Methods for water quality sampling and load estimation in monitoring of Norwegian agricultural catchments

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Abstract Different sampling procedures are applied to monitor water quality in agricultural catchments in the Nordic countries. The need for comparing monitoring results from the Nordic countries was the incentive for establishing a project aimed at comparing estimates of nutrient losses determined using different sampling strategies. Three different sampling methods were compared in three Norwegian catchments: weekly flow-proportional composite sampling (FPCS), weekly composite sampling with temporally equidistant subsampling every second hour (TECS) and temporally equidistant weekly point sampling (PS). Differences in load estimated between the three tested sampling strategies were smaller for nitrogen than for phosphorus or suspended solids. Point sampling tended to miss some of the peaks in concentrations of phosphorus and suspended solids, particularly during flow events, causing significantly lower load estimates for phosphorus and suspended solids by point sampling compared with composite sampling strategies. Flow-proportional composite sampling gave the most reliable data for event-responsible compounds or when the predictability of peaks was low. Based on this investigation, and similar studies in the other Nordic countries, a flow-proportional sampling strategy is recommended for studies of water quality in agricultural streams.

Keywords Flow; nitrogen; phosphorus; suspended solids

Introduction

In monitoring programmes for water quality studies in agricultural catchments determination of stream concentrations and loads of nitrogen (N), phosphorus (P) and suspended solids (SS) are often included. Because considerable resources are being expended on national and international programmes aimed at reducing nutrient loading of surface water, it is of great importance that the procedures for water sampling are adequate for load estimation of different substances. Data on hydrology (water discharge) and the concentrations of nutrients in water are needed to calculate nutrient loads, but the techniques employed to obtain such data vary between different countries due to divergent methodological traditions and hydrological conditions. There is no particular method that can be generally regarded as optimal. Several case studies have shown that aspects, such as the procedures used to sample water, can have a substantial impact on calculation of nutrient loads (Rekolainen 1989; Rekolainen *et al.* 1991; Kronvang *et al.* 1993; Kronvang and Bruhn 1996). In order to compare results from different monitoring programmes, standardisation is required of both the sampling strategies and the methods of data analysis used to measure nutrient loads in streams. In Europe, this will be particularly important in coming years, when various member nations and associated countries implement the Water Framework Directive (European Commission 2000), for example, in connection with the river basin management plans and ecological classification of waters.

The two principal methods of water collection are point sampling and composite sampling. With the latter strategy, water quality subsamples are collected in proportion to the

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water flow or performed at regular intervals with equal time steps between the subsamples. A flow-proportional composite sample consists of a number of subsamples, each of which is taken when a certain volume of water has passed the monitoring site, so that the subsample collection frequency is a function of the runoff intensity. Point sampling is the most commonly applied strategy in many water-quality monitoring programmes, because it entails lower cost and is simpler to perform than more advanced methodology. This approach is normally used in lakes, rivers and larger streams. Considering monitoring of small streams or nutrient losses from agricultural fields or plots in the Nordic countries, strategies and frequencies of sampling for water quality vary, whereas methods for continuous flow measurements are the same (Øygarden and Botterweg 1998; Vagstad *et al.* 2001).

Because different sampling techniques which was used in the Nordic countries had not been evaluated in the same streams, sampling equipment for comparison of three different sampling procedures was installed in three Norwegian streams with different climate and soils. In this paper these results are presented and compared with results from similar investigations, which have been carried out in the other Nordic countries and elsewhere.

Material and methods

Characteristics of the studied catchments

The catchments of the three streams Kolstadbekken, Timebekken and Vasshaglona are located in southern Norway (Figure 1), and they were chosen because they differ in regard to hydrological regime and types of agricultural production (Table 1). Considering annual runoff, the Kolstadbekken shows a pronounced peak during the snowmelt in April, whereas a more even pattern is seen in the other two streams, with elevated levels in March in the Vasshaglona and in March and October–December in the Timebekken (Figures 2–4). The specific runoff and precipitation are substantially (i.e. more than fourfold) lower in the Kolstadbekken than in the Timebekken and Vasshaglona catchments (Table 1). The climate in Timebekken, and to some extent Vasshaglona, is typical of coastal areas in Southern Norway, with relatively warm winters in combination with high precipitation.

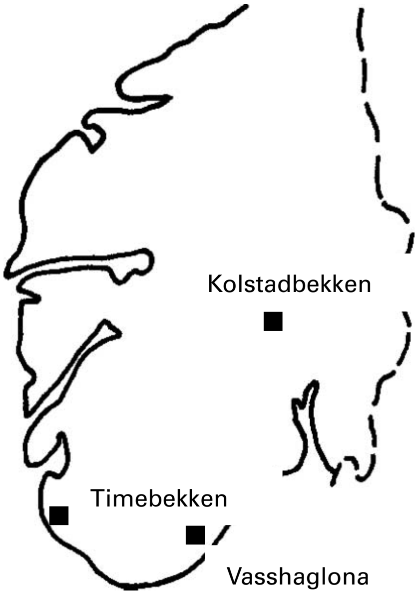

Figure 1 Map of southern Norway showing the locations of the three studied streams

Table 1 General characteristics of the Kolstadbekken, Timebekken and Vasshaglona catchments in 1994

	Kolstadbekken	Timebekken	Vasshaglona
Total area (ha)	308	114	65
Agricultural land (ha)	209 (68%)	97 (85%)	40 (62%)
Cereals/oil plants (ha)	162	22	9.2
Grassland (ha)	30	55	3.7
Vegetables (ha)	0	0	26
Autumn-ploughed area (ha)	84	–	15
Runoff (mm)	346	1394	1262
Annual precipitation (mm)	559	1475	1400
Annual mean temperature (°C)	3.1	7.4	7.1

In 1994, annual runoff and precipitation were normal in the Kolstadbekken but were substantially higher in the Timebekken catchment and higher than the long-term means in the Vasshaglona catchment. The temperatures in all three catchments were normal that year, compared to the long-term means, although the annual mean value was somewhat lower in Kolstadbekken.

Agriculture in the three catchments is dominated by the following: Vasshaglona, 70% vegetable and potato production; Kolstadbekken, 78% spring cereals; Timebekken, 57% grassland (Table 1). Nutrient contributions from point sources (sewage water from scattered dwellings) are almost negligible in all three catchments, and the animal farms are small in Kolstabekken and Vasshaglona (1.0 and 1.4 AU/ha, respectively) but are comparatively larger in Timebekken (2 AU/ha).

Sampling strategies

In 1994, intensive monitoring was carried out in the three streams, and water was collected for quality analysis as follows:

- weekly flow-proportional composite sampling (FPCS);
- weekly composite sampling with temporally equidistant subsampling every second hour (TECS);
- temporally equidistant weekly point sampling (PS).

Composite sampling was carried out by ISCO 3700 sampler.

Water discharge was monitored continuously by Campbell CR 10 logger using a Crump weir in Vasshaglona and Timebekken and a V-notch weir in Kolstadbekken.

Chemical water analysis methods

The water samples were analysed according to accredited methods, described in Norwegian Standards (NS) (http://www.standard.no/imaker.exe) or methods developed by Tecator AB. Nitrogen was determined using Technicon Autoanalyser AAII according to NS 4743 (total-N) and NS 4745 (Nitrate + nitrite-N). Total phosphorus was analysed according to NS 4725, and dissolved phosphate-P was determined according to Tecator methods (ASN 60-05/90 and ASN 111-01/92) (accredited as an alternative to NS 4724) and analysed using a flow injection analyser (FIA). Suspended solids (SS) was determined according to NS 4733.

Load estimation

Daily loads for the two composite sampling strategies were calculated by multiplying the observed daily runoff by the concentration for each weekly composite sampling period. Loads for arbitrary time periods were estimated by summing the daily loads. For the point-sampling

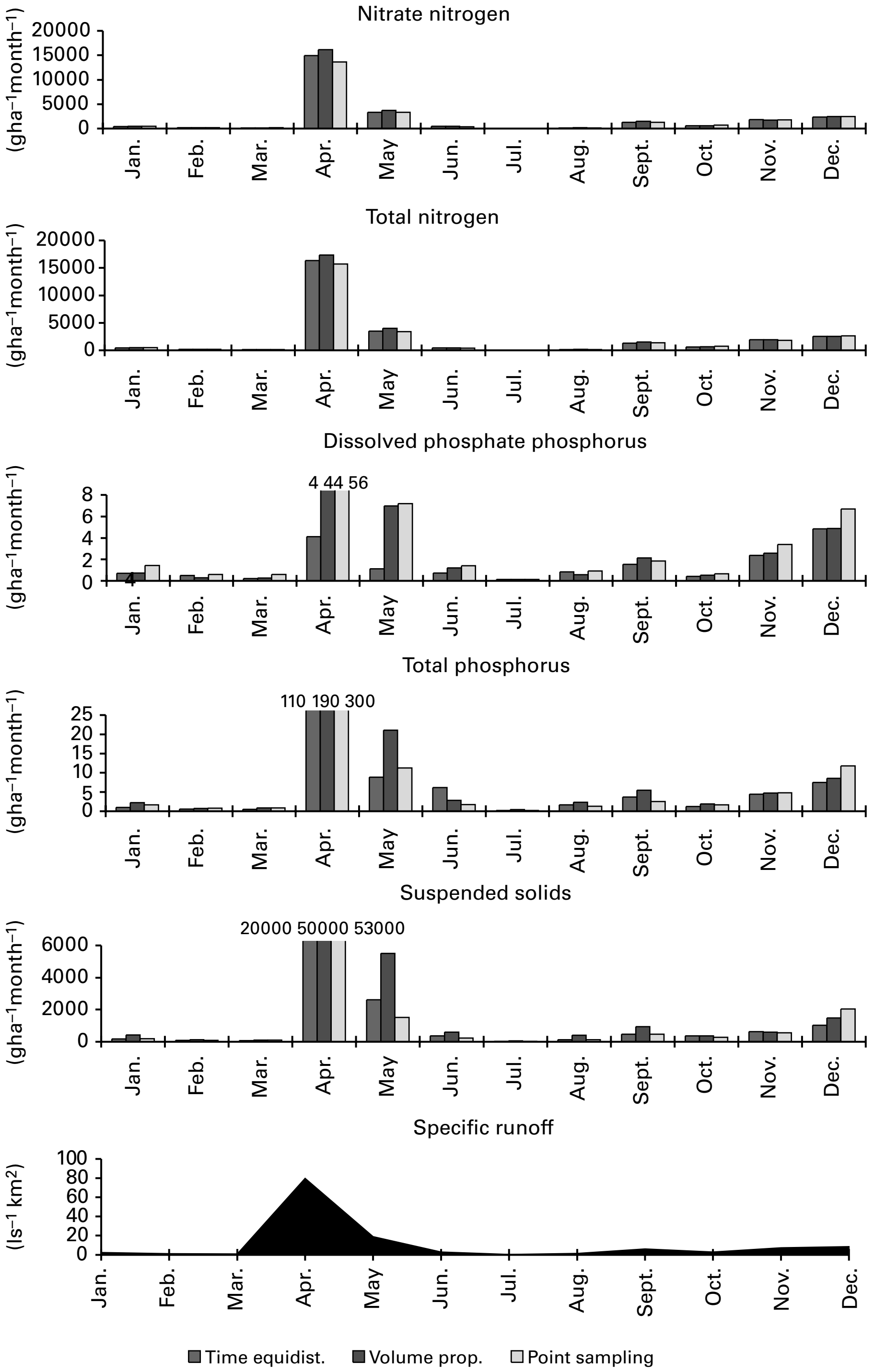

Figure 2 Estimated monthly loads in the Kolstadbekken stream based on the three sampling strategies

strategy, we used linear interpolation to produce a complete series of daily concentration values. Linear interpolation has shown to be more robust and less prone to producing highly imprecise results than more sophisticated methods which attempt to correct for bias in rating relationships fitted to transformed concentration and flow data (Ferguson 1986). Loads for

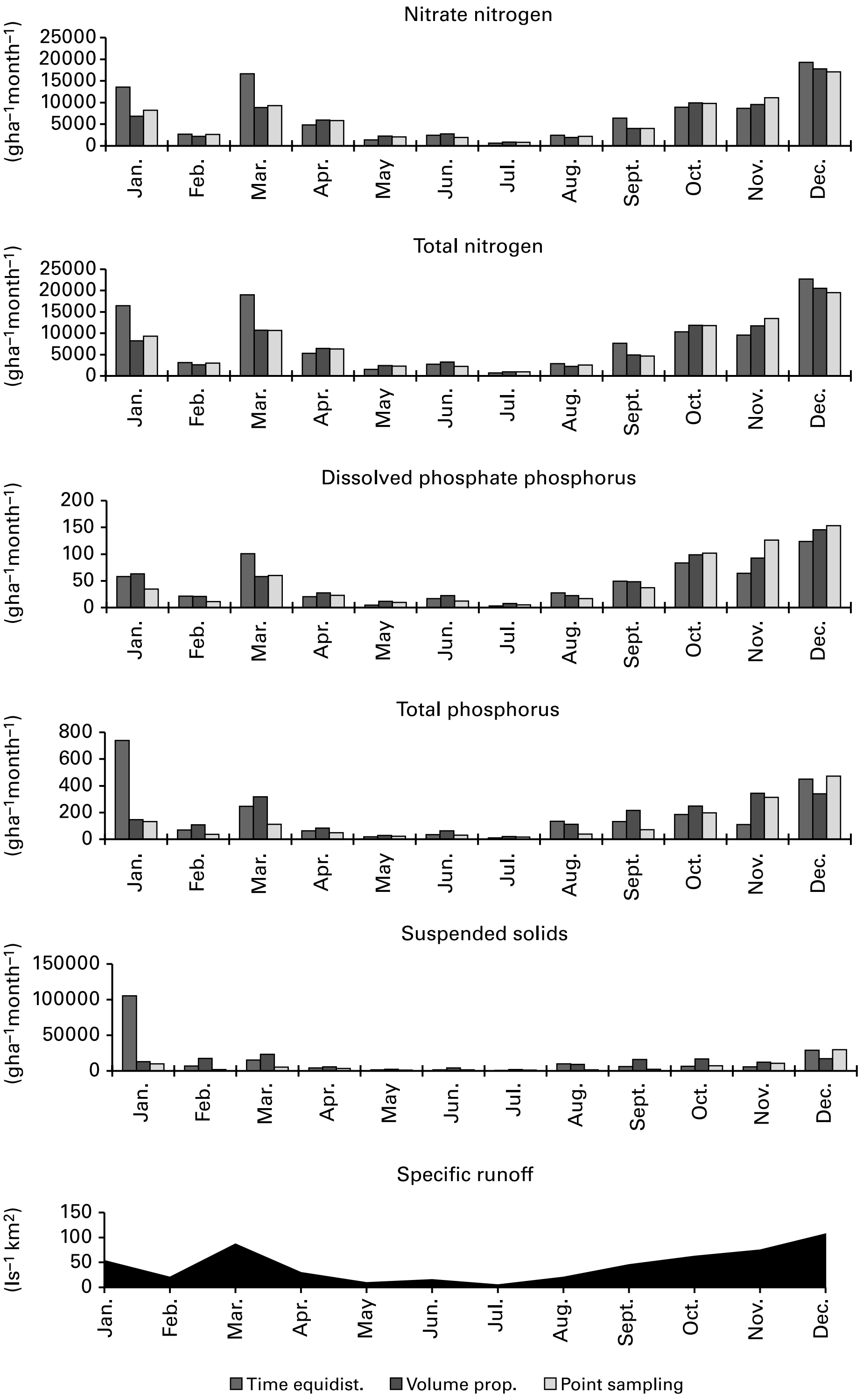

Figure 3 Estimated monthly loads in the Timebekken stream based on the three sampling strategies

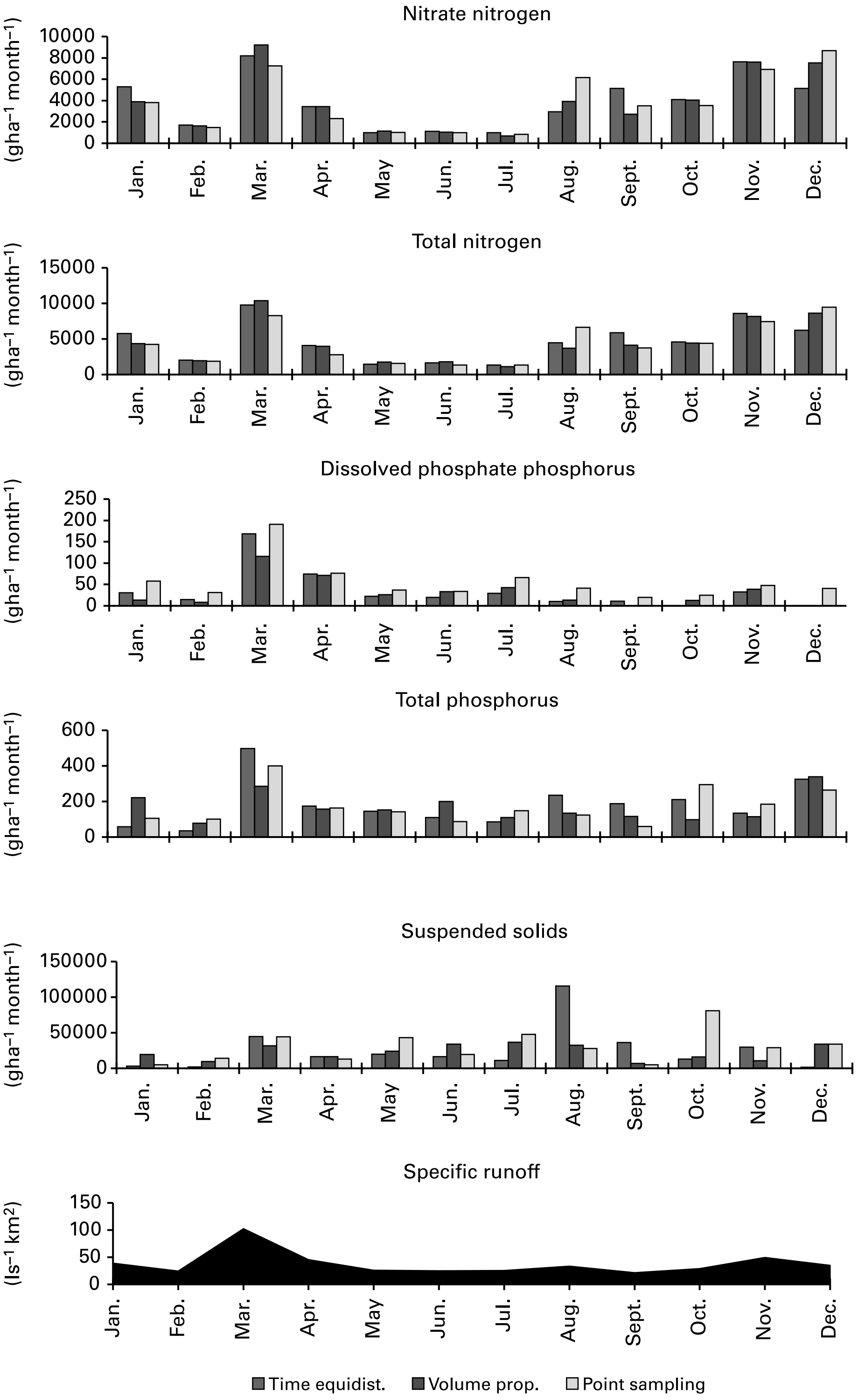

Figure 4 Estimated monthly loads in the Vasshaglona stream based on the three sampling strategies

arbitrary time periods were then estimated by multiplying the daily concentrations by the observed daily runoff data according to Equation (1):

$$\hat{L}_1 = \sum_{j=1}^{N} q_j \hat{c}_j \tag{1}$$

where $\hat{c}_j$ denotes the observed or interpolated daily concentrations, q_j the observed daily runoff and N the number of days in the selected study period (monthly or annual loads in this study).

According to Deelstra (pers. comm.), errors were made in the construction of the Crump weirs in two of the studied catchments (Timebekken and Vasshaglona), and this has influenced the total amount of runoff measured, but it has not biased the flow-proportional sampling.

Results

Load estimates by different sampling strategies

Annual loads of nitrogen and phosphorus occurred predominantly as NO_3-N and particulate phosphorus in all three catchments. The annual loads according to TECS and PS were compared with the loads obtained with the FPCS strategy (Table 2), which were assumed to give estimates closest to the unknown "true" value.

For *nitrogen*, the estimates in the Kolstadbekken stream were almost similar for TECS and PS, but somewhat (10%) higher for FPCS (Table 2). In the Timebekken stream TECS gave the highest estimates of both total-N and NO_3-N (approximately 20% higher than FPCS). FPCS and PS gave lower and relatively similar estimates. In the Vasshaglona stream there was only small differences between the sampling strategies for the nitrogen estimates.

Comparison of load estimates for the *phosphorus* compounds and *suspended solids* was more problematic. The most extreme deviations were found for PO_4-P and SS (Table 2). For example, based on TECS, the estimated annual PO_4-P load in the Kolstadbekken stream was only 27% of the corresponding FPCS estimate. A more thorough examination of the monthly loads showed that the low annual estimates with TECS were chiefly due to the extremely low estimates during the high-flow period in April and May (Figure 2). This is difficult to explain but may have been due to P-transformation processes in the composite samples or to temporary problems with sampling equipment. In this context, it is also interesting that, for PO_4-P, the PS estimate was 29% higher than the FPCS value; this was due to the high concentration values during the first five months of the monitoring. The single concentration peak for Tot-P in the same stream in April explains the high annual load obtained with the PS estimator (Figure 2).

Table 2 Annual loading estimates based on the three sampling strategies (all values in kg ha^{-1})

Catchment	Strategy	NO_3-N	Tot-N	PO_4-P	Tot-P	SS
Kolstadbekken	FPCS	27.5	29.6	0.063	0.237	60.5
	TECS	25.5	27.6	0.017	0.146	26.3
	PS	24.4	27.1	0.081	0.341	58.7
Timebekken	FPCS	73.0	85.9	0.619	2.026	139.8
	TECS	87.9	102.1	0.572	2.192	193.0
	PS	76.3	86.9	0.591	1.493	75.8
Vasshaglona	FPCS	46.9	54.4	0.376	2.008	272.0
	TECS	46.7	55.8	0.413	2.200	309.9
	PS	46.5	53.1	0.668	2.074	363.6

The other extreme deviation between the strategies was found in the Vasshaglona stream, where the annual SS load was 34% higher with PS than with FPCS and the annual PO_4-P load was 78% higher with PS than with FPCS (Table 2). Considering the Timebekken stream, the corresponding SS load according to PS was 54% of the load based on FPCS (Table 2). High TPCS estimates were also noted for Tot-P and SS due to substantial loads in January (Figure 3), which were related to two very high composite sample values in January (Figure 3).

Thinning of the time series of weekly point-sampling data

Load estimates based on concentration data from the weekly point sampling were compared with estimates calculated using time series that were thinned to fortnightly and monthly sampling (Table 3). The results showed that thinning to fortnightly sampling gave load estimates that were similar to those obtained using the weekly sampling values. For example, considering Kolstadbekken, deviations from the weekly estimates were about 3% for total and nitrate nitrogen, but were up to 54% for total phosphorus and 80% for suspended solids. In contrast, fortnightly sampling gave fairly accurate estimates for almost all parameters

Table 3 Loads of nutrients and suspended solids in 1994 in the Kolstadbekken, Timebekken and Vasshaglona streams given as deviation (in %) from values calculated from weekly point samples (100%) after thinning the water quality data to fortnightly and monthly sampling (n.e.: not estimated)

Substance	Sampling frequency	Kolstadbekken	Kolstadbekken[1]	Timebekken	Vasshaglona
Nitrate nitrogen	Fortnightly, odd weeks	−2.9	−0.5	1.1	−1.1
	Fortnightly, even weeks	10.8	2.0	−2.2	−2.2
	Monthly, 1st week	6.9	−4.8	−0.2	n.e.
	Monthly, 2nd week	10.9	2.5	4.0	n.e.
	Monthly, 3rd week	4.4	2.1	4.0	n.e.
	Monthly, 4th week	4.4	0.7	1.0	n.e.
Total nitrogen	Fortnightly, odd weeks	−1.4	−0.8	3.3	1.4
	Fortnightly, even weeks	4.9	2.5	−3.0	−3.0
	Monthly, 1st week	0.1	−5.6	2.1	n.e.
	Monthly, 2nd week	4.6	−3.6	−6.2	n.e.
	Monthly, 3rd week	4.2	4.2	5.0	n.e.
	Monthly, 4th week	−9.1	0.1	1.0	n.e.
Dissolved phosphate-P	Fortnightly, odd weeks	4.6	−1.3	18.4	25.3
	Fortnightly, even weeks	−10.6	−2.4	−7.8	0.1
	Monthly, 1st week	0.6	−1.8	32.4	n.e.
	Monthly, 2nd week	−11.8	0	−13.0	n.e.
	Monthly, 3rd week	18.4	2.1	−6.9	n.e.
	Monthly, 4th week	−20.4	−2.2	4.4	n.e.
Total phosphorus	Fortnightly, odd weeks	17.5	−4.1	11.0	23.2
	Fortnightly, even weeks	−53.6	28.2	0.3	−10.7
	Monthly, 1st week	−55.4	−9.7	28.9	n.e.
	Monthly, 2nd week	−56.2	−2.2	23.3	n.e.
	Monthly, 3rd week	97.5	93.2	−19.0	n.e.
	Monthly, 4th week	−65.9	−1.9	−16.1	n.e.
Suspended solids	Fortnightly, odd weeks	14.0	−11.8	2.8	35.0
	Fortnightly, even weeks	−49.7	80.7	1.8	−30.1
	Monthly, 1st week	−66.8	−20.1	28.1	n.e.
	Monthly, 2nd week	−55.5	−16.3	39.4	n.e.
	Monthly, 3rd week	89.4	167	−40.5	n.e.
	Monthly, 4th week	−59.8	2.6	−37.1	n.e.

[1] April excluded

considered in the Timebekken stream. It was also evident that further thinning of data to a strategy based on samples collected only once a month will have a significant impact on the uncertainty of the annual load estimates. This was particularly noticeable for data on monthly suspended-solid samples collected in the Kolstadbekken stream, which gave up to 67% lower and 90% higher estimates compared to loads with weekly sampling (Table 3). Occasional large deviations were also noted for the other substances, partly due to a tendency towards elevated or single extremely high concentration values during the spring-flow period in April 1994. Excluding this month for both fortnightly and monthly sampling in the Kolstadbekken in most cases resulted in smaller deviations from weekly sampling. This was true particularly for nitrate-N and dissolved phosphate.

Discussion

If, for the sake of simplicity, we assume that the results of FPCS reflect the unknown true load, some common features become apparent. First, with the point-sampling strategy, phosphorus loads were nearly always both underestimated and overestimated, and this agrees with the results of a study of two agricultural lowland streams in Denmark reported by Kronvang and Bruhn (1996). Secondly, both better precision and more stable accuracy were achieved when analysing total nitrogen, as compared to phosphorus and suspended solids, which is in accordance with the findings of Rekolainen (1989) regarding phosphorus and the results reported by Walling and Webb (1981), Richards and Holloway (1987) and Webb *et al.* (1997) concerning suspended sediment yields.

It has previously been shown that event sampling and sampling during high flows are important components of small-scale monitoring programmes (Rekolainen *et al.* 1991; Reinelt and Grimvall 1992), particularly when a substantial part of the annual load occurs during a few rain periods (Grimvall 1996), even though Kronvang and Bruhn (1996) have maintained that stratified sampling (i.e. intensive sampling under certain flow conditions) does not necessarily increase the accuracy and precision compared to regular, temporally equidistant sampling.

To our knowledge, few evaluations in the literature deal with the difference between point and composite sampling strategies. However, Eggestad *et al.* (1994) comment that FPCS is the most accurate method for obtaining what can be regarded as true loads. Comparing weekly PS and FPCS, the cited authors found that the former strategy resulted in highly underestimated loads in all four catchments they studied in Norway. These results concur well with our findings, except that we did not find such a marked discrepancy between loads calculated with PS and FPCS data, and we obtained both highly underestimated and highly overestimated loads.

In conclusion, temporally equidistant sampling obviously leads to considerable bias towards low-flow conditions, and this places limitations on calculations of loading estimates for sediment and nutrient compounds, which tend to depend on discharge.

Low sampling frequency can introduce serious errors in load estimates for streams and small rivers (Rekolainen *et al.* 1991; Reinelt and Grimvall 1992; Kronvang and Bruhn 1996). However, this problem is less pronounced for large rivers, as has been demonstrated by Tonderski *et al.* (1995), who studied large rivers in Eastern and Western Europe, and by Grimvall (1996), who performed a similar investigation of data on Swedish rivers. The results of the present study and the findings of Ulén (2002), clearly show that a relatively high sampling frequency is necessary to ensure unbiased results. The risk of underestimation, and to some extent lower precision, increases rapidly with decreasing sampling frequency (Rekolainen 1989), and this was very obvious when comparing the estimates based on fortnightly and monthly sampling. The various substances analysed also differed as follows: results for phosphorus and suspended solids were highly affected by a lowered sampling frequency, whereas nitrogen estimates were less sensitive. Essentially equivalent estimates were obtained using fortnightly and weekly data from the Norwegian catchments, and fortnightly sampling has been found to be sufficient in Denmark (Kronvang and Bruhn 1996). Unfortunately, nitrogen was not included in the Swedish study.

Although composite water sampling provides better estimates of nutrient loads than point sampling does, chemical changes may occur in composite samples that are stored at a measuring station for a week or more. EU has established guidelines on preservation and handling of samples (European Standard ISO 5667-3) and studies have shown that it is very important to keep samples cool in summer and protect them from freezing in winter in order to prevent chemical alteration during storage (Deelstra *et al.* 1998; Turtola 1989; Kotlash and Chessman 1998). Turtola (1989) found that storage of water samples for two weeks did not cause significant changes in total and phosphate phosphorus, and addition of sulphuric acid did not safeguard concentrations. Turtola also observed that two weeks of storage led to only an insignificant increase in total and nitrate nitrogen, but caused a drastic decrease in ammonium N. Furthermore, Braskerud (1998) and Kotlash and Chessman (1998) have published similar results regarding ammonium N, thus composite water sampling is not suitable for this compound, which should instead be investigated by point sampling and immediate analysis. Composite sampling can be recommended for monitoring total N, nitrate N, total P, and phosphate P.

Another potential problem is that the volume of composite samples can decrease during the sampling period. Moreover, sedimentation and growth of algae have been reported to interfere with the sampling equipment used in Norway.

Sampling of suspended particles has to be isokinetic to be representative, which means that the particles entering the sampler must have the same velocity as the running water. This is due to the fact that particles and water move as two different systems, and the degree of coupling between water and sediments depends on particle size. The nature of the sites where running water is sampled also influences the type and amount of sediment in the water samples (Bogen 1998). However, silt and clay are usually the dominating grain fractions in agricultural streams, which tend to follow the flow of the water. The sampling systems used in agricultural catchments in the Nordic countries have not been designed to ensure isokinetic sampling, but the water velocity in streams is often relatively low compared to that in smaller rivers.

Conclusions

The objective of the case study we performed in Norway was to analyse time series of nutrient concentrations, determined in three streams in agriculturally dominated areas by use of three different sampling strategies. Our findings show that, during some seasons in the different catchments, the three strategies gave load estimates that were fairly similar for nitrogen but varied considerably for phosphorus and, in particular, suspended solids.

In greater detail, our results can be summarised as follows:

- Differences in load estimates between the three tested sampling strategies were smaller for nitrogen than for phosphorus and suspended solids.
- Point sampling tended to miss some of the peaks in concentrations of phosphorus and suspended solids, particularly during flow events.
- The results based on fortnightly and weekly point sampling were fairly similar.
- Comparing load estimates calculated using data from weekly point sampling data and the two composite sampling strategies, the results for nitrogen were relatively similar, whereas the weekly sampling gave significantly lower values for phosphorus, as well as for suspended solids.

- In most cases, the load estimates for nitrogen that were based on temporally equidistant composite sampling agreed well with the estimates calculated using data from flow-proportional composite sampling; a corresponding comparison for phosphorus and suspended solids gave more ambiguous results that at times showed temporary, unacceptably high deviations.

In light of these findings, it is recommended that data be obtained by flow-proportional composite sampling for calculations to be done for event-responsive compounds or when the predictability of peaks is low. The differences in results between the catchments and between the nutrient compounds show that sets of data should be collected before making a final decision regarding sampling strategy and frequency. This recommendation is in line with conclusions by Rekolainen *et al.* (1991) and Preston *et al*. (1989).

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