

Comparison of sampling methods for animal manure

P.J.L. DERIKX*, N.W.M. OGINK AND P. HOEKSMA

Institute of Agricultural and Environmental Engineering (IMAG-DLO), P.O. Box 43,
NL-6700 AA Wageningen, The Netherlands.

* Corresponding author: (fax: + 31-317-425670; e-mail: p.j.l.derikx@imag.dlo.nl)

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Abstract

More stringent legislation on undesirable mineral losses to the environment at the farm level creates the need for the farmer to obtain accurate information on the mineral flows at his farm, including the mineral content of manure. Both currently available and recently developed new sampling methods for slurry and solid manure were tested for bias and reproducibility in the determination of total phosphorous and nitrogen content of the samples. Sampling methods were based on techniques in which samples were taken either during loading from the hose or from the transporting vehicle after loading.

It was demonstrated that most methods were unbiased. New methods for slurry, based on sampling from the hose, were up to a factor two better reproducible compared to existing methods. Sampling of solid manure can be done accurately in a relatively simple way. For practical reasons mechanisation of sampling is desirable. To minimize the influence of human activity on the quality of the sample automatization is strongly advisable.

Keywords: sampling methods, accuracy, animal waste, transport, minerals, manure composition

Introduction

In The Netherlands increasing specialisation and expansion of farms has led to larger numbers of animals per farm over the last decades. This expansion did not coincide with an increase in available arable land or grassland on the farm, especially for chicken and pig farms. As the majority of these animals are fed with concentrates from outside the farm, mineral surpluses were created on farm level, which become manifest as large amounts of animal manure.

Future government strategies are focusing on balancing the mineral input and output of farms, taking into account the unavoidable losses due to soil processes such as denitrification. To create such a balanced situation, livestock producers with mineral surpluses on their own land have to transport animal manure to arable land of other farms. These mineral surpluses are not only a problem in terms of quantity itself, which could be addressed by monitoring nutrients feed going into the farm and ani-

mal production leaving the farm. The water management on the farm determines the dilution factor of the surplusses. To enable farmers and authorities to keep track of the minerals on the farms, correct figures for the mineral contents of manures should be used. Therefore, samples from manure transports originating from the particular farm have to be taken and analyzed. Evidently, the availability of accurate and practical sampling methods for manure transports plays a key role in this approach. So far, only a few sampling methods for manure transports have been used in practice, and no information on their accuracy was available. Given these restrictions a research scheme started in 1995 to develop new methods for manure transports and to test, under practical conditions, both conventional and newly developed sampling methods.

Sampling large quantities of other inhomogeneous products is subject of many reports (Leschber, 1986; Süß, 1988; Johnson *et al.*, 1993). Sampling techniques and strategies highly depend on the structure of the material and its homogeneity. Impurities in these products may cause specific troubles. Animal manure is an example of material which demands special attention in respect to sampling. Although figures about manure composition are included quite frequently in literature, information on the applied sampling method and technique is rather sparse (Japenga & Harmsen, 1990; Flachowsky and Hennig, 1990; Stephenson *et al.*, 1990).

A major characteristic of animal waste is the water content of the product. Depending on the housing and the management system the water content of animal manure ranges from 990 g/kg for veal calf manure to 200 g/kg for pre-dried chicken manure; different sampling techniques are required for sampling these two extremes. In general there is a differentiation between slurries with a water content down to 850 g/kg and solid manure with a water content of 700 g/kg or less. In between these levels the manure is very difficult to handle as it cannot be transported by means of pumping and sticks to transport means for solid products such as conveyor belts. For these reasons farmers try to avoid to produce animal manure with a water content between 700 and 850 g/kg.

In sampling slurries particular attention should be paid to the aspect of sedimentation. As slurry contains solid particles with a high specific density, these particles tend to settle. This process proceeds faster in a slurry with a high water content. Under Dutch circumstances this counts in particular for sow manure and veal calf manure. On the contrary, cattle slurries contain a considerable amount of solid particles with a tendency to float. The sampling technique required depends also on the size and nature of the particles present. This may be either particles which should be considered as belonging to the product such as hair, feathers and undigested plant fibres or impurities such as stones and debris from the livestock building.

The strategy to be used for sampling is governed by the high degree of inhomogeneity of the manure. Agitation can overcome of course this difficulty but creates in the meanwhile another problem, as during the agitation process hydrogen sulphide is released from the manure. This may create live threatening circumstances for both human beings and animals (Patni and Clarke, 1990), in particular when slurries are stored underneath the houses. Therefore, in general agitation is only applicable in storages outside the livestock buildings and little is known about agitation strategies

and the degree of homogeneity reached (Cumby, 1987).

Taking into account above limitations, the research into sampling methods for manure focused on sampling of manure at the moment of being loaded for transport from the farm. Homogeneity and accessibility are most optimal at that moment. The most important requirement of a sampling method is its accuracy, in terms of bias and reproducibility. Furthermore, aspects like technical reliability, labour requirements, possibilities for automatization, and costs play a role in the selection of methods.

The objective of the investigations described in this paper was to quantify the bias and reproducibility of different sampling methods, differing either in technical aspects or in the sampling protocol, for both slurry and solid manure. Both available and newly developed methods were included in sampling experiments at practical farm level.

Materials and Methods

Farms

The manure was sampled at Dutch commercial farms, located in the south and east of the country. The restriction made was that the origin of the manure was unambiguous in terms of animal type. Differentiation was made for slurry sampling between slurries from pigs, cattle and laying hens. Solid manure originated either from broilers kept on embedded floors or from laying hens kept in batteries with conveyor belts for manure discharge. No more than three loads were sampled at the same farm.

Sampling and experimental design

The investigations were performed in three different experiments. Experiments I and III refer to slurry sampling and were executed in the Autumn 1994 and in the Summer 1996, respectively. Experiment II, dealing with solid manure sampling, was executed in the Spring 1995. In each experiment about 30 loads of slurry or manure were sampled. The accuracy of a number of practical sampling methods was investigated by comparing their results with those obtained with a reference method. The reference method chosen in each of the experiments, represented a more elaborated sampling method with a high accuracy.

For slurry sampling, transport tankers with a volume of approximately 35m³ were used. Samples were taken during or immediately after loading depending on the sampling method. Mixing of the tankers was done by recirculation (experiment I) or by an additional mixer built in the tanker (experiment III). After mixing reference samples were taken in duplicate (Table 1, method K).

Solid manure was loaded in containers (approximately 40m³). Samples were taken from the container top after loading. Reference samples were taken during loading either from the conveyor belt (Table 1, method L) or from the loader (method M). To

obtain a final sample volume of about 1–2 l, subsamples were mixed thoroughly and spread on a clean surface in a layer of about 0.1 m. A grid of 0.1 by 0.1 m was drawn and a randomly selected square was transferred completely into a bag as the final sample for analysis. As for the slurry, reference samples were taken in duplicate.

Sampling methods and techniques

Slurry:

In Figure 1 a schematic impression is given of the experimental set-up for the slurry sampling at each farm. The different locations for the slurry sampling techniques are indicated and depend on the technique used. Slurry sampling techniques used in these investigations are schematically represented in Figure 2 to 7. Figure 2 and 3 refer to slurry sampling techniques which have been available to farmers for some years. In Figures 4, 5 and 6 newly developed slurry sampling techniques are represented. Figure 7 presents a sampling tube creating the possibility to obtain a core sample covering the length of the sampling tube. An extended description of technical details of the sampling techniques is given elsewhere (Hoeksma *et al.*, 1995; Hoeksma *et al.*, 1996).

In Table 1 an overview is given of the investigated sampling methods A to J, the reference methods K to M, the sampling techniques on which the methods are based and their major characteristics. Methods A to G were practical methods applied to slurry, where method K served as the reference method for slurry sampling. Methods A to C are based on an available slurry sampling technique, built in the tanker wall (Figure 2), and differ with respect to taking the sample during or after loading and mixing. Method D uses the available slurry sampling technique with ball valves operating in the loading hose (Figure 3). Method E to G (Figures 4 to 6) include recently developed slurry sampling techniques, all designed to obtain a sample from the loading hose. In each of the methods A to G five subsamples were taken to make a final sample for the laboratory with a volume of about 0.75 l. Method K based on the sampling tube (Figure 7) was used as a reference for the slurry sampling methods.

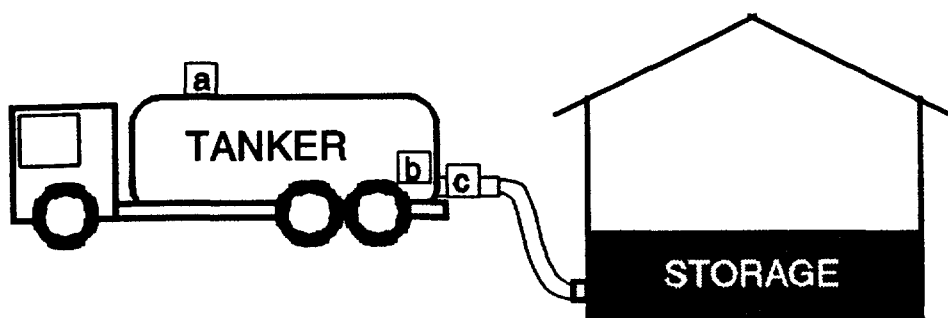


Figure 1. Schematic representation of the experimental set-up of investigations of sampling techniques for slurry. a = sampling of tanker with reference technique, b = sampling in tank wall, c = sampling in the hose during loading.

Table 1. Sampling methods investigated in the 3 experiments, field of application and technical details.

Method	Exp.	Type of manure	Sampling technique	Specifications
A	I	slurry	tanker wall	Subsamples during loading
B	I	slurry	tanker wall	Subsamples after loading
C	III	slurry	tanker wall	Subsamples during loading with mixing in tank
D	I	slurry	ball valves	Subsamples from the hose during loading
E	III	slurry	Side tube	Subsamples from the hose during loading
F	III	slurry	Apple core	Subsamples from the hose during loading
G	III	slurry	bypass	Subsamples from the hose during loading
H	II	solid manure	sampling lance	1 sample
I	II	solid manure	sampling lance	6 samples, reduction of final sample volume after mixing
J	II	solid manure	hand picking	9 samples from the top surface
K	I and III	slurry	sampling tube	Two samples from different locations after mixing the tank
L	II	belt litter	hand picking	10 subsamples during loading, volume reduction of final sample after mixing
M	II	broilers	hand picking	10 subsamples from the loader, volume reduction of final sample after mixing

Solid manure:

In case of solid poultry manure, open containers were used for transport and loading was performed either by conveyer belts or by a loader. For sampling solid poultry manure in such containers a hand operated device is used (Figure 8). A more detailed description and technical aspects of the technique is given elsewhere (Derikx *et al.*, 1995). Sampling method H and I are based on this technique, where method J refer to a hand picking method from the upper surface of the container. Method L and M, used as a reference method for solid poultry manure, both included hand picking during the loading process of the containers.

Chemical analyses

The samples taken were kept at a cool place and transported to the lab within 5 days. In the laboratory samples were stored at 4°C prior to analysis. Samples were analyzed for dry matter (DM), ash, total nitrogen, total phosphorous and total potassium using standard methods for manure (Anonymous, 1996a-g). Results are expressed as g of the element/kg fresh weight.

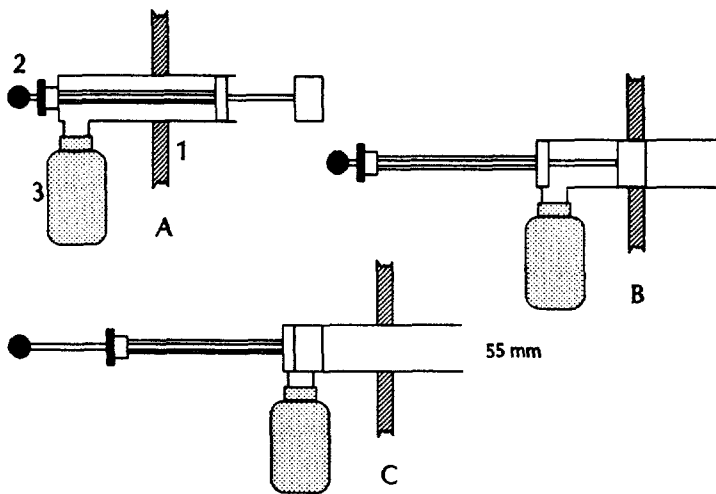


Figure 2. Schematic representation of subsequent positions of slurry sampling device built in tanker wall. A = start of sampling, B = isolation of subsample from slurry tanker, C = discharge of subsample in sample bottle. 1 = tanker wall, 2 = sampling handles, 3 = sample bottle.

Statistical analysis

The accuracy of each sampling method was assessed by estimating the size of two types of error: the systematic error and the random error. They are referred to here as the bias (systematic error) and the reproducibility (random error) of a measuring method (Anonymous, 1993). These error types were determined for each combination of sampling method, manure type and constituent. In the analysis, the distribution of the random errors was treated as being lognormal. This means that random errors of measurements, when expressed on original scale, were assumed to be proportional to their true value.

In the analysis first all data were transformed to their (natural) logarithmic value. For each data set, representing one of the combinations of (sampling method \times manure type \times mineral), the following calculations were performed on the measurements of the loads:

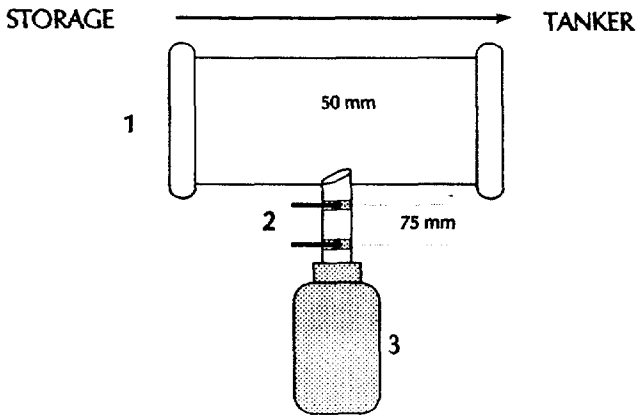


Figure 3. Schematic representation of slurry sampling device built in the hose connecting the transport tank with the slurry storage. 1 = loading hose, 2 = sampling handles of ball valves, 3 = sample bottle.

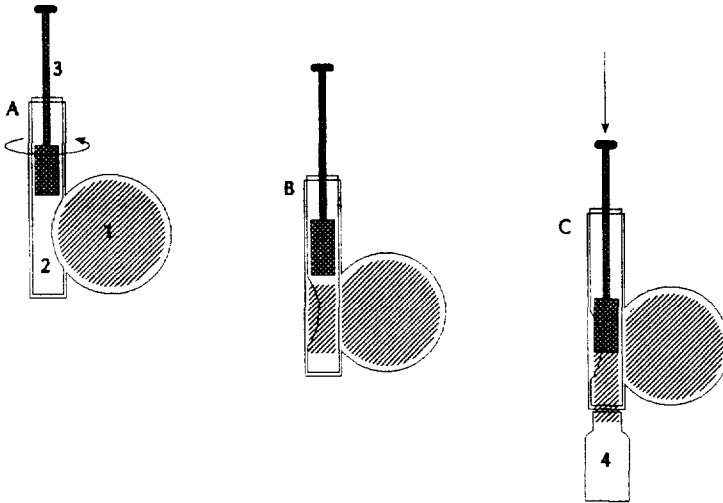


Figure 4. Schematic representation of subsequent positions of slurry sampling device according to the side tube technique built in the hose connecting the transport tank with the slurry storage. A = start of sampling, arrow indicates turning of inner side tube; B = final position of side tube, isolation of subsample from slurry stream; C = discharge of subsample in sample bottle. 1 = cross section of loading hose, 2 = two partly open, closely fitting tubes, 3 = piston, 4 = sample bottle.

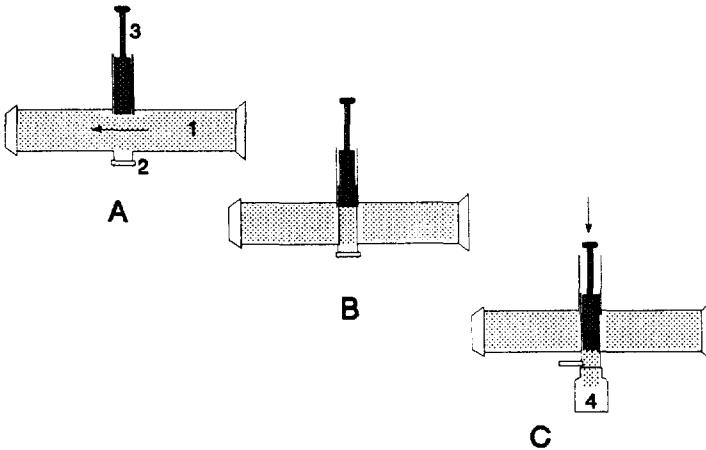


Figure 5. Schematic representation of subsequent positions of slurry sampling device according to the apple core technique built in the hose connecting the transport tank with the slurry storage. A = begin situation, arrow indicates flow direction of manure; B = isolation of subsample from slurry stream by sample tube; C = discharge of subsample into sample bottle. 1 = loading hose, 2 = ball valve, 3 = piston and sample tube, 4 = sample bottle.

$$\Delta_i = x_i - 1/2(r_{i,1} + r_{i,2}) \quad (1)$$

where:

Δ_i the difference for load i with $i = 1 \dots n$, and n the total number of loads; x_i the min-

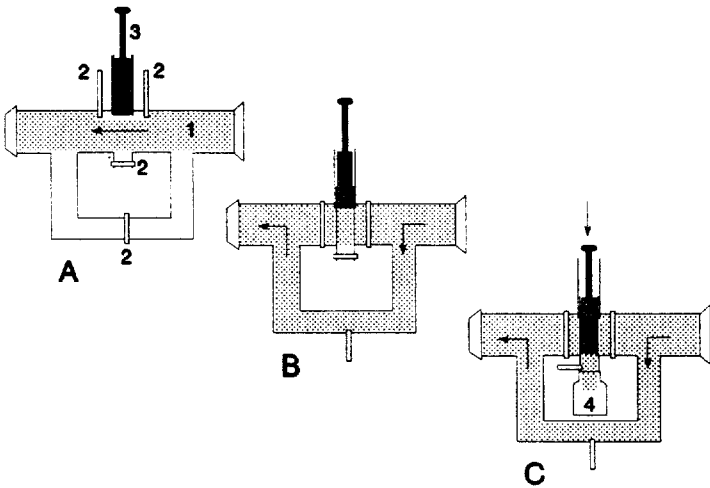


Figure 6. Schematic representation of subsequent positions of slurry sampling device according to the bypass technique built in the hose connecting the transport tank with the slurry storage (arrows indicate flow direction of manure). A = begin situation; B = isolation of subsample from slurry stream by sample tube; C = discharge of subsample into sample bottle. 1 = loading hose, 2 = ball and slide-valves, 3 = piston and sample tube, 4 = sample bottle.

eral content (log scale) of load *i* as determined by the applied sampling method; $r_{i,1/2}$ the mineral content (log scale) for load *i* determined by duplicates 1 or 2 of the reference method.

The bias was estimated as the mean Δ , averaged over *n* loads. The deviation of Δ from 0 was tested by two sided t-tests (Oude Voshaar, 1994) with confidence level $\alpha = 0.05$. Significant deviations indicate that the applied sampling method is biased. For ease of interpretation the systematic differences on log scale were expressed on original scale as the relative deviations (%) from the reference values.

The reproducibility of the method is determined by the variance of the random error. On the premise that for load *i* the random errors of the tested sampling methods were independent from those of the reference method, the following relationship holds:

$$\sigma^2\Delta = \sigma^2M + 1/2\sigma^2R \tag{2}$$

where:

$\sigma^2\Delta$ the variance of the differences Δ_i as defined in equation (1); σ^2M the variance of the random error of sampling method *M*; σ^2R the variance of the random error of the

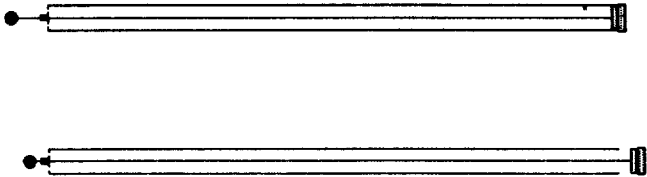


Figure 7. Sampling tube (inner diameter 57 mm, length 2 m) used after mixing as reference method for slurry sampling in the tanker.

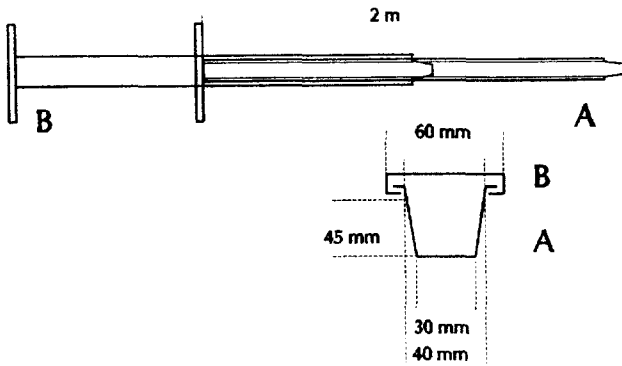


Figure 8. Schematic representation of a sampling lance for sampling of solid chicken manure in the container. A = gutter, B = lid.

reference method. By calculating within each set of data $s^2\Delta$ and s^2R , being the estimators of $\sigma^2\Delta$ and σ^2R , it was possible to derive the estimator s^2M from $s^2\Delta - 1/2s^2R$. For each set, 95%-confidence intervals were constructed from the estimated deviation sM and t -values based on $n-1$ degrees of freedom. The lower and upper boundaries of these 95%-intervals were expressed on log scale. For ease of interpretation these boundaries were converted to percentual deviations on original scale by exponentiating and multiplying with 100, these intervals were called the relative 95%-confidence intervals. These relative intervals are approximately symmetrical when the boundaries have low values ($< 10\%$) but become increasingly asymmetrical at larger boundary values with higher absolute values for the upper boundaries compared to lower boundaries. To facilitate comparison of the reproducibility levels between methods, the mean values of the upper and lower boundaries were tabulated instead of the complete interval.

Calculations were made with help of the statistical software package Genstat 5 (Anonymous, 1989).

Results

In Table 2 the mean composition of the sampled loads is given for each of the three experiments. Within the sampled slurries (Experiment I and III) pig slurry was lowest in DM. Both pig and cattle slurry showed a considerable degree of variation between loads in all measured parameters, with variation coefficients up to 47% for DM and P in the pig slurry of Experiment I. Poultry slurry was more homogeneous in composition. In four out of the 30 poultry slurry loads in experiment I an amount of potassium had been added before filling of the tank (for reasons of field application). Therefore only the composition of 26 loads is given in Table 2 to present an adequate picture of poultry slurry offered for sampling. Calculations related to the accuracy of mineral determination in this slurry however were based on 30 loads except for parameter K and method D ($n = 26$), in which method the unaltered slurry was sampled in the hose and hence differed from the slurry sampled in the tank by the reference method.

Table 2. Mean composition of loads in DM, ash, N, P, K (g/kg fresh manure) as sampled by the reference methods, and variation coefficients (%) of deviations between sampled loads for the different types of manure in each of the three experiments.

Experiment/Manure	DM		Ash		N		P		K	
	mean	VC	mean	VC	mean	VC	mean	VC	mean	VC
Experiment I										
Pig slurry (n = 30)	77	47	25	40	7.2	35	1.5	47	5.9	34
Cattle slurry (n = 30)	90	20	23	22	4.9	20	0.8	25	5.1	16
Poultry slurry (n = 26)*	156	15	55	22	12.0	8	3.4	21	5.0	8
Experiment II										
Belt manure (n = 25)	491	18	124	26	26.8	18	8.5	35	11.4	19
Broiler man. (n = 35)	558	16	132	34	26.6	27	9.3	25	17.0	14
Experiment III										
Pig slurry (n = 30)	86	31	27	30	7.3	27	1.7	29	5.9	32
Pig slurry (n = 26)#	94	18	29	19	7.9	16	1.8	22	6.3	25
Cattle slurry (n = 30)	75	28	20	20	4.7	19	0.6	33	5.5	15
Poultry slurry (n = 30)	146	14	53	25	10.4	10	2.6	23	5.1	20

* without 4 loads with addition of potassium

without 4 loads with diluted sow slurry

Compositions of slurries were alike when compared between Experiment I and III with only some minor differences. For the pig slurry in Experiment III also the composition without 4 loads of sow slurry is given as the results from these loads were not included in the final analysis for reasons explained hereafter. The sampled belt and broiler manures showed to be about similar in composition and the size of variation between loads was the same as observed in poultry slurry.

In Table 3 the estimates for the systematic differences between tested methods and the reference method are given. In case of slurry sampling the differences generally were small with only in a few cases significant deviations from the reference. Differences were lowest for parameter K and generally higher for the solid particles related parameters DM, ash and P. Cattle and poultry slurries showed higher differences than pig slurry, with a notable difference for ash in case of sampling with method G in poultry slurry. There were no indications of different performances of the tanker wall related methods (A,B,C) compared to the hose related methods (D,E,F,G). For the methods applied in belt and broiler manure systematic difference were low with only one significant deviation. All applied methods performed equally well in both manures.

The relative 95%-confidence intervals (based on errors of the random type) in Table 4 describe the reproducibility levels that were achieved by the tested methods. For the slurries, there is a clear difference between the levels reached for parameters N and K on one hand, and those for DM, ash and P on the other, the latter being considerably lower, resulting in high values for the 95% confidence intervals. The parameters that are most closely related to the solid particles (DM, ash and P) are appar-

Table 3. Systematic differences between the tested sampling methods and the reference method expressed as relative deviations (%) from the reference, per type of manure and parameter, with n the number of sampled loads.

Manure	Method	n	DM	Ash	N	P	K
Pig slurry	A	30	+1.0	+2.0	-1.0	+0.1	+0.7
	B	30	-1.6	-1.8	-1.7*	-3.5	-0.1
	C	26	+1.4	+3.8	+0.1	+0.3	-0.5
	D	30	+0.1	-0.1	-1.9	-1.1	-0.9
	E	26	-1.3*	-0.3	-1.1*	-1.1	+0.2
	F	26	-0.5	+0.4	-0.4	-0.4	-0.3
	G	26	+0.8	-2.1*	+0.3	0.0	+0.5
Cattle slurry	A	30	+0.7	+2.5	+0.3	+4.8	-0.2
	B	30	-1.8	-1.3	-0.1	-2.7	+0.5
	C	30	-0.9	+4.2*	+1.4	-6.2*	+0.4
	D	30	-6.0*	-1.4	-0.9	-5.1*	-0.7
	E	30	-3.9*	-1.7	-0.7	-4.0*	+0.9
	F	30	+2.8	+3.7	0.0	+6.6*	-0.2
	G	30	+4.2	+6.6*	+1.6	+2.8	-0.5
Poultry slurry	A	30	-0.4	-0.6	-1.4*	+0.9	+0.6
	B	30	+2.3	+1.5	-0.3	+4.5	-1.1
	C	30	-0.9	-0.5	-0.5	-1.4	+1.7*
	D	26	-2.3	+1.8	-5.1*	-6.0*	-1.6
	E	30	-2.6*	-3.0	-2.5*	-2.0	-1.0
	F	30	+1.5	+6.7	-2.3*	-1.8	-1.6*
	G	30	+5.4*	+15.3*	-2.1	-1.9	-1.8
Belt manure	H	25	-1.1	+1.5	+2.8	+1.4	+2.7
	I	25	0.0	+4.8	0.0	+0.8	+1.5
	J	25	+2.5	+3.4	-1.6	+2.5	+0.5
Broiler manure	H	35	-1.7	-4.1*	-0.5	-0.1	+0.4
	I	35	-1.1	-2.4	-2.8	-0.2	+1.4
	J	35	-1.0	-2.2	0.0	-1.9	-1.8

* significantly differing from 0 ($P < 0.05$)

ently more difficult to sample. The reproducibility levels reached for the parameters of particular interest to the farmer, i.e. P and N, show a wide range of variation between the applied methods. Especially for pig slurry the newly developed methods E, F and G with sampling from the hose showed a much better performance than the conventional methods (A, B and D), with reproducibility levels that were up to a factor three better (method E) in parameter P. It is noted here that in Experiment III 4 loads of sow slurry with extremely low DM contents (around 30 g/kg compared to 94 g/kg in the others) were excluded from this analysis as they showed disproportionately high errors compared to the other loads and distorted the error levels as indicated by the other loads.

For cattle and poultry slurry, discrepancies in reproducibility between the conventional methods and newly developed ones were less distinct but still noticeable. Here

Table 4. The mean value of the lower and upper boundaries of the relative 95%-confidence intervals of the sampling methods per type of manure and parameter.

Manure	Method	DM	Ash	N	P	K
Pig slurry	A	14	9.5	9.5	21	5.5
	B	20.5	19.5	6.5	24	6
	C	17	28	1	13	4
	D	25.5	26.5	11.5	29.5	8.5
	E	5.5	3	4	6.5	4
	F	7	5	4	8	4
	G	10.5	8.5	6	12	4
Cattle slurry	A	13	16	9.5	32	8
	B	10.5	10.5	8	18.5	6
	C	7	9.5	8.5	22	3.5
	D	9	14.5	6.5	20	4
	E	15	11.5	7.5	16	6
	F	15.5	16	7	24	7
	G	22.5	23.5	8.5	19	5
Poultry slurry	A	16.5	25.5	5.5	21.5	7
	B	17.5	27.5	8.5	32.5	8
	C	14	26.5	8.5	14	7.5
	D	21.5	39	12	20	15
	E	7.5	16	6	12	6
	F	22.5	45.5	11	16.5	8
	G	25.5	44.5	11	17.5	9.5
Belt manure	H	28	36	39.5	33	27.5
	I	16.5	25	20	19.5	14
	J	22	26	17	30.5	18.5
Broiler manure	H	22.5	33.5	16.5	17.5	13
	I	10.5	19	14	10.5	9
	J	22.5	33.5	15	18.5	15

again the side tube method E showed a considerable improvement for the important parameter P when compared to the conventional methods, although reproducibility levels remain behind those achieved in pig slurry.

Method I is the best performing method in the solid manures for most of the determined parameters. Compared between both types of manures reproducibility levels were better for the broiler manure.

Discussion

The composition of loads sampled in the three experiments reflect a large degree of variation within each type of manure. Table 2 shows variation coefficients for P and N that range for example in case of pig slurry between 29 and 47%. These results demonstrate the necessity for a individual farm approach for an accurate mineral

management system. Sources of variation are related to farm management (nutrition, feeding system) and sedimentation processes in unstirred stored slurry. The observed inhomogeneity between loads underlines the need for accurate sampling methods when farmers require information on manure composition for monitoring or reasons of manure application. Using estimates from average figures for these purposes inherently may lead to gross errors. Inadequate information on manure composition may reduce the acceptability of manure for application on arable lands. Furthermore, overestimation of the mineral surplus leads to incorrectly high taxes for the farmer, whereas underestimation gives rise to undesirable environmental risks.

The sampling methods were assessed on two aspects, bias and reproducibility. Generally speaking, methods especially varied in terms of reproducibility and behaved more consistently with regard to bias. With a few exceptions left, methods showed no serious levels of bias for both slurry and solid manure.

In terms of the phosphorous content, cattle slurry appeared to be the most troublesome in this respect with significant differences up to 5–6% (Table 3), whereas in pig slurry and the solid manures no significant bias at all was observed. However, when compared to the reproducibility levels reached in cattle slurry (Table 4) the size of this systematic error is relatively moderate.

Performances with regard to reproducibility varied both between techniques and parameters. Reproducibility levels were better for N and K compared to P, DM and ash in all types of slurry. Within the solid manure types reproducibility levels were similar for all parameters. The observed differences in slurry sampling can be attributed to their high water content. Nitrogen and K are mainly present as dissolved components and therefore evenly distributed in the slurry, whereas DM, ash and P are concentrated in the solid particles.

From the results of experiment I and III it can be concluded that the newly developed sampling techniques for slurry are better in reproducibility than those of more traditional design. These new techniques are based on sampling at regular intervals from the hose and are not affected by sedimentation and mixing processes in the tank. The traditional method of sampling from the hose, method D in which ball valves are used, was subject to selective accumulation of heavy particles in the sub-samples. Although more accurate, the newly developed sampling techniques still offer the opportunity to the operator to influence the result by shifting the timing of sampling. Furthermore, almost continuous attention of the operator during the loading process is required. Therefore, a fully automatic version of the sampling device is highly recommended. With the help of the newly developed techniques in future both farmers and authorities can monitor the mineral quantities related to animal slurry more accurately. For both slurry and solid manure sampling it is recommended to continue the development of the sampling techniques described here to improve the practical value of these techniques. As the influence of human activity on the quality of the sample should be minimized automatization of the techniques is unavoidable. In combination with modern communication technology a high degree of control for both the authorities and the farmer come within reach.

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References

- Anonymous, 1989. Genstat 5 Reference Manual. Genstat 5 committee of the statistics department Rothamstead Experimental Station. Clarendon Press, Oxford, 749 pp.
- Anonymous, 1993. International vocabulary of basic and general terms in metrology. Second Edition. International Organization for Standardization, Geneva, 59 pp.
- Anonymous, 1996a. NEN 7430 Manure and derivatives. Sample pre-treatment by homogenizing. Slurries (In Dutch). Dutch Standardization Institute (NNI), Delft, 4 pp.
- Anonymous, 1996b. NEN 7431 Manure and derivatives. Sample pre-treatment by mixing and milling. Manure (In Dutch). Dutch Standardization Institute (NNI), Delft, 4 pp.
- Anonymous, 1996c. NEN 7432 Manure and derivatives. Determination of the contents of dry matter and organic matter. Gravimetric methods (In Dutch). Dutch Standardization Institute (NNI), Delft, 6 pp.
- Anonymous, 1996d. NEN 7433 Manure and derivatives. Sample pre-treatment for the determination of nitrogen, phosphorous, and potassium. Destruction with sulphuric acid, hydrogen peroxide and copper sulphate (In Dutch). Dutch Standardization Institute (NNI), Delft, 6 pp.
- Anonymous, 1996e. NEN 7434 Manure and derivatives. Determination of the content of nitrogen in digest (In Dutch). Dutch Standardization Institute (NNI), Delft, 4 pp.
- Anonymous, 1996f. NEN 7435 Manure and derivatives. Determination of the content of phosphorus in digest (In Dutch). Dutch Standardization Institute (NNI), Delft, 6 pp.
- Anonymous, 1996g. NEN 7436 Manure and derivatives. Determination of the content of potassium in digest (In Dutch). Dutch Standardization Institute (NNI), Delft, 6 pp.
- Cumby, T.R., 1987. A review if slurry aeration. 2. Mixing and foam control. *Journal Agricultural Engineering Research* 36: 157-174.
- Derikx, P.J.L., P. Hoeksma, N.W.M. Ogink & G.W.M. Willems, 1995. Sampling of solid poultry manure in loaded containers (In Dutch). Report 95-26. Institute of Agricultural and Environmental Engineering, Wageningen, 30 pp.
- Flachowsky, G. and A. Hennig, 1990. Composition and digestibility of untreated and chemically treated animal excreta for ruminants - a review. *Biological Wastes* 31: 17-36.
- Hoeksma, P., N.W.M. Ogink, P.J.L. Derikx & G.W.M. Willems, 1995. Sampling of slurry in transport tankers (In Dutch). Report 95-12. Institute of Agricultural and Environmental Engineering, Wageningen, 38 pp.
- Hoeksma, P., N.W.M. Ogink, P.J.L. Derikx and G.W.M. Willems, 1996. Evaluation of prototypes for sampling of slurry in transport tankers (In Dutch). Report 96-52. Institute of Agricultural and Environmental Engineering, Wageningen, 20 pp.
- Japenga, J. & K. Harmsen, 1990. Determination of mass balances and ionic balances in animal manure. *Netherlands Journal of Agricultural Science* 38: 353-367.
- Johnson, G., S. Crawford & S.A. Stark, 1993. Sampling municipal solid waste compost. *Biocycle* 34(12): 61-64.
- Leschber, R., 1986. Sampling, fundamental aspects. In: A Gomez, R. Leschber & P. L'Hermite (Eds.), Sampling Problems for the Chemical Analysis of Sludge, Soils and Plants, Elsevier Applied Science Publishers, London, pp. 2-3.
- Oude Voshaar, J.H., 1994. Statistics in Research (In Dutch). Wageningen Pers, Wageningen, 253 pp.
- Patni, N.K. & S.P. Clarke, 1990. Transient hazardous conditions in animal buildings due to manure gas during slurry mixing. In: C.C. Ross (Ed.), Proceedings of the Sixth International Symposium on Agricultural and Food Processing Waste. Amer. Soc. Agric. Engrs., St. Joseph, Michigan, pp. 449-459.
- Stephenson, A.H., T.A. McCaskey & B.G. Ruffin, 1990. A survey of broiler litter composition and potential value as a nutrient source. *Biological Wastes* 34: 1-9.

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Süss, A., 1988. Chemical analysis of animal slurry. In: H. Vetter, G. Steffens & P. L'Hermite (Eds.), Safe and Efficient Slurry Utilization. Cost project 681. Commission of the European Communities, Brussels, pp. 91–94.