Determining water extractable P in animal manure and biosolids

Ann Wolf, Pennsylvania State University
Philip Moore, Jr., USDA-ARS, Fayetteville, AR
Peter Kleinman, USDA-ARS, University Park, PA
Dan Sullivan, University of Oregon

Introduction:

The water extractable P content of land-applied manures and biosolids is a key water quality indicator, and has been used in a variety of contexts to evaluate different materials on the basis of their potential to release dissolved P to runoff water. Consistent and meaningful measurement of water extractable P has been obtained by a variety of methods, each with its own set of advantages and limitations(Self-Davis and Moore, 2000; Sharpley and Moyer, 2000, Haggard et al., 2005; Baum et al., 2006; Vadas and Kleinman, 2006). Key differences in methods include how materials are processed (drying, grinding), whether fresh materials are extracted on a dry weight equivalent basis or on a wet basis, the amount of sample that is extracted, extraction ratio (solution:solids), extraction time, solid separation technique, and method of P determination (ICP vs. colorimetry). Recent efforts by SERA-17 culminated in the selection of a universal water extraction protocol that addresses the concerns of testing laboratories (reproducibility, ease of implementation, and adaptation to different manures and biosolids) while providing a strong correlation between WEP and with dissolved P in runoff (Kleinman et al., 2007).

Two water extraction methods are reported in this chapter. The first, adapted from Kleinman et al. (2007) represents the consensus method of SERA-17 and, more importantly, has been shown to work well with a broad array of manures and biosolids (dry and liquid). The second method, derived from Self-Davis and Moore (2000), is also reported because it is currently required by the Arkansas P Index for Pastures. That method has been used consistently and reliably with poultry litter samples. However, because the Self-Davis and Moore (2000) method does not fix the extraction ratio, it does not provide reliable comparisons of water extractable P across manures with varying moisture content (see Kleinman et al., 2002). Furthermore, due to the relatively low extraction ratio (approximately 10:1) of the Self-Davis and Moore (2000) method, it presents practical difficulties and cannot be used with certain manures and biosolids (see Kleinman et al., 2007).

Universal Water Extractable P Test for Manure and Biosolids (adapted from Kleinman et al., 2007)

Summary of Method:

A representative sample containing 2.0 g of solids is extracted with water at a 100:1 solution:solids ratio for 1 hr, centrifuged and, if necessary, filtered. The filtrate is analyzed for phosphorus (P) by inductively coupled argon plasma spectroscopy (ICP).

Apparatus and Materials:

1. Analytical balance, 300 g capacity, minimum accuracy of $\pm$ 0.001 g

2. 250 mL centrifuge bottles
3. Reciprocating shaker capable of *180-200* epm
4. Centrifuge capable of 3,000 or greater rpm
5. Whatman No. 40 filter paper and filter funnels

Reagents:
1. Reagent grade water, minimum resistance of 17 MΩ cm
2. ICP Standards: P standards ranging from 0 (Reagent grade water blank) up to 100 mg/L P.

Sample Preservation and Handling :
1. All sample containers should be pre-washed with phosphate-free detergents and rinsed with deionized water. Plastic and glass containers are both suitable.
2. Samples shall be refrigerated (4^{o} C) upon receipt and analyzed within three weeks.

Procedure
1. Determine the percent solids of the manure on a separate subsample (Peters, 2003).
2. Based on the percent solids determination, weigh or pipet a sample containing 2.0 g solids (see calculations below) into a 250 mL centrifuge bottle.
3. Add reagent grade water to bring to a final weight of 202 g. If solids content of sample is less than or equal to 1%, do not add additional water. Use 200 g of the as-received sample.
4. Shake samples on a reciprocating shaker (180-200 epm) for 60 minutes.
5. Remove bottles from shaker and centrifuge (3,000 rpm or greater) for 10 minutes. If particulates are present, filter the centrifugate (No. 40 Whatman).
6. Analyze centrifugate for P on the ICP. If sample extract can not be analyzed immediately, acidify to prevent precipitation of calcium phosphates by adding 5 drops of concentrated HCl for each 20 mL of extract to lower pH to approximately 2.0. Acidified extracts can be held for up to three weeks before P measurement. *Samples may also be analyzed colorimetrically but should be referenced to ICP analyses due to matrix interference issues.*

Calculations:
1. Sample size needed to provide 2.0 g solids is determined as follow:

$$\text{Sample size to provide 2.0 g} = 2 \times \frac{100}{\%\ \text{solids}}$$

2. The P concentration on a dry weight basis in % is determined as follows:

$$\text{P (\%) dry weight basis} = \frac{\text{ICP P result (mg/L)} \times 0.02}{\text{Sample size wet (g)}} \times \frac{100}{\%\text{solids}}$$

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