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THE EFFECT OF SAMPLING FREQUENCY ON THE ACCURACY OF NITROGEN LOAD ESTIMATES FROM A DRAINED LOBLOLLY PINE PLANTATION IN EASTERN NORTH CAROLINA

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ABSTRACT Nutrient loading in drainage outflow is estimated from measured flows and nutrient concentrations in the drainage water. The loading function is ideally continuous, representing the product of continuously measured outflows and nutrient concentrations in drainage water. However, loading is often estimated as the product of continuously measured outflow and nutrient concentrations measured at less frequent time intervals (weekly, monthly etc.). In this study we investigate the effects of sampling frequency and method on estimating the loading of nitrate nitrogen (NO_3-N) and total Kjeldahl nitrogen (TKN) from a drained loblolly pine plantation in Eastern North Carolina. The loading of NO_3-N and TKN computed from continuous flows and daily concentrations were compared to loadings computed from weekly, bi-weekly, and monthly discrete samples. In this study, the NO_3-N concentrations had a greater range than those of the TKN concentrations and had a more distinct relationship between concentrations and flow; consequently, the load estimation methods were less precise and more biased when estimating NO_3-N than when estimating TKN. If NO_3-N loads from small drained forest watersheds are going to be calculated from discrete samples, the sample interval should be well less than 7 days, probably in the range of 1 to 3 days. Load estimates for TKN may be acceptable from samples collected every 7 days.
Keywords: Nitrogen Loading; Water Quality; Sampling strategy; Uncertainty; Forestry

INTRODUCTION Quantifying nitrogen load from watersheds is an important part of assessing the water quality status of watersheds and the impact of landuse on that status. Ideally N loading is calculated from continuously measured outflows and nutrient concentrations in frequently (daily or hourly) collected samples of the drainage water. The high cost of sample analyses makes frequent sample collection and analysis prohibitively expensive for many water quality monitoring programs; consequently, most program rely on less frequent samplings (weekly, biweekly, or monthly) which makes load determination more of an exercise of load estimation. Various methods have been developed to estimate N loads from less frequently collected samples and these methods have been evaluated for a variety of watersheds (e.g. Phillips et al., 1999). This study evaluates the ability of three of these methods to estimation annual nitrate nitrogen (NO_3-

N) and total Kjeldahl nitrogen (TKN) loads from drained loblolly pine plantation watershed.

METHODS Nitrogen concentrations from water samples collected frequently (every eight hours) were used with continuously measured flow data to calculate annual nitrogen load from a drained loblolly pine plantation watershed for three years. The time series of flow and concentration data was then numerically sampled to simulate discrete sampling at lower frequencies. Three different algorithms were used with the simulated discrete sampling data to estimate annual N load for each year. The N load estimates were compared to N loads calculated with high frequency sampling to determine the accuracy and precision of the estimates for different sampling rates at lower frequencies.

Study site and data collection The study watershed is a 24 ha loblolly pine plantation located in the lower coastal plain of North Carolina, U.S. (34^o 48' N, 76^o 42' W). Soils at the site are deep, fine sandy loams of the Deloss series (Fine-loamy, mixed, semiactive, thermic Typic Umbraquults) which overlay sandy marine terraces. Due to flat topography and low elevation (<3 m), a parallel ditch system (1.2 to 1.5 m deep and spaced 100 m apart) was installed in the early 1970s to improve drainage. The loblolly pine trees were planted in 1974 at a density of 2100 trees ha^{-1}. The site was thinned to 988 trees ha^{-1}in 1981 and again thinned to 370 trees ha^{-1} in late 1988 when the pine was about 14 years old. The last thinning was followed by an application of nitrogen fertilization (195 kg Urea-N ha^{-1}) in 1989. Flash-board riser structures with 120° V-notch weirs were installed at the watershed outlet in 1988. Water stage upstream of outlet v-notch weirs was recorded continuously with water level recorders equipped with dataloggers. Rainfall was measured near the outlet with tipping bucket recorders and backup manual rain gauges. Other climatological data including air temperature, relative humidity, wind speed and direction, solar and net radiation were measured by an on-site weather station. Monitoring has continued at the site until the present. The reader is referred to McCarthy *et al*. (1991) and Amatya *et al*. (1998, 2001) for a detailed description of the study site and experimental procedure. The watershed is referred to as the Carteret D1 watershed

Water samples for the study were collected at the outlet by an automated ISCO sampler. During the period from September 1991 through September 1994, samples were collected every 2 hours with four samples composited into one bottle. Concentrations of nitrate nitrogen (NO_3-N) and total Kjeldahl nitrogen (TKN) determined for each bottle, therefore, represented those for an eight period.

Data analysis A time series of flow and concentration data was compiled from the three year data set collected at the field site. Since the numerical sampling procedure required an hourly data set of flow and concentration, the hourly values for concentration were calculated by linear interpolation between the concentration values measured every eight hours. The data were divided into three one year periods starting 1 September and ending 31 August. The numerical sampling procedure was used for each year of data and the annual load estimates for each of the load estimation algorithms were compared to the load calculated from the hourly flow and concentration data.

The procedure for simulating discrete sampling considers that there are many possible sampling dates and times for a particular sampling interval. If sampling occurred once a week, the samples could just as possibly be collected at one set day and time (say

Tuesday at 3:00 pm) as another set day and time (say Friday at 9:00 am). Differences in the set days and times for sample collection will result in differences in N load estimated for a given load estimation algorithm; therefore, a set of estimated N loads will be determined for a particular sampling interval.

Distributions of the sets of calculated loads for each estimator were used to express uncertainty in terms of bias and precision. Precision was computed as the 90% confidence interval between the 5th and 95th percentile of the set of estimated loads. Bias was computed as the average of the set of estimated loads, although 50th percentile of the set could just as easily been used.

Load estimation algorithms Many algorithms for estimating pollutant loads from infrequently collected concentration data have been developed and tested. Birgand et al. (2010) evaluated the accuracy and precision of eight algorithms to estimate NO_3-N load from nine watersheds in Brittany, France. All eight of these methods were used with the data set from the pine plantation, but only the three better performing methods are reported in this paper. These methods are shown in Table 1.

Table 1. Load estimation algorithms used, tested and presented in this study.

Method	Description (and source)	Equation
M3	Constant concentration for the period before sampling (Meybeck et al. 1994)	$Load = K\sum_{i=1}^{n} C_i \overline{Q_{i,i-1}}$
M5	Product of annual flow volume by the flow weighted average of the concentration (for the times of sampling) (Littlewood, 1992)	$Load = KV \dfrac{\sum_{i=1}^{n} C_i Q_i}{\sum_{i=1}^{n} Q_i}$
M6	Linear interpolation of concentrations times the daily flow rate for each day (Moatar and Meybeck, 2005)	$Load = K\sum_{j=1}^{365} C_j^{\text{int}} Q_j$

K = Conversion factor to adjust for units and sampling intervals (changes with method)

C_i = Concentration measured at the time of the i^{th} sample (mg/L)

Q_i = Flow rate measured at the time of the i^{th} sample (m^3/s)

n = Number of samples

C_{int} = Linearly interpolated concentration value between two consecutive samples

V = Annual cumulative volume (calculated from continuous data) (m^3)

$\overline{Q_{i,i-1}}$ = Average flow rate between the i^{th} and $(i-1)^{th}$ sample

RESULTS Rainfall at Carteret D1 for the three years was 1577 mm for 91-92, 1398 for 92-93, and 1548 for 93-94. The mean rainfall at the site for the period between 1988 and 2008 was 1525; therefore, the rainfall for the three years was within 10% of the average.

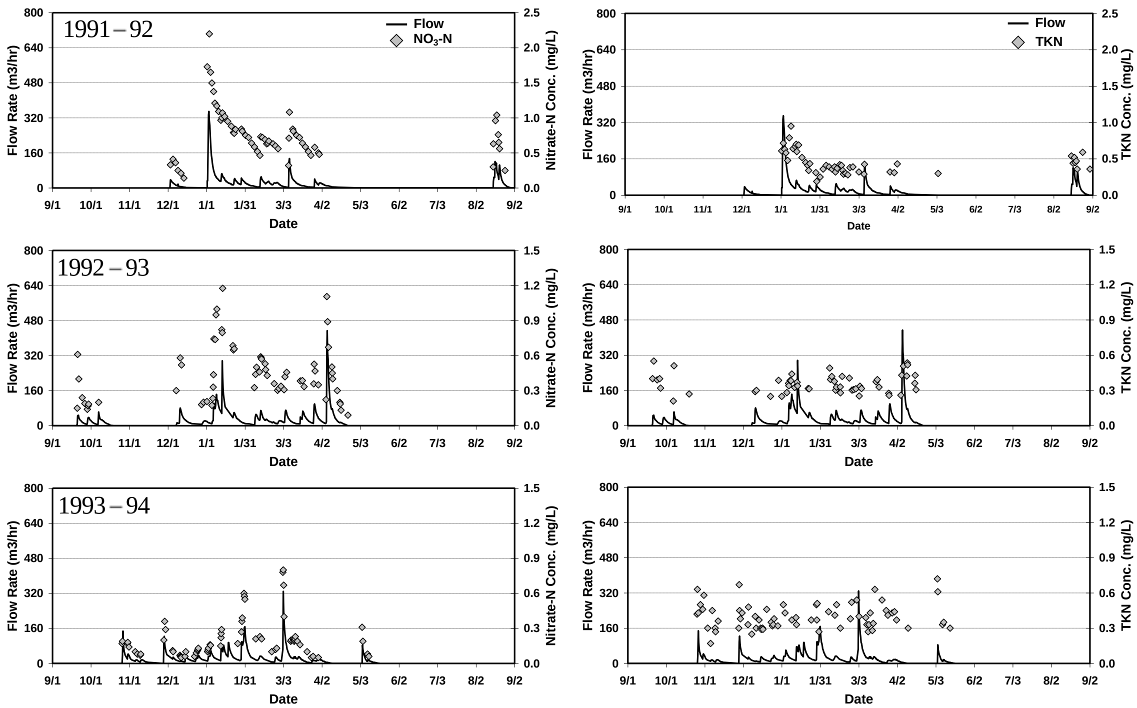

Figure 1. Measured flow and N concentrations for three years at the Carteret D1 site.

Flow and concentration Flow rates from the site ranged from 0 to 433 m³/hr (Figure 1). As is typical for forested watersheds in eastern North Carolina, most of the flow occurred during the winter and early spring months (December – April) and flow rates were 0 for most of the time during the summer months (June – August).

Nitrate N concentrations ranged from 0.05 to 2.20 mg/L and varied with the flow rates. In general, NO_3-N concentrations were higher during high flow rates and lower during low flow rates. That is to say that high flow rates had a concentrating effect on NO_3-N (e.g. Webb and Walling, 1985). This is in contrast to most of the watersheds in the study by Birgand et al. (2010) where high flow rates had a diluting effect on NO_3-N concentrations. NO_3-N concentrations were higher and had a greater range (0.10 – 2.20 mg/L) in first year of the data set compared to the other years.

The range of concentrations for TKN (ranging from 0.14 to 0.95 mg/L) was less than for NO_3-N. While there were some periods where TKN concentrations were more concentrated during high flow periods, a concentration effect was not as evident or pronounced as for NO_3-N. TKN concentrations were higher and had a greater range (0.14 – 0.95 mg/L) in the first year of the data set compared to the other years.

Load estimates The bias and precision of the load estimates for each method are shown on vertical histograms for different sampling rates (Figures 2-4). Lines representing the 5th and 95th percentiles of the estimated loads are shown to display precision while lines representing average and median values are shown to display bias. All of the estimation methods induced bias when estimating NO_3-N loads for the first year of the data set (1991-92). On average, all of the methods estimated loads that were lower than the calculated load. The bias for the M5 method was not as great as for M3 and M6; however, the precision of the M5 was not as good as the precision for M3 and M6. Using the 14-day sampling interval for example, the average load estimated by M5 was 11%

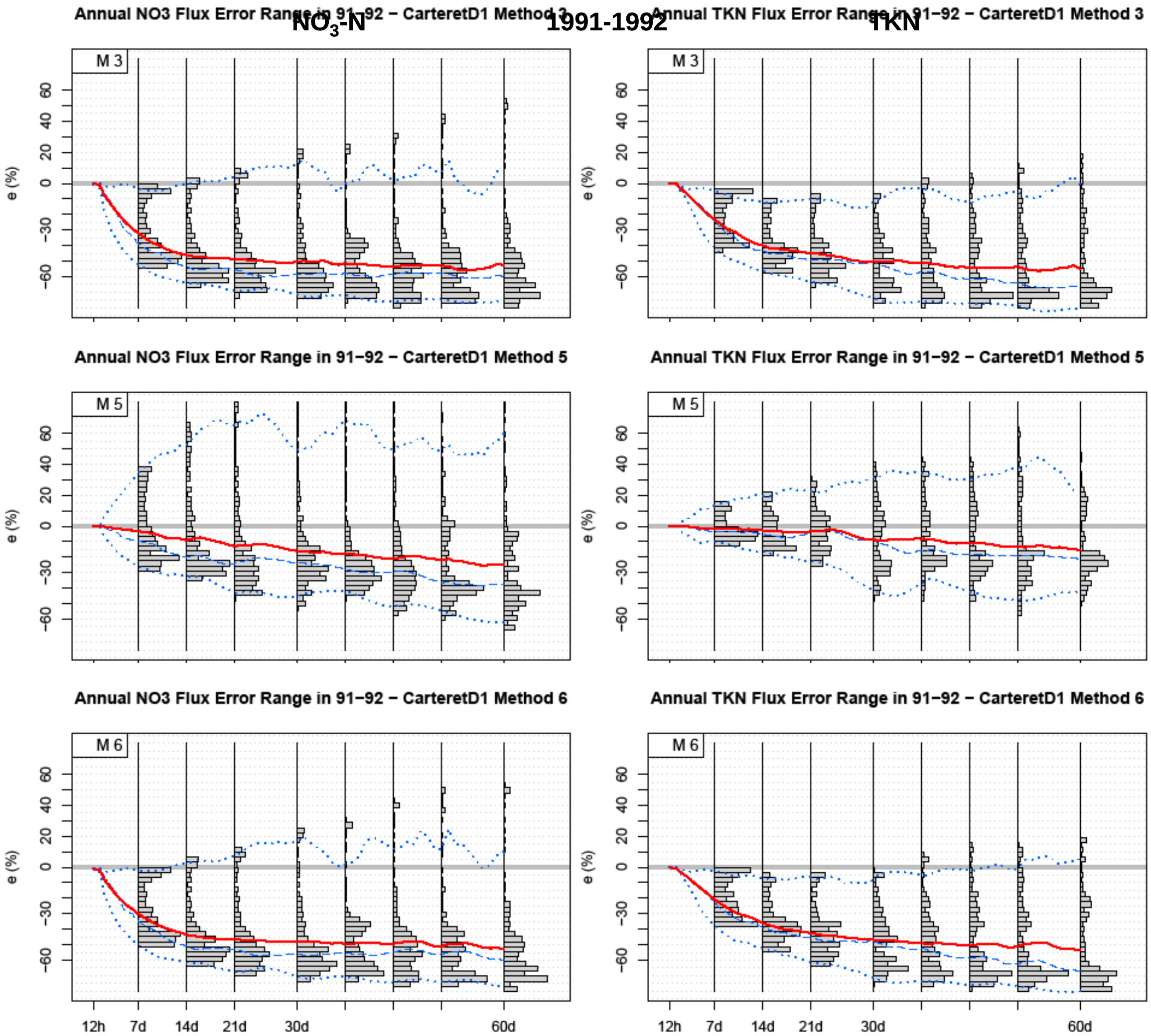

Figure 2. Bias and precision of annual N load estimates for 1991-92 by three different load estimate methods.

below the reference load, while the average loads estimated by M3 and M6 were 37% and 43%, respectively, below the calculated load (Figure 2). The span between the 5th and 95th percentiles of the estimates for M5 was 85 percentage points, while only 71 and 62 percentage points for estimate for M3 and M6 respectively.

While all of the estimation methods also induced a negative bias when estimating TKN loads for 1991-92, the bias for the M5 method (e.g. -4% for 14-day sampling interval) was much improved compared to NO_3-N load estimates (Figure 2). The precision of the M5 load estimates for TKN (e.g. span of 38 percentage points for 14-day sampling interval) was also improved over the load estimates for NO_3-N. The bias of the M3 and M6 TKN load estimates were not much different than those for NO_3-N, but the precisions for these methods were improved.

The bias and precision of the NO_3-N load estimates for the different methods were mostly improved for the last two years of the data set. Bias of the load estimates for the

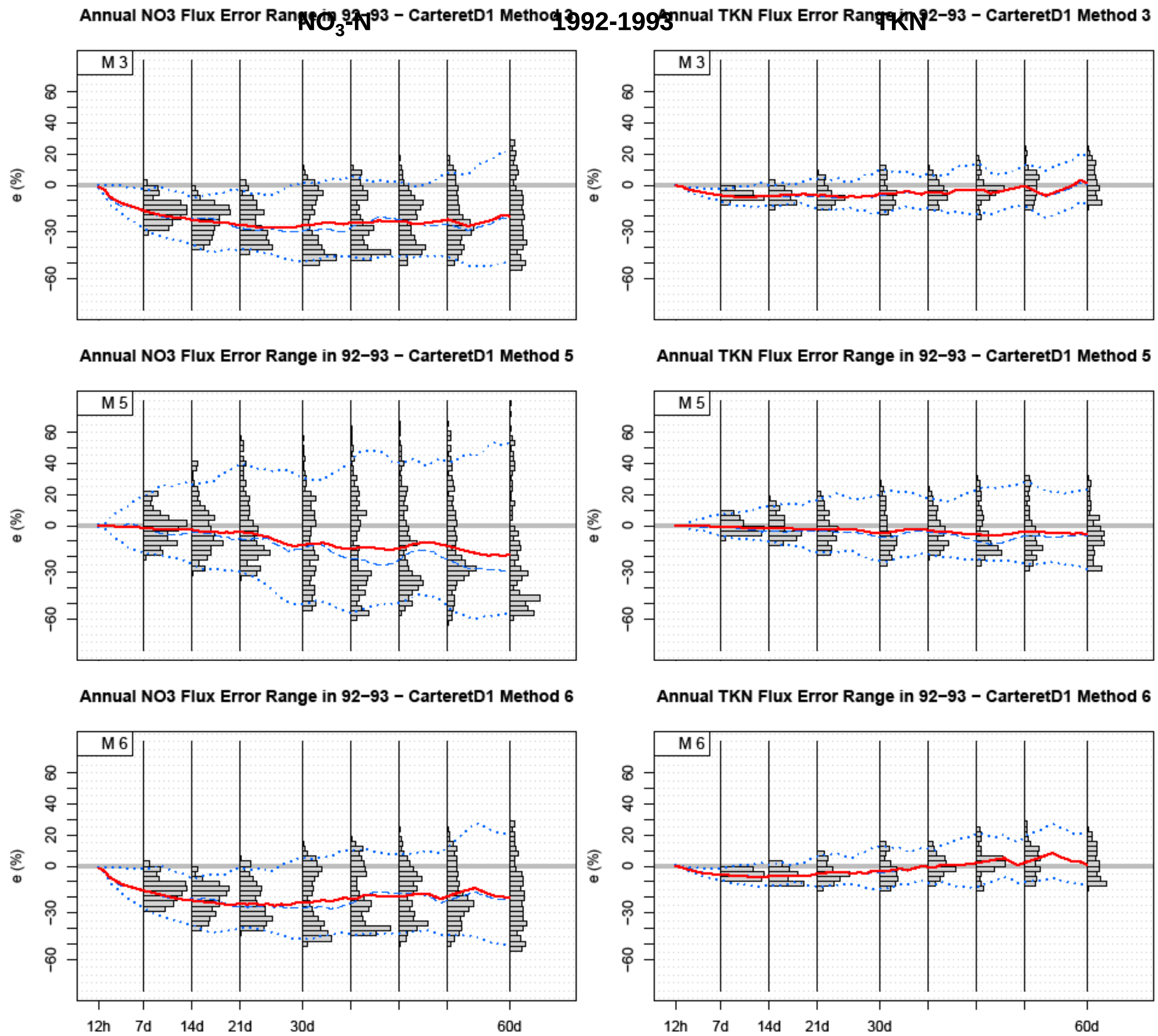

Figure 3. Bias and precision of annual N load estimates for 1992-93 by three different load estimate methods.

M3 and M6 methods were lower for 92-93 and 93-94 than for 91-92 as were the spans between the 5th and 95th percentiles of the M3 and M6 estimates (Figures 3 and 4). Bias of the load estimates for the M5 methods were less for 92-93 than for 91-92, but were not much different than 91-91 for 93-94. The spans between the 5th and 95th percentiles of the M5 estimates were also lower for 92-93 than for 91-92, but were not much different than 91-91 for 93-94. As with the first year the M5 method induced less bias than the M3 and M6 methods for the last two years, and the M5 method was also less precise than the M3 and M6 methods.

All of the bias and precisions of the TKN load estimates for all of the methods were improved for the last two years of the data set (Figures 3 and 4). Bias of the load estimates for all of the methods were less than 10% for 92-93 and less than 5% for 93-94 for sampling intervals up to 30 days. For methods M3 and M6, spans between the 5th and 95th percentiles of the TKN load estimates were less than 30 percentage point for 92-93 and less than 20 percentage points for 93-94 for sampling intervals up to 30 days. As

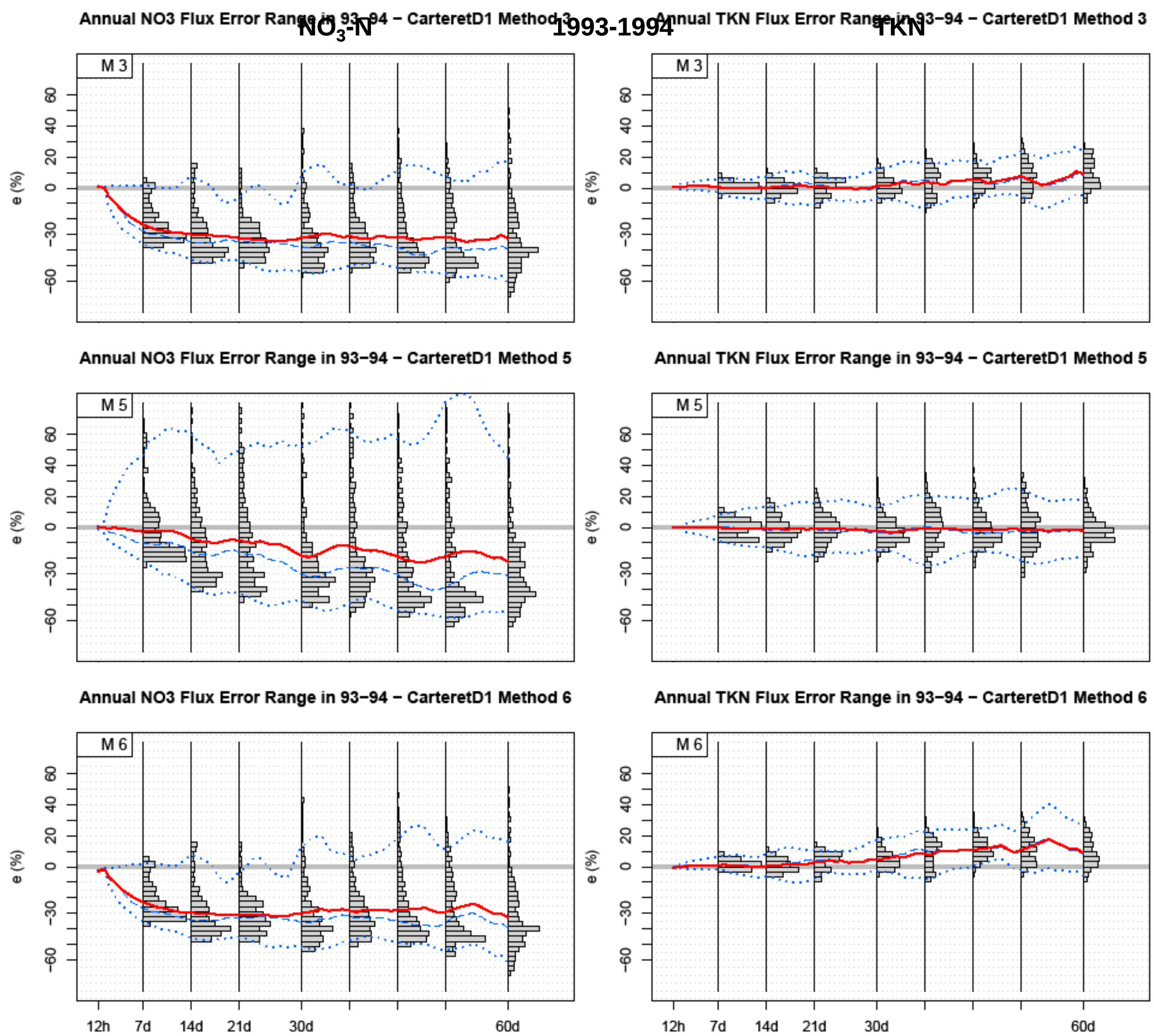

Figure 4. Bias and precision of annual N load estimates for 1993-94 by three different load estimate methods.

with the first year, the M5 method induced less bias than the M3 and M6 methods for the last two years, and the M5 method was also less precise than the M3 and M6 methods.

DISCUSSION To determine whether or not a load estimation method was acceptable for a particular sampling frequency, we arbitrarily chose the following acceptability criteria: the 5th and 95th percentile values will both fall with the range of $\pm 20\%$. This is to say that the method has a 90% chance of estimating a value within ± 20% of the actual value. All of the methods induced unacceptable annual NO_3-N load estimates for all sampling intervals except for the 7 day interval in 92-93 for M3 and M5 (Table 2). In the case of M3, the precision was good, but the bias of the method resulted in a better than 95% chance that M3 would under estimate the NO_3-N load. For the M5 estimate the bias was only -1%, but there was a 10% chance that the estimate would be greater than 19% higher or less than 17% lower. The number of acceptable estimates was greater for the TKN estimates. While only two conditions, 7 and 14 days for M5, were acceptable in 1991-92, nearly all of the combinations of methods and intervals were acceptable in 1992-93 and 1993-94.

Table 2. Summary of bias and precision of annual N load estimates for different sampling intervals for three different load estimate methods

NO_3-N		1991 - 1992			1992 - 1993			1993 - 1994		
Conc. Range		0.10 – 2.20 mg/L			0.09 – 1.18 mg/L			0.05 – 0.80 mg/L		
Method	Interval	Bias	Prec		Bias	Prec		Bias	Prec	
		e_{avg}	e_5	e_{95}	e_{avg}	e_5	e_{95}	e_{avg}	e_5	e_{95}
M3	7 d	-28	+2	-52	-10	-2	-20	-21	+6	-36
	14 d	-37	+10	-61	-18	-1	-36	-27	+14	-45
	21 d	-42	+15	-65	-22	+5	-40	-28	+3	-45
	30 d	-44	+20	-68	-22	+13	-48	-28	+16	-51
M5	7 d	-5	+35	-30	-1	+19	-17	-4	+55	-25
	14 d	-11	+50	-35	-2	+28	-24	-10	+55	-42
	21 d	-16	+55	-45	-4	+39	-29	-9	+52	-47
	30 d	-20	+45	-47	-12	+32	-50	-21	+52	-56
M6	7 d	-30	-2	-49	-15	-2	-27	-22	+2	-35
	14 d	-43	+2	-60	-21	-6	-36	-30	+7	-45
	21 d	-46	+7	-67	-23	-1	-40	-31	-3	-45
	30 d	-48	+15	-70	-23	+6	-47	-30	+10	-50

TKN		1991 - 1992			1992 - 1993			1993 - 1994		
Conc. Range		0.14 – 0.95 mg/L			0.21 – 0.55 mg/L			0.17 – 0.72 mg/L		
Method	Interval	Bias	Prec		Bias	Prec		Bias	Prec	
		e_{avg}	e_5	e_{95}	e_{avg}	e_5	e_{95}	e_{avg}	e_5	e_{95}
M3	7 d	-23	-4	-31	-7	-2	-11	+1	+5	-3
	14 d	-39	-12	-55	-7	0	-15	0	+7	-6
	21 d	-43	-10	-62	-6	+5	-15	+1	+7	-10
	30 d	-50	-12	-73	-6	+9	-18	+2	+12	-7
M5	7 d	3	+15	-10	-1	+7	-6	0	+8	-8
	14 d	4	+20	-18	-2	+12	-11	-1	+13	-13
	21 d	4	+23	-22	-3	+17	-16	-1	+15	-17
	30 d	8	+32	-40	-5	+20	-22	-2	+14	-15
M6	7 d	-20	-2	-40	-7	-1	-9	+2	+7	-2
	14 d	-36	-6	-52	-8	+1	-14	+1	+9	-6
	21 d	-42	-6	-61	-7	+6	-15	+4	+11	-8
	30 d	-46	-8	-72	-5	+13	-17	+5	+16	-3

The bias and precision of the different methods for estimating N load are greatly influenced by the range of N concentrations and by the relationship of N concentrations to flow rates (Table 2 and Figure 1). In this study, the NO_3-N concentrations had a greater range than those of the TKN concentrations and had a more distinct relationship between concentrations and flow; consequently, the load estimation methods were less precise and more biased when estimating NO_3-N than when estimating TKN. The range of concentrations was greatest for both NO_3-N and TKN for the first year of the data set, thus load estimation by the methods were less precise and more biased for this year when compared to the last two years.

The relationship between flow and concentration for NO_3-N was a concentrating relationship for the drained pine site. That is, concentration increased when flow increased. The concentrating relationship for NO_3-N is very likely due to the drainage characteristics of the drained pine plantation site. This site had good subsurface drainage due to the ditches and relatively high hydraulic conductivities of the soils, and poor surface drainage due to the large beds (>0.25 m) for the trees. Therefore, nearly all of the water leaving the watershed drained by subsurface drainage. The efflux of soluble NO_3-N predominately occurs in subsurface drainage water. NO_3-N builds up in the soil as a result of mineralization and nitrification between flow events and is flushed out during the high flow events. This is in contrast to the watersheds with good surface drainage characteristics where NO_3-N concentrations draining by subsurface drainage may be diluted by surface runoff during high flow events.

The load estimation methods consistently underestimated NO_3-N load at the drained pine site, which was a result of the concentrating effect in the relationship between flow and concentration. For the watersheds analyzed by Birgand et al. (2010) in Brittany, France, the load estimation methods tended to overestimate NO_3-N loads. Relationships between flow and NO_3-N concentrations in seven of the nine watersheds in Brittany exhibited diluting effects. These watersheds most likely had better surface drainage characteristics than the drained pine watershed. The M5 method for estimating NO_3-N load was selected as the best of the methods tested in the Birgand et al (2010) study and the biases and precisions for the M5 method in the study were better than for those for the drained pine watersheds. In other studies where the relationships between flow and concentrations showed concentration effects (e.g. Coynel et al, 2004; Moatar et al, 2006; Littlewood, 1992: and Walling and Webb, 1985), the load estimation methods have tended to underestimate annual loads. This is consistent with the tendency to underestimate N loads from the drained pine watershed. For most of these studies, the determinant was total suspended solids.

The variation of NO_3-N loads in the drainage water from the drained pine watershed presents a challenge to the load estimation methods. Another factor that compounds this challenge is the hydrological reactivity of the watershed. Hydrological reactivity, defined as the proportion of annual outflow that occurs in 2% of the time corresponding to the highest flow rates, ranged from 22% for 1993-94 to 31% for 1991-92. Hydrological reactivity has been shown to correlate to precision limits of a given sampling frequency (Moatar and Meybeck, 2007). With the wide ranges of both flow and NO_3-N concentrations and the strong positive correlated between flow and concentrations, the reactivity of the NO_3-N loads flowing from the pine watershed were very high, ranging from 40% for 1992-93 to 50% for 1991-92 (that is respectively 40% and 50% of the total annual load occurred in 2% of the time).

Forested lands do not typically exhibit high hydrological reactivity on a per area basis. The relatively (i.e. relative to other watersheds used in load estimation studies) high hydrologic reactivity of the drained pine watershed is more likely due to the relative small size of the watershed. Nevertheless, if NO_3-N loads from small drained forest watersheds are going to be calculated from discrete samples, the sample interval should be well less than 7 days, probably in the range of 1 to 3 days. Since the range of TKN concentrations is lower than for NO_3-N, load estimates for TKN may be acceptable from samples collected every 7 days.

CONCLUSIONS The precision and bias of annual NO_3-N and TKN load estimates for the drained pine watershed varied depending on sampling interval, method of calculation, range of concentrations, and the relationship of concentrations with flow rates. On average, all of the calculation methods estimated loads that were lower than the calculated load. The bias for the M5 method was not as great as for M3 and M6; however, the precision of the M5 was not as good as the precision for M3 and M6. In this study, the NO_3-N concentrations had a greater range than those of the TKN concentrations and had a more distinct relationship between concentrations and flow; consequently, the load estimation methods were less precise and more biased when estimating NO_3-N than when estimating TKN. Errors in load estimations are likely exacerbated by the relatively high hydrological reactivity of the drained pine watershed. High hydrological reactivity is likely more due to the small area of the watershed than by the forest land use. If NO_3-N loads from small drained forest watersheds are going to be calculated from discrete samples, the sample interval should be well less than 7 days, probably in the range of 1 to 3 days. Load estimates for TKN may be acceptable from samples collected every 7 days.

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