0.86	1.06	0.83

Effect of Extraction Ratio on Manure Water-Extractable Phosphorus

In our extractions, manure WEP_I or WEP_T always increased with the solution to solids extraction ratio, likely due to dilution of P in solution and greater P desorption or dissolution from manure (Fig. 1–3) (Kleinman et al., 2002b). The relationship between manure WEP_I or WEP_T and extraction ratio was consistently nonlinear across the entire range of extraction ratios, regardless of manure type or drying method. For fresh manures, WEP_I or WEP_T extracted at a 250:1 ratio was in all cases, except one for Pennsylvania poultry manure WEP_I, statistically the same as that extracted at 200:1. Thus, a single 1-h water extraction of manure at an extraction ratio of 250:1 may be a method to approximate total manure WEP (Vadas et al., 2004a). However, such extractions are not exhaustive (Chapuis-Lardy et al., 2003), as was evidenced by our sequential extraction data. For some manures, we were still extracting significant amounts of P even after five extractions.

Water-extractable P in surface-applied manures is often well related to dissolved P concentrations in runoff shortly after manure application, but only a few studies have investigated the effect of methodology in measuring manure WEP on the relationship between WEP and runoff P. These studies concentrated mostly on the effect of solids to solution extraction ratios. Kleinman et al. (2002b) showed that when analyzing different manure types for WEP, a consistent extraction ratio gives a better relationship between manure WEP and runoff P than variable ratios. Their limited data also suggest that greater extraction ratios provide a more consistent relationship between manure WEP and runoff P across manure types than lesser ratios (Fig. 4). However, Haggard et al. (2005) observed strong relationships between manure WEP and runoff P from poultry litter for extractions ratios ranging from 10:1 to 200:1. Other studies have also observed strong relationships between manure WEP and runoff for lesser extraction ratios (Vadas et al., 2004b; 20:1 to 40:1 for poultry manure) or greater extraction ratios (Elliott et al., 2005; 200:1 for biosolids). Therefore, it is not clear that one specific extraction ratio ultimately gives the best assessment of potential P loss in runoff from surface-applied manures, especially considering that rainfall and runoff hydrology can have just as an important impact on P loss in runoff as manure WEP (Haggard et al., 2005; Vadas et al., 2004a, 2005).

Effect of Drying Manure on Water-Extractable Phosphorus

Dairy Manure

Figures 1 to 3 show that drying our manures significantly affected WEP_I or WEP_T, but effects depended on drying method, manure type, and extraction ratio. For dairy manure with bedding, the pattern of WEP_I or WEP_T increase with extraction ratio was similar for both air-dried and fresh manure, with WEP in air-dried ty-

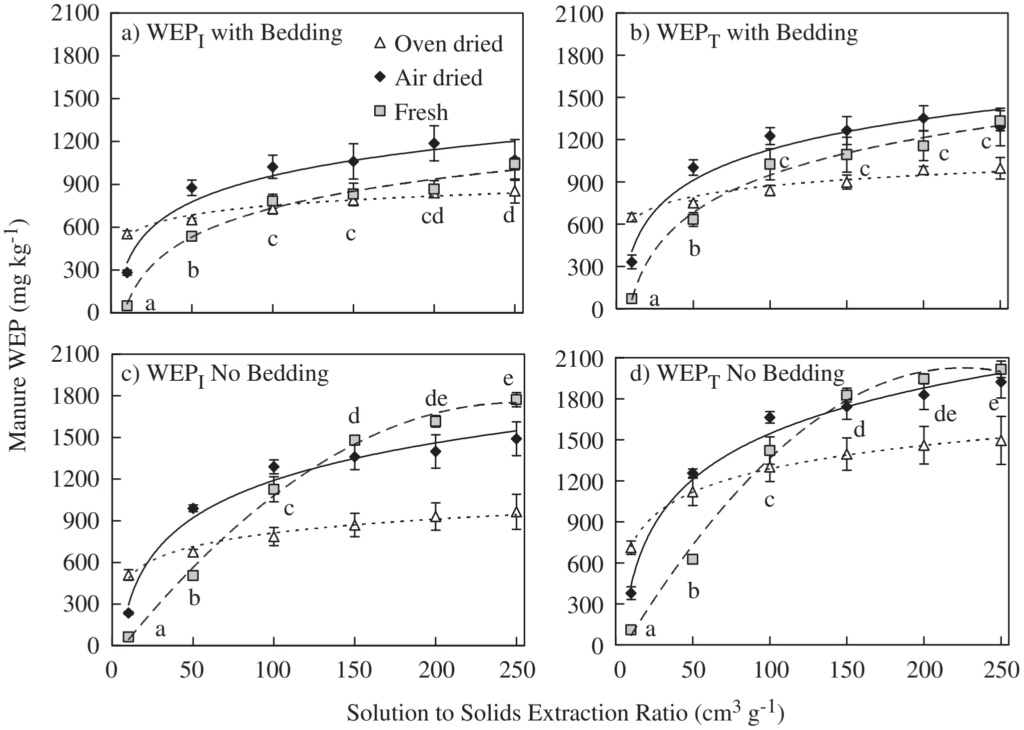

Fig. 1. Effect of extraction ratio on inorganic water-extractable phosphorus measured colorimetrically without digestion (WEP$_I$) and total water-extractable phosphorus measured colorimetrically with digestion (WEP$_T$) in two dairy manures, either (a and b) with or (c and d) without bedding, as analyzed fresh, air-dried, or oven-dried. Data are averages of single and sequential batch extractions.

pically greater than in fresh manure (Fig. 1a and 1b). The same was true for dairy manure without bedding, but only up to an extraction ratio of 100:1 (Fig. 1c and 1d), after which WEP was often greater in fresh manure. McDowell and Stewart (2005) estimated WEP$_I$ at an extraction ratio of 300:1 in cattle manure collected from grazing animals in the field and found that air-drying decreased WEP$_I$, with P apparently transformed to more recalcitrant forms. These observations are consistent with our dairy manure without bedding results, but opposite to manure with bedding results. This suggests that the presence of bedding material might change how air-drying manures affects WEP.

Compared to our fresh manures, WEP$_I$ or WEP$_T$ in oven-dried dairy manure was greater at low extraction ratios but less at greater ratios, with the crossover occurring at a ratio of about 75:1. These variable effects of oven-drying across extraction ratios agree well with results of Chapuis-Lardy et al. (2003) who measured WEP$_T$ in fresh and oven-dried (65°C) dairy manures with bedding over five sequential extractions at an initial extraction ratio of 60:1 and cumulative ratios of 60, 120, 180, 240, and 300:1. They also observed that WEP$_T$ in oven-dried manure was greater than in fresh manure at low extraction ratios, but less at greater extraction ratios, with the crossover occurring at a ratio of about 180:1. Therefore, the effect of oven-drying dairy manure on WEP, and any subsequent interpretation of results, apparently depends on the extraction ratio used. This scenario is demonstrated well by results from Chapuis-

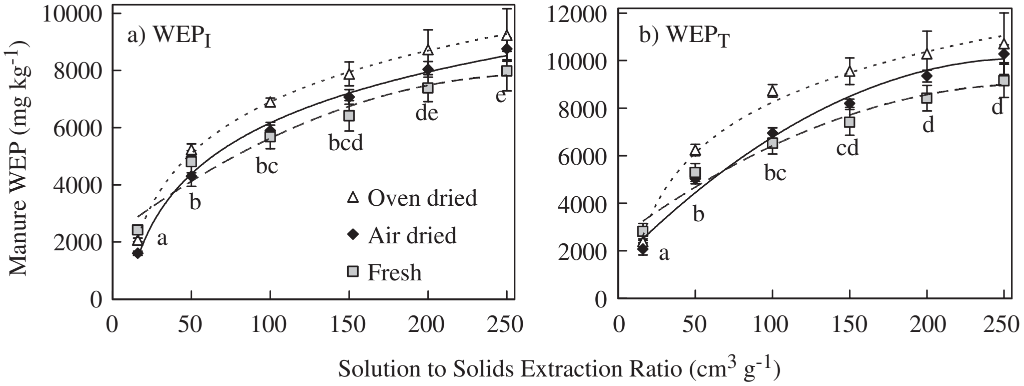

Fig. 2. Effect of extraction ratio on (a) inorganic water-extractable phosphorus measured colorimetrically without digestion (WEP$_I$) and (b) total water-extractable phosphorus measured colorimetrically with digestion (WEP$_T$) in swine manure as analyzed fresh, air-dried, or oven-dried. Data are averages of single and sequential batch extractions.

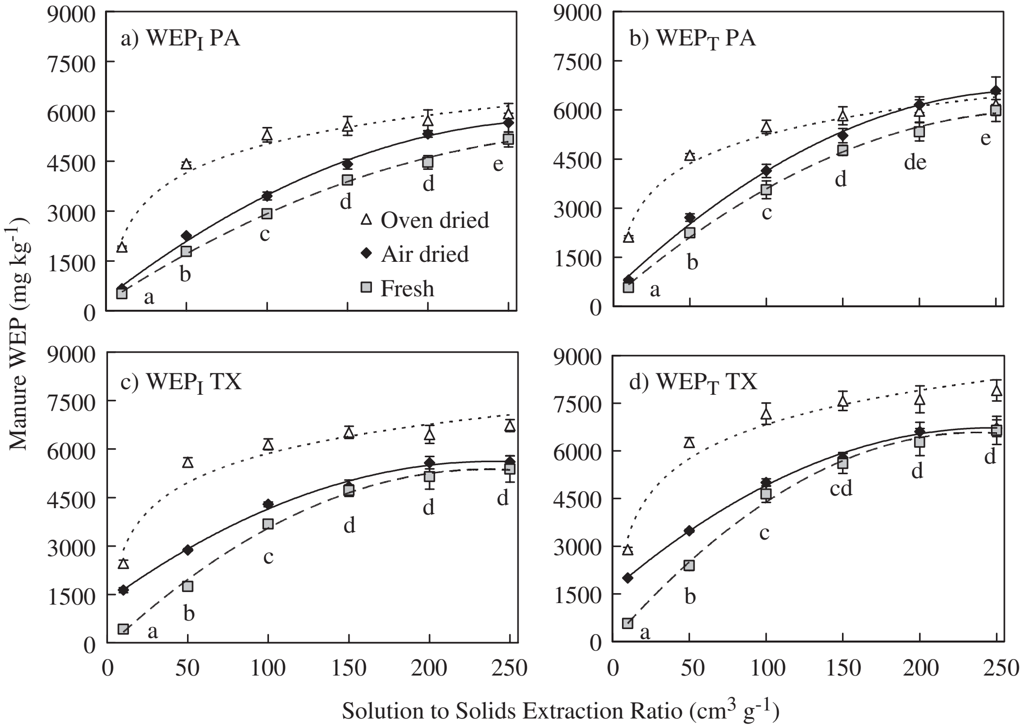

Fig. 3. Effect of extraction ratio on inorganic water-extractable phosphorus measured colorimetrically without digestion (WEP$_I$) and total water-extractable phosphorus measured colorimetrically with digestion (WEP$_T$) in two poultry manures from (a and b) Pennsylvania or (c and d) Texas, both with sawdust–woodchip bedding and as analyzed fresh, air-dried, or oven-dried. Data are averages of single and sequential batch extractions.

Lardy et al. (2004) and Ajiboye et al. (2004). Chapuis-Lardy et al. (2004) measured WEP$_I$ in 40 fresh and oven-dried (65°C) dairy manures without bedding at an extraction ratio of 333:1, which is relatively great. Oven-drying decreased WEP$_I$ in 38 of 40 manures relative to fresh manure, which is consistent with our results and results from Chapuis-Lardy et al. (2003). Conversely, Ajiboye et al. (2004) estimated WEP$_I$ in fresh and oven-dried (105°C) dairy manures without bedding at an extraction ratio of 100:1 and found oven-drying increased WEP$_I$, apparently due to P transformations from $NaHCO_3$–extractable pools. However, this 100:1 ratio was just about at the point where we and Chapuis-Lardy et al. (2003) observed a crossover on the effect of oven-drying on manure WEP. Therefore, based on our data and data from Chapuis-Lardy et al. (2003), it is possible that Ajiboye et al. (2004) could have had different WEP$_I$ results had they used a greater extraction ratio. Overall, our results and results of cited studies show that the effect of drying manures on WEP is complex, involving interactions of extraction ratios, method of drying, animal diet, and bedding material. For example, the data from Chapuis-Lardy et al. (2004) mentioned above showed that decreases in WEP for oven-dried manures ranged widely from 8 to 82%. Such variability suggests that comparing manure WEP data across studies that use fresh or dried manures may be difficult.

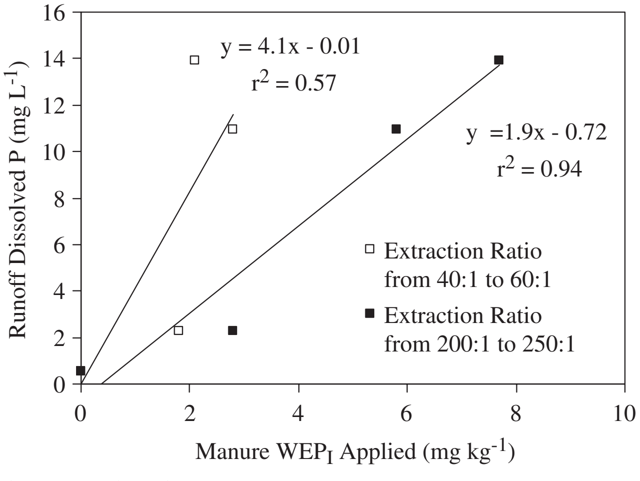

Fig. 4. Relationship between water-extractable phosphorus (WEP) applied to soil boxes in dairy, poultry, and swine manure and dissolved P measured in runoff from the same boxes shortly after manure application. Data are from Kleinman et al. (2002a) and represent manure WEP measured from two different groups of similar extraction ratios.

Swine Manure

In our experiments with swine manure, the pattern of WEP$_I$ or WEP$_T$ increase with extraction ratio was much more similar for fresh and dried manures than it was for dairy manure (Fig. 2). Compared to fresh swine manures, air-drying had a minimal effect on manure WEP$_I$ or WEP$_T$, other than decreasing it slightly at a ratio of 16:1 and increasing it sometimes at ratios greater than 100:1. However, compared to fresh manures, oven-drying consistently increased WEP$_I$ or WEP$_T$ at extraction ratios greater than 16:1. Ajiboye et al. (2004) estimated

WEP_I at an extraction ratio of 100:1 in swine manure and also found that oven-drying (105°C) increased WEP_I relative to fresh manure, apparently due to transformation of organic WEP to WEP_I. Overall, our swine manure data suggest that, at least compared to dairy or poultry manures (see discussion below), drying may not have as great an effect on manure WEP or subsequent interpretations of results. However, oven-drying may tend to increase manure WEP.

Poultry Manure

In our experiments with poultry manures, the pattern of WEP_I or WEP_T increase with extraction ratio was similar for fresh and air-dried manures, with air-drying often, but not always, increasing WEP across the range of extraction ratios (Fig. 3). Compared to fresh manures, oven-drying typically increased manure WEP_I or WEP_T more than air-drying, especially at ratios between 50:1 and 150:1. These increases on oven-drying poultry manures were much greater than for swine manures, and often opposite compared to dairy manures. This may be because poultry manures contain much greater relative concentrations of organic P, such as phytic acid (Turner, 2004), which may break down into inorganic P during oven-drying.

Sistani et al. (2001) estimated WEP_I at an extraction ratio of 15:1 on three poultry manures without bedding that were fresh, air-dried, or oven-dried at both 65 and 105°C. For all manures, WEP_I was greatest in fresh manure, least in manure air-dried or oven-dried at 65°C, and intermediate in manure oven-dried at 105°C, which is inconsistent with our results. Interestingly for our swine manure, which also had no bedding material, WEP_I measured at a 16:1 ratio, which is the same as the ratio of Sistani et al. (2001), was greatest in fresh, least in air-dried, and intermediate in oven-dried manure (Fig. 2a). These results agree with those of Sistani et al. (2001) for poultry manure without bedding. Because swine and poultry are both non-ruminants and have similar diet types and manure P composition (Turner, 2004), it may be plausible to compare results between their manures. Thus, agreement in results between our swine manure and poultry manure of Sistani et al. (2001), and the disagreement in results between our poultry manures and those of Sistani et al. (2001) may have something to do with the presence of bedding material, as we suggested earlier for dairy manure. Had Sistani et al. (2001) used greater extraction ratios, their interpretation of the effect of drying on WEP_I may have changed, as they did for our swine manure (Fig. 2).

Researchers may prefer to dry manures because dried manures are more stable physically and chemically during storage (McGrath et al., 2005) and are more homogenous for weighing and analysis, which enables smaller quantities of manure and extraction vessels and assumedly more efficient laboratory protocols, although this is debatable (Wolf et al., 2005). Our results and those in the literature show drying clearly and inconsistently changes WEP relative to fresh manures, especially for oven-drying. Therefore, drying manures raises the question of how well WEP in dried manures represents that P available to runoff from fresh manures applied to fields. Extraction of fresh manures may represent conditions of a runoff event shortly after manure application, while extraction of air-dried manures may represent conditions of a later runoff event after manures have dried out in the field. Oven-drying may be least representative of field conditions, especially considering it may drastically change manure WEP compared to fresh and air-dried manures. The nature and magnitude of the effect of drying is complicated and inconsistent and could be a function of animal species, animal diet, drying method, bedding material, and extraction ratio. Thus, care must taken to at least acknowledge these factors when reporting and interpreting manure WEP results.

Measurement of Inorganic and Total Water-Extractable Phosphorus

For our manure extractions, WEP_T was always greater than WEP_I, indicating presence of organic and/or colloidal P in extracts not detected by colorimetric analysis. Figures 1 to 3 show that trends in the effect of drying on manure WEP were similar whether P was measured as WEP_T or WEP_I. However, closer examination of data in Table 4 shows that differences in the ratio of WEP_T to WEP_I depended on manure type and drying method. For dairy and poultry manure with bedding, fresh manures had the greatest WEP_T to WEP_I ratio, followed by air-dried and then oven-dried manures. For dairy and swine manure without bedding, oven-dried manures had the greatest WEP_T to WEP_I ratio, followed by air-dried and then fresh manures. Chapuis-Lardy et al. (2004) measured WEP in dairy manures without bedding at an extraction ratio of 333:1 and also found that the ratio of WEP_T to WEP_I was greater in oven-dried manures than fresh manures. McDowell and Stewart (2005) observed a similar result for cattle, sheep, and deer manure without bedding. Apparently, drying can decrease the ratio of WEP_T to WEP_I for manures with bedding, but can increase the ratio in manures without bedding. Therefore, interpretation of manure WEP results can be confounded by the method of P analysis, but to a varying degree based on manure type and bedding management.

Table 4. Average ratios of manure total water-extractable phosphorus measured colorimetrically with digestion (WEP_T) to inorganic water-extractable phosphorus measured colorimetrically without digestion (WEP_I) estimated by single and sequential water extractions at water to manure ratios ranging from 10:1 to 250:1. Values are averages over all extractions.

	Ratio of WEP_T to WEP_I		
Manure	Oven-dried	Air-dried	Fresh
Dairy with bedding	1.16 a†	1.19 a	1.28 b
Pennsylvania poultry	1.04 a	1.18 b	1.20 b
Texas poultry	1.16 a	1.19 a	1.25 b
Dairy no bedding	1.62 a	1.29 b	1.21 c
Swine	1.20 a	1.17 ab	1.14 b

† Values followed by the same letter within a row of a manure type are not significantly different at the 0.05 level.

CONCLUSIONS

Manure WEP data are often used in indexing tools and models that assess the risk of P transport in runoff. Reported methods for estimating manure WEP vary in their prescription of fresh (“as is”) or dried manures, extraction ratios, and method used to analyze P in solutions, typically without acknowledgment of how these methods affect how much P is extracted or interpretation of results. We conducted extractions of dairy, swine, and poultry manures to assess the effect of single and sequential extractions, drying manures, extraction ratios, and method used to analyze P in solutions on WEP. We found little consistent difference between single and sequential extractions across extraction ratios. We also found that stepwise increases in the extraction ratio from 10:1 to 250:1 always increased manure WEP, but in an incrementally decreasing manner so that manure P extracted at a 250:1 ratio was not significantly different from that extracted at a 200:1 ratio. The extent to which manure P increased with extraction ratio varied as a function of manure type and presence of bedding material, whether or not manures were fresh or dried, and the method of drying manures. Fresh and air-dried manures often behaved similarly, but oven-dried manures behaved quite differently. Oven-drying tended to increase WEP slightly in swine manures, but much more in poultry manures. Oven-drying had variable effects on WEP in dairy manures depending on extraction ratio and presence of bedding material.

We analyzed manure water extracts colorimetrically both before and after digestion, which represented the common practice of using ICP to analyze extracts. Digested extracts always contained more P, but the difference depended on manure type and drying treatment. For dairy and poultry manures with bedding, drying decreased the difference in extracted P measured before and after digestion. For the dairy and swine manure without bedding, the opposite was true. Future research could concentrate on how manure treatments such as drying affect specific P forms in manures (Ajiboye et al., 2004; McDowell and Stewart, 2005) to better understand why we observed the changes in manure WEP that we did. Such experiments might still need to vary WEP extraction ratios to see the effect on extractability of specific P forms.

Table 5. Summary of data from Maguire et al. (2005b) and Toor et al. (2005) showing the effect of solution to solids extraction ratio on total water-extractable phosphorus measured colorimetrically with digestion (WEP_T) in manures from various animal diets.

Animal diet	WEP_T 10:1	WEP_T 200:1	Difference from 10:1 to 200:1
	mg kg^{-1}		%
		Maguire et al. (2005b)	
Turkey litter			
Normal	4947	6447	30
Low P	3454	5070	47
Normal + phytase	4383	6285	43
Low + phytase	3178	5010	58
		Toor et al. (2005)	
Turkey litter			
Normal	3184	6313	98
Low P	491	1000	104
Normal + phytase	550	949	73
Low + phytase	447	1178	164
Poultry litter			
Normal	9112	12581	38
0.1 + phytase	9474	14607	54
0.2 + phytase	9798	11062	13
HAP† corn	9899	13501	36
0.1 + phytase	6848	12029	76
0.2 + phytase	5898	7751	31

† High available phosphorus.

Our findings highlight potential difficulties in comparing data across studies that use different methods to estimate manure WEP, especially when inferring differences in the potential environmental impact of practices affecting manure properties. A case in point is the interpretation of manure WEP data from animal diet manipulation studies. Research has shown that phytase or high-available-phosphorus (HAP) corn in feeds with or without reductions in inorganic P supplementation has both increased and decreased manure WEP (Maguire et al., 2005a). However, of 12 phytase or HAP corn studies we reviewed, methods used to estimate manure WEP varied widely in use of dry or fresh manures, extraction ratios, times of shaking, acidification of extracts, and analysis of P in extracts (Table 1). Results from our present study and the literature show that such variations can inconsistently affect manure WEP. For example, Maguire et al. (2005b) and Toor et al. (2005) analyzed oven-dried manures at extraction ratios of both 10:1 and 200:1 (Table 5). Using an extraction ratio of 200:1 always extracted more WEP_T, but by an inconsistent relative amount that ranged from 13 to 164%. Similarly, McGrath et al. (2005) analyzed fresh poultry litters at an extraction ratio of 10:1 and oven-dried (60°C) litters at an extraction ratio of 200:1 (Table 6). They observed that the 200:1 extraction of dried litters always produced more WEP, but by an inconsistent relative amount that ranged from 102 to 359%. The greater magnitude of these increases compared with data from

Table 6. Summary of data from McGrath et al. (2005) showing the effect of solution to solids extraction ratio and drying manures on total water-extractable phosphorus measured colorimetrically with digestion (WEP_T) in manures from various animal diets.

Animal diet†	Fresh manure WEP_T at 10:1	Oven-dried manure WEP_T at 200:1	Difference from 10:1 to 200:1
	mg kg^{-1}		%
		Initial	
NRC	1065	4164	291
NRC + phytase	1043	3600	245
UMD	897	1812	102
UMD + phytase	633	2226	252
		Stored dry	
NRC	1573	4247	170
NRC + phytase	1349	5009	271
UMD	838	2782	232
UMD + phytase	819	2621	220
		Stored wet	
NRC	2585	8624	234
NRC + phytase	2170	8031	270
UMD	1582	6955	340
UMD + phytase	1115	5113	359

† NRC, National Research Council recommendations; UMD, University of Maryland recommendations.

Maguire et al. (2005b) and Toor et al. (2005) is likely due to the compounded influence of oven-drying.

These examples and results from our study clearly point to a need to establish a standard method of WEP analysis in manures so that results across and even within studies are more comparable. Data suggest that given the inconsistent effect of drying manures on WEP, such a method should extract fresh manure to best represent what will be applied to fields. While our study does not conclusively identify an extraction ratio that is most effective for estimating P runoff potential (although it suggests greater ratios produce more consistent results across manure types), it clearly highlights the need to control the ratio to consistently estimate manure WEP. In addition, when manure WEP data are intended to estimate dissolved inorganic P transport in runoff, manure extracts should be analyzed colorimetrically without digestion, although this may be most important for quantitative simulation models that attempt to differentiate between organic and inorganic P. Analysis of extracts after digestion or by ICP could be used to estimate total P transport in runoff, and not just inorganic P. Most importantly, when presenting and comparing data across studies, the effect of variations in manure WEP methodology must be acknowledged and taken into account.

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