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## **NOVEL PROCEDURES FOR ACCURATE SAMPLING OF SWINE MANURES**

**P. M. Ndegwa, and J. Zhu**

### **ABSTRACT**

Three novel sampling methods for obtaining representative samples prior to the actual pump out time from manure stored in pits were investigated in this study. In addition, empirical relationships between nutrients (N and P) and total solids that could help in quicker determinations of the manure nutrient contents on-site during scheduled pump-out time were also investigated. Results showed that, stirring representative manure in a 55-gallon barrel for a period of at least five minutes using a paddle-stirrer such as the one described in this study is adequate for complete homogenization of manure essential for drawing off a representative sample from any point in the barrel. Further, results showed that, the use of a sump pump (such as the one described in this study) placed at the bottom of a similar barrel and oriented to shoot manure horizontally is also adequate for homogenizing the manure after five minutes of operation: a procedure that also allows for collection of a representative sample from any point in the container. Using the above two techniques, correct nutrient determinations are possible before the pump-out date, allowing for proper planning of the manure application in the field. The correlation coefficient ( $R = 0.26$ ) of a linear regression between total solids (TS) and total Kjeldahl nitrogen (TKN) using data obtained in this study was too low, suggesting that the content of TS cannot accurately be used to estimate the content of the TKN. The correlation coefficient ( $R = 0.92$ ) of a linear regression between TS and total phosphorus (TP) obtained using data from this study indicated that, the content of TS can be used to accurately estimate the content of TP in the manure.

**Keywords.** Swine manure, Representative sampling, Nutrient estimation, Phosphorus, Nitrogen.

### **INTRODUCTION**

The vast majority of manure management systems employed in hog production facilities in the state of Minnesota and other neighboring states consist of collection and storage of liquid/slurry manure in pits. These pits are located either within or outside the production facilities. The designs

of the pits also, differ in many respects: construction material, shape, size, etc. In addition, the variation of manure slurries stored in these pits exists due to differences in feed rations, stage of growth of the pigs/piglets, and the differences in management schemes. The net effect of the preceding scenarios is the uniqueness of nutrient composition of each manure-holding pit.

Critical to accurate determination of manure application rates is undoubtedly the correct nutrient contents of the manure in question. In turn, the correct nutrient content depends upon the integrities of the samples and the sampling methods. In practice today, there are generally two ways in which nutrient contents are obtained: (1) book values (ASAE, 1993; MWPS, 1985; Lorimor et al., 1995; Campbell et al., 1997; Killorn and Lorimor, 1999) based on average of manure samples from a particular region, or (2) analysis of manure samples in commercial analytical labs. The book values are useful to provide reasonable ranges for planning purposes but should not be used to develop site-specific nutrient plans. Specifically, book values do not allow accounting for large variations in manure nutrients because of such factors as animal age, diet, productivity, managements and storage practices. The commercial lab analyses of manure samples though more reliable involves waiting one to two weeks prior to getting the results, which is not practical since application must be done immediately. This latter approach could, however, be feasible if the sampling could accurately be done one to two weeks prior to scheduled pump-out time. Currently, there is no standard method(s) of manure sampling that ensures representative samples are obtained prior to pump out.

The problem of obtaining representative samples of manure either at the time of pump-out or prior to the pump-out time is generally well recognized not only because of the non-homogenous nature of the manure but also because of the tendency of manure to naturally stratify. On the other hand, however, increasing public concerns of possible deleterious effects of farming on the environment are reflected in new and impending federal and state legislation regarding animal operations. In a number of states or regions, swine producers are (will be) required to file detailed nutrient management plans with local authorities. These plans are designed to track the quantity of nutrients (primarily N and P) that are being imported onto and exported from each farm. To protect swine producers and also to enhance compliance, there exists an acute need for a reliable sampling and testing method to accurately determine the manure nutrients content prior to land application of manure. In areas where these kinds of legislations are not in place, in order to encourage farmers to make better use of manure slurries, and therefore, reduce pollution risks, convenient and modest methods to accurately quantify the nutrients are necessary in order to spread these slurries efficiently and in accordance with codes of good agricultural practice. These goals can be realized by either identifying and developing suitable sampling methods that would allow sufficient time to get back the analytical results from the commercial laboratory without delaying the pump-out schedule or by providing some form of quick nutrient determination that can be used on-site during the scheduled pump-out time.

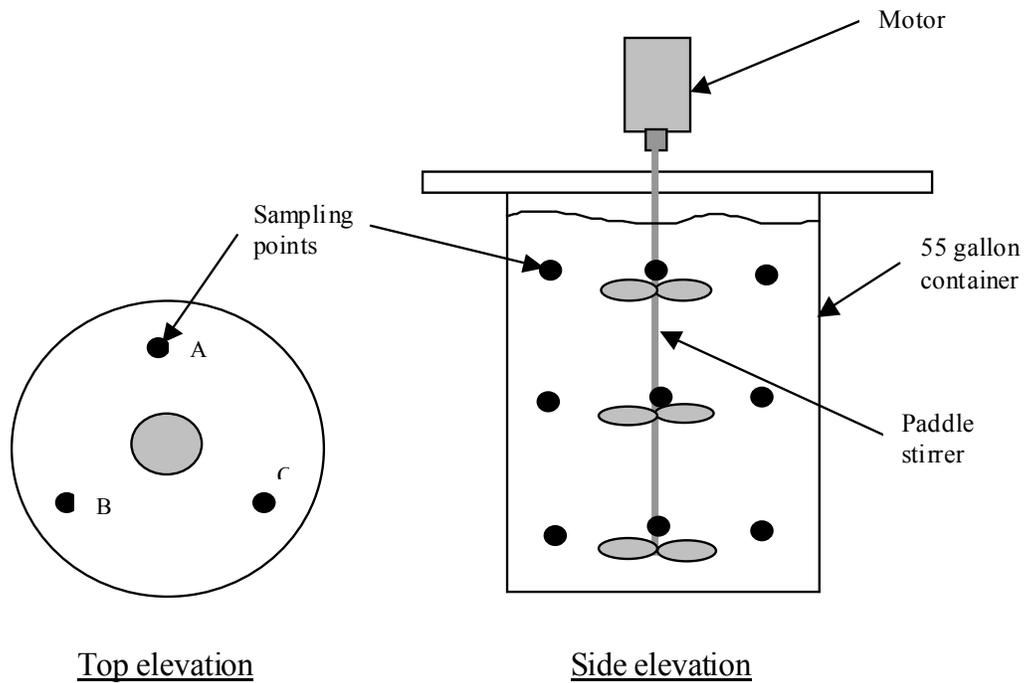
The objectives of this study were to: (i) evaluate novel sampling methods that can be employed prior to pump-out to provide representative manure samples comparable to the samples obtained at pump-out when complete mixing is assumed, and (ii) determine an empirical relationship between the nutrients and another more easily measurable quantity that can be used on-site during scheduled pump-out time.

## **MATERIALS AND METHODS**

### Sampling Methods

#### Method I

A schematic of the manure sampling device and the sampling locations (points) for method I, is shown in Figure 1. In this arrangement, a paddle-stirrer (Model C6C17FBIE, Cat No. 110012.00, RPM 1725, ½ HP, Lesson Electric Co.) was allowed to run for approximately five minutes prior to sampling. This stirrer created a vertical and outward swirling mixing of manure. After this mixing period and while still stirring the manure, samples were drawn off from top to bottom at 2.5 cm (1 in.) from top and bottom ends, and at mid-depth using a vacuum siphon. At each sampling depth, the manure was sampled at approximately the three points marked A, B, and C, equally spaced on an imaginary circle concentric with the barrel's rim but leaving approximately 5.0 cm (2 in.) from the wall. These points had previously been marked on the manure container to provide a guide during sampling. Each run therefore resulted in triplicates samples for each depth and for each location. Three runs with manure from the same manure batch/source resulted in nine samples at each depth, and nine samples for each lateral location, resulting in a total of 81 samples. Between runs, the barrels were refilled to the initial level to compensate for the samples drawn during the preceding run.



**Figure 1. Schematic of manure sampling device and sampling locations (container dimensions: height=91 cm; diameter=56 cm)**

### Method II

The same setting as that used in method I was used except that, instead of using the paddle stirrer to mix the manure, a sump pump (Model 6E-CIM, 1/3 HP, 1 Phase, 3200 GPH @ 5 Feet of Head, Little Giant Pump Co.) sitting at the bottom of the container with the outgoing stream shooting horizontally was used to facilitate manure agitation. This agitation scheme closely simulated the common practice used by producers when emptying manure pits or other similar storages. After five minutes of manure agitation, the manure was sampled in exactly the same depths and lateral locations as in method I. Just like in method I, three runs with same batch of manure from each of the three different sources resulted in a total of 81 samples.

### Method III

Again, the same setting as in the previous two methods was used except that after thoroughly mixing, the manure was left undisturbed for five hours. Samples were then drawn off from the exactly the same depths and locations as in the previous two methods. This method simulates the sampling procedure in deep pits without agitation and has been preciously been investigated by

various researchers (Campbell et al., 1997; Lorimor and Kohl, 2000). It was therefore chosen to provide a baseline for evaluating the other two procedures.

### Source of Swine Manure

For the purpose of evaluating and/or comparing the above three methods, it was necessary to use the same batch from each source to minimize variations in manure characteristics for a particular growth phase. Three farms from around Waseca County in Minnesota were randomly selected to represent swine manure at different growth phases (gestation, nursery, and finishing). From each of these farms, enough manure to fill a 55-gallon barrel was obtained for the sampling study. To ensure adequate representation, the manure was drawn off by continuously and steadily lowering and raising the sump pump in the manure pits in a random manner in each case.

### Laboratory analyses

Although manure solids is the major factor governing the uniformity of the samples and therefore, the best indicator to determine if a method of sampling produces uniform or representative samples, manure nutrients (total Kjeldahl nitrogen (TKN) and total phosphorus (TP)) contents were also determined for a more thorough evaluation of the sampling procedures as well as to provide data for the determination of the empirical relationships between TS and the nutrients.

The following parameters were determined for each collected sample using standard laboratory methods (APHA, 1998): Total Solids (TS), TP and TKN. For determination of TS, a well-mixed sample was evaporated in a pre-weighed dish and dried to constant weight in an oven at 105°C. The increase in weight over that of the empty dish represented the TS. Total phosphorus was determined using the Persulfate Digestion Method. In this method all the species of P in a sample are first converted to orthophosphates. The samples are then filtered and the P is measured quantitatively using the ascorbic acid method. Nitrogen (N) was determined as TKN in which both organic N and ammonia N are converted to ammonium in the presence of concentrated H<sub>2</sub>SO<sub>4</sub> and cupric sulphate (CuSO<sub>4</sub>) catalyst. After addition of base, ammonia is distilled from an alkaline medium and absorbed in boric acid. The distilled ammonia is determined by titration with a standard mineral acid.

### Experiment design and statistical analyses

To determine the homogeneity (or lack of homogeneity) of the samples obtained by each sampling method, TS, TP, and TKN were measured at three depths and three lateral locations for each of the manure collected from the gestation, finishing, and nursery, at three replications, for each method. This is a typical four-factor factorial design (sampling method, source of manure, depth, lateral location) with three replicates and was the model used to perform analyses of variance on the responses (TS, TKN, and TP). In addition to this, each method was individually evaluated with respect to manure for the three sources on the basis of uniformity of samples with respect to depth of sampling and location of sampling. Analyses of variances (ANOVA) were performed using

standard PC-based statistical analysis software (SAS, 2000). Unless otherwise stated statistical significance is implied at the 5% significance level.

## RESULTS AND DISCUSSION

### Evaluations of Each Method Individually

The summary of the statistics of TS, TKN, and TP of the samples obtained with each of the three different methods at the three different depths and lateral locations for the three different sources of manure are presented in Tables 2 and 3. Apart for sampling by method III, the results of sampling using the other two methods are not significantly different with either the depth or lateral location of sampling. Although significantly more TS are observed in the samples drawn off at the top using method I for the manure obtained from the nursery, this does not have any significant effect on the nutrient determinations. These results suggest that, as long as at least a five minute mixing or agitation period exists prior to sampling using the mixing/agitation equipment similar to the one used in this study, a representative sample can be drawn just about anywhere in this or a similar container for nutrient determination.

**Table 1. Summary statistics of TS, TKN and TP of the manure sampling by source and depth (n=9).**

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**Table 2. Summary statistics of TS, TKN and TP of the manure sampling by source and Location (n=9)**

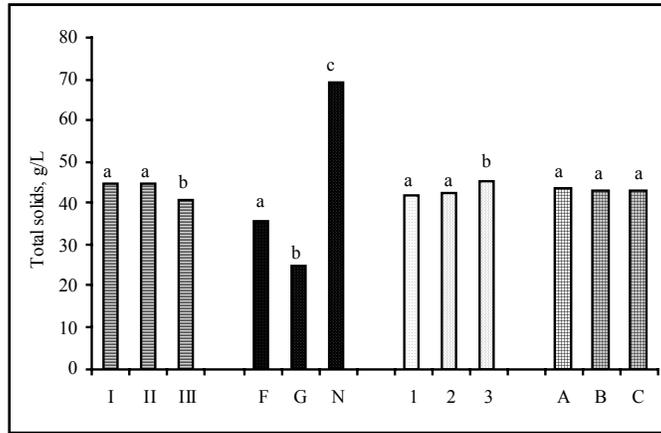
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Method III depicts typical stratification of the manure in holding or storage pits especially with respect to the manure slurries from the gestation barn. This natural stratification of the manure is not observed in the manure from the nursery-barn pit and only moderate stratification is observed in the manure from the finishing barn. The authors believe that the manure slurry obtained from the nursery-barn (approximately 6.9% solids) and the finishing barn (approximately 3.6%) was perhaps too thick to significantly stratify during the five hours of quiescent settling. This observation agrees with previous research (Ndegwa et al., 2001), which showed that, solid sedimentation decreased with increasing solid content from approximately 66% to only 8% effective sedimentation at 1% and 6% TS, respectively. Based on this observation, it can be inferred that, for such thick manure slurries and within 5 hours of agitation, representative manure samples can be obtained at any point at any time. Although this finding may not be important for the purpose of manure sampling (no need for such long delays after agitation before collecting samples), it is very important in mixed reactor design and operations where complete mixing is desired over the reaction periods. However, more work is necessary to further validate this postulation.

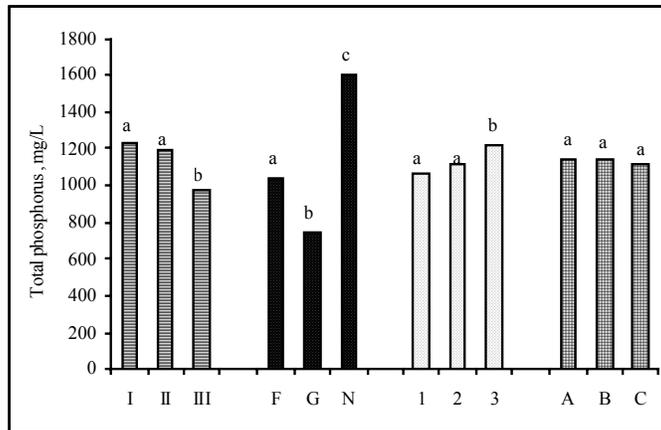
### Effects of all the four factors on representative sampling

The analyses of variance of the manure samples by method of sampling, source of manure (gestation, finishing, nursery), depth of sampling and, lateral location of sampling for the TS, TKN, and TP are summarized in Figures 2, 3, and 4, respectively. The results of TS presented in Figure 2 shows that, with a sample size (n) of 81, methods I and II produced the same results for the TS while method III produced significantly different results. The results of both the nutrients (N and P), shown on Figures 3 and 4 are similarly quite interesting. The quantities of TP follows the same trend as that of TS, while that of TKN does not. These results suggest that, the distribution of TP is closely linked to the distribution of TS, i.e., most of the TP is found in the solids. On the other hand, it appears that, for N, most of it is found in solution and hence does not follow the natural stratification of the manure solids. This observation is more evident in Figures 5 and 6, which shows the linear regressions of TKN and TP versus TS. There is obviously little correlation between TS and TKN ( $R = 0.24$ ) while a high correlation of TP against TS ( $R = 0.92$ ) is existed.

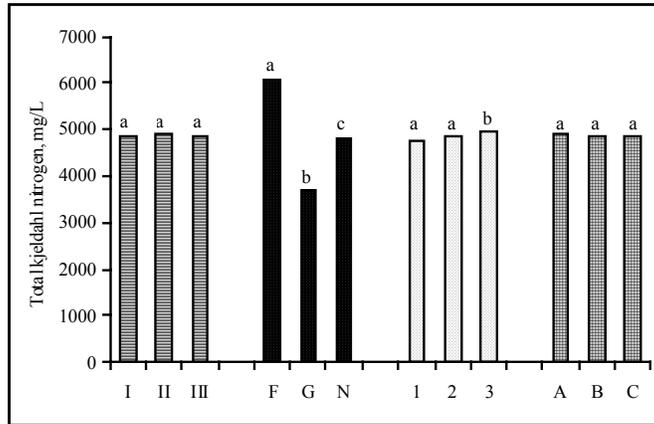
Apparently, close versions of method III have been evaluated in the past for manure sampling. Lorimor and Kohl (2000) compared the means of TKN and TP of a vertical profile with the means of top, middle and bottom samples of several pits in Iowa and found that, while TKN values were not significantly different, the TP values were significantly different. Further, they observed that unagitated vertical profile samples were not significantly different from samples taken during the complete agitation for TS, TP except TS. Campbell et al. (1997) on the other hand noted that, compared to a composite sample of top, mid-depth, and bottom of the pits, the mid-depth sample generally represented the average manure nutrient concentration in the storage. Since it is reasonable to expect the manure homogenized by mixing or agitation to give more accurate results irrespective of the depth or location of sampling as is observed in this study with respect to methods I and II, then, from the data obtained in this study, the means of top, mid-depth and bottom samples are found to underestimate the TS and TP. The authors believe this happens because the sludge occupies a smaller depth or volume of the total depth or volume, i.e. the top and the mid-depth samples are likely to come from above the sludge thus skewing the effective TS and TP towards these lower values. On the other hand, since most of the TKN is perhaps in solution, the settling of the solids do not skew the means towards either top or bottom. Although Lorimor and Kohl (2000) and Campbell et al. (1977) procedures can be used to provide estimates of nutrients (TKN and TP), they are more likely to underestimate the TP compared to the complete mixing of manure described in this study.



**Figure 2. Summary of TS with respect to method of determination, source of manure, depth, and location of sampling (means with same letter within the same category are not significantly different at  $\alpha=0.05$ )**



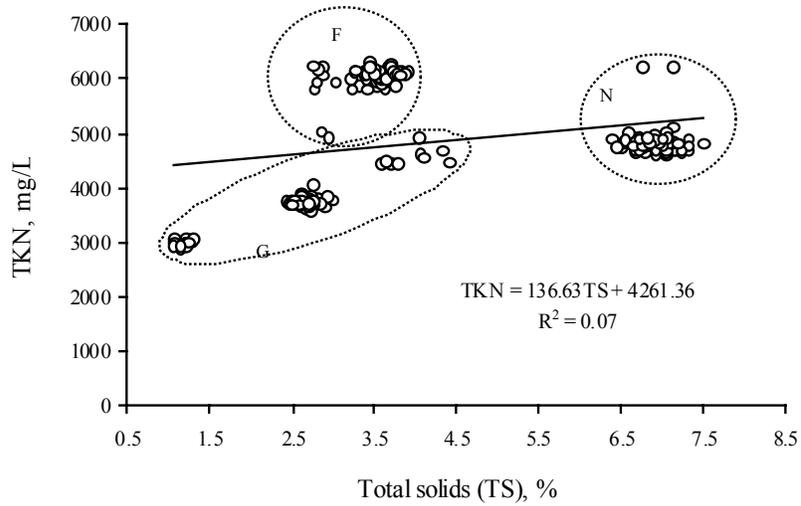
**Figure 3. Summary of TKN with respect to method of determination, source of manure, depth, and location of sampling (means with same letter within the same category are not significantly different at  $\alpha=0.05$ ).**



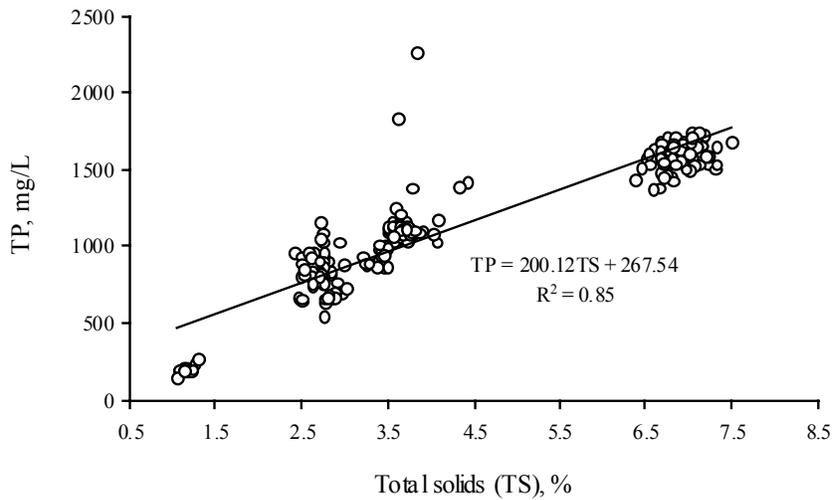
**Figure 4. Summary of TP with respect to method of determination, source of manure, depth, and location of sampling (means with same letter within the same category are not significantly different at  $\alpha=0.05$ )**

### TS versus TKN and TP

The relationships between nutrients: TKN and TP, and TS are shown in Figures 5 and 6, respectively. These plots were made using the consolidated data obtained in this study ( $n=241$ ). Since previous research (Ndegwa et al., 2001; Tunney, 1969; Dragun, 1978; Chescheir et al., 1985; Piccinini and Bortone, 1991; Scotford et al., 1998) indicated linear relationships between the nitrogen and phosphorus in the manure, linear regressions relationships were chosen to enable comparison with previous studies. As noted above, there was little correlation between TS and TKN ( $R = 0.24$ ) while a high correlation of TP against TS ( $R = 0.92$ ) is observed. In Figure 5, it is quite apparent that, the TKN values were easily separable by the source of manure (N=nursery, G=gestation, F=finishing) and the correlations between TKN and TS were easily not discernable except for the manure from the gestation barn. The TKN in the manure from the latter barn correlated fairly well with the TS while the TKN in the manure from the former barns remained more or less constant within the narrow TS ranges.



**Figure 5. Linear correlations between TKN and total solids (TS) for the consolidated data (N=nursery, G=gestation, & F=finishing)**



**Figure 6. Linear correlations between TP and total solids (TS) for the consolidated data (nursery, gestation, & finishing)**

The results of this study are within the ranges of the results obtained in past studies as can be seen in Table 3. However, inconsistencies from one study to another are still large leading to huge variation on nutrient estimations based on individual regressions. In view of this, no individual empirical equation(s) can be recommended as a representative for accurate estimates of nutrients in the swine manure slurries based on only the analysis of TS of manure. It is, however, likely that

for a given production facility with more or less unchanging pig/piglets diet and unchanging other facility management schemes, such an empirical relationship could be feasible. However, research is still needed to verify this postulation.

**Table 3. Regression equations for TS versus N and P from existing literature and this study**

Related Parameter <sup>†</sup>	Regression Equation	R	Source
TS vs. N	N = 1194 + 424 (TS)	0.92	Tunney, 1979
	N = 3753 + 186 (TS)	0.28	Literature*
	N = 362 + 598 (TS)	0.95	Dragun, 1978
	N = 2433 + 396 (TS)	0.88	Chescheir, et al., 1985
	N = 1095 + 600 (TS)	0.90	Piccinini & Bortone, 1991 <sup>‡</sup>
	N = 3363 + 415 (TS)	0.95	Ndegwa et al., 2001
	N = 4261 + 136 (TS)	0.26	Current study
TS vs. P	P = -117 + 232 (TS)	0.92	Tunney, 1979
	P = 305 + 186 (TS)	0.75	Literature*
	P = 112 + 239 (TS)	0.94	Dragun, 1978
	P = -112 + 334 (TS)	0.77	Chescheir, et al., 1985
	P = 26 + 269 (TS)	0.84	Scotford et al., 1998
	P = 320 + 312 (TS)	0.88	Piccinini & Bortone, 1991 <sup>‡</sup>
	P = 264 + 383 (TS)	0.85	Ndegwa et al., 2001
	P = 200 + 267 (TS)	0.85	Current study

\*Compiled by Chescheir, et al. (1985) from other literature sources

<sup>†</sup>Units: TS (%), N (ppm), and P (ppm)

<sup>‡</sup>Converted from different units by the authors

### Methods use at Farm Level

Both methods I and II can be used on the farm to help in getting representative samples prior to scheduled pump-out time. The authors envisage the use of similar barrels without bottoms that can be placed in the pits to curve out or separate limited quantities of manure for agitation with either of the two devices described in this study. This way, representative samples can be obtained without agitation of the entire pit but just by agitating smaller portions of manure that represent the manure in the respective pits.

## CONCLUSIONS

Stirring representative manure in a 55-gallon barrel for a period of at least five minutes using a paddle-stirrer such as the one described in this study is adequate for complete homogenisation of the manure. This implies that a representative sample of the manure in question can be drawn off from any point in the barrel.

The use of a sump pump (such as the one described in this study) placed at the bottom of a similar barrel and oriented to shoot the manure horizontally is also adequate for homogenizing the manure after five minutes of operation. This procedure, therefore, also allows for collection of a representative sample from any point in the container.

Using the above two techniques, correct nutrient determinations are possible before the pump-out date, allowing for proper planning of the manure application in the field.

The correlation coefficient ( $R = 0.26$ ) of a linear regression between TS and TKN using data obtained in this study was too low. This result suggests that the content of TS cannot accurately be used to estimate the content of the TKN. The correlation coefficient ( $R = 0.92$ ) of a linear regression between TS and TP obtained using data from this study indicates that, the content of TS can be used to accurately estimate the content of TP. This result is corroborated by numerous past research findings.

#### Acknowledgement

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