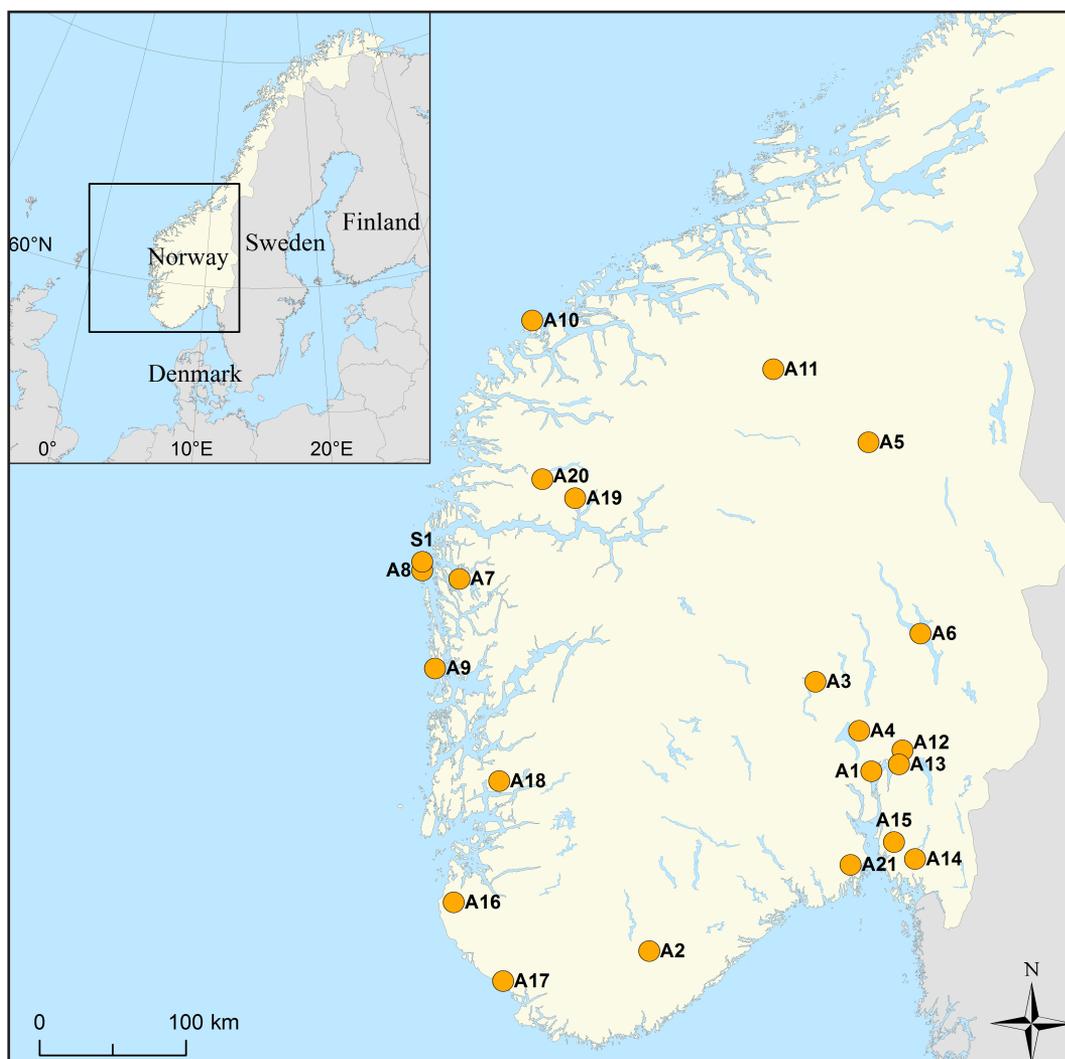


Measuring amines in Norwegian surface waters



Main Office

Gaustadalléen 21
 NO-0349 Oslo, Norway
 Phone (47) 22 18 51 00
 Telefax (47) 22 18 52 00
 Internet: www.niva.no

Regional Office, Sørlandet

Jon Lilletuns vei 3
 NO-4879 Grimstad, Norway
 Phone (47) 22 18 51 00
 Telefax (47) 37 04 45 13

Regional Office, Østlandet

Sandvikaveien 59
 NO-2312 Ottestad, Norway
 Phone (47) 22 18 51 00
 Telefax (47) 62 57 66 53

Regional Office, Vestlandet

Thormøhlens gate 53 D
 NO-5006 Bergen Norway
 Phone (47) 22 18 51 00
 Telefax (47) 55 31 22 14

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Abstract

In summer 2012 we carried out a survey of amine concentrations in 21 Norwegian lakes with the aim of assessing the prevalence of amines in Norwegian lakes and identifying the main factors affecting concentrations of these compounds. The lakes were chosen to cover a range of lake types, water chemistry, catchment land use, and influence of anthropogenic pollution. As intended, the sites chosen covered a wide range in major component water chemistry and spanned gradients in the influence of several natural and anthropogenic drivers, including eutrophication, prevalence of wetlands in the catchment, and acidification.

The lack of available data on amines in surface waters is in part due to the analytical challenges associated with analysis of amines in complex environmental matrices, including surface waters. Due to analytical challenges, uncertainty surrounding the measured amine concentrations, and further development of analytical methods, several iterations of the method have been employed by NILU during this study and over the course of NIVA's collaborative amine work with NILU. Due to this uncertainty in analyzed amine concentrations, we are unfortunately not able to report robust and quality assured data from amine analyses in the current study, nor are we able to assess seasonality in amine concentrations for samples collected for the seasonal study.

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1. CO ₂ fangst	1. CO ₂ capture
2. amin	2. amine
3. innsjøer	3. lakes
4. forurensing	4. pollution



Merete Grung
 Project Manager



Elisabeth Lie
 Research Manager



Thorjorn Larsen
 Senior Adviser

Measuring amines in Norwegian surface waters

Preface

This survey of amine concentrations in Norwegian lakes was carried out by NIVA (Norwegian Institute for Water Research) in cooperation with NILU (Norwegian Institute for Air Research), where amine analyses were carried out. The personnel involved in the project are outlined in Appendix 8.2, and are thanked for their important contributions to this study.

This study was performed with financial support from Statoil and the CLIMIT programme, and we thank the funding sources for their support and input throughout the project.

Oslo, December 2014

Amanda Poste

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1. Summary

Amine-based CO₂ capture is one of the most promising technologies for emissions reductions from stack gases from fossil-fuel power plant and other industrial waste gases. Amines are widespread in the environment and have many natural as well as industrial sources. Organisms can produce and release amines to the environment, and many precursors to amines (including amino acids) are released by aquatic organisms either while alive, or during decomposition of deceased plants and animals. Other potential natural sources of amines (and amine precursors) to freshwaters include runoff from the terrestrial environment as well as sea birds and other migratory wildlife. However, globally there are few data available regarding concentrations of amines in natural surface waters.

In summer 2012 we carried out a survey of amine concentrations in 21 Norwegian lakes with the aim of assessing the prevalence of amines in Norwegian lakes and identifying the main factors affecting concentrations of these compounds. The lakes were chosen to cover a range of lake types, water chemistry, catchment land use, and influence of anthropogenic pollution. Samples were collected from the lake outlets. Water samples for amine determination were delivered to the Norwegian Institute for Air Research (NILU) within 48 hours of collection, derivatized, and then detected by reversed phase liquid chromatography coupled with mass spectrometry. Seven amines were determined (methylamine: MA; ethylamine: EA; dimethylamine: DMA; diethylamine: DEA; monoethanolamine: MEA; 2-amino-2-methyl-propanol: AMP; and piperazine: PIP). Water samples were also delivered to the NIVA laboratory for determination of several water chemistry parameters, including: TOC, pH, TP, TN, SO₄, NO₃, Cl and Na, as well as chlorophyll *a* (as a measure of phytoplankton biomass).

As intended, the sites chosen covered a wide range in major component water chemistry and spanned gradients in the influence of several natural and anthropogenic drivers, including eutrophication, prevalence of wetlands in the catchment, and acidification.

The lack of available data on amines in surface waters is in part due to the analytical challenges associated with analysis of amines in complex environmental matrices, including surface waters. Due to analytical challenges, uncertainty surrounding the measured amine concentrations, and further development of analytical methods, several iterations of the method have been employed by NILU during this study and over the course of NIVA's collaborative amine work with NILU (including a baseline study (Grung et al. 2012), monitoring of TCM activities, and the current study). This has strongly complicated the interpretation and comparison of measured amine concentrations for these studies.

Due to this uncertainty in analyzed amine concentrations, we are unfortunately not able to report robust and quality assured data from amine analyses in the current study, nor are we able to assess seasonality in amine concentrations for samples collected for the seasonal study. Future work should entail resolving analytical uncertainty. In particular, it would be useful to carry out an inter-lab comparison for analysis of amines in water samples.

The challenges we have encountered with obtaining reliable data on amine concentrations in water samples are extremely unfortunate, given that this study was designed to yield key insights into the determinants of natural amine concentrations in surface waters, and of potential external and internal sources of amines to lakes. This information is critical for the robust assessment of the importance and potential impact of future increases in amine loading to boreal lakes (e.g. due to amine-based CO₂ capture).

2. Norsk sammendrag

Aminbasert CO₂-fangst er en av de ledende teknikkene for å redusere utslipp av denne klimagassen fra industrielle avgasser, både fra kullfyrte og oljefyrte energikilder. Aminer finnes overalt i miljøet, og har ulike naturlige så vel som industrielle kilder. Organismer kan produsere og avgi aminer til miljøet. Mange forløpere til aminer (bl.a. aminosyrer) stammer fra akvatiske organismer, enten mens de er i live, eller ved nedbrytning av plantemateriale eller døde organismer. Andre naturlige kilder til aminer i ferskvann er tilsig fra terrestrisk miljø, samt avføring fra fugler eller dyr. Det er imidlertid få undersøkelser av miljøkonsentrasjoner av aminer i naturlig overflatevann.

Sommeren 2012 gjennomførte vi en undersøkelse av aminkonsentrasjoner i 21 norske innsjøer. Målet var å finne ut hvilke nivåer av aminer som var vanlige, samt å identifisere hvilke faktorer som bidro til de observerte nivåene. Innsjøene ble valgt for å omfatte ulike typer innsjøer, blant annet når det gjelder vannkjemi, arealbruk i nedbørsfeltet og grad av menneskelig påvirkning. Vannprøver ble samlet fra utløpet av innsjøen. Vannprøver til bestemmelse av aminer ble levert til Norsk institutt for luftforskning (NILU) senest 48 timer etter prøvetakning. Etter mottak ble prøvene derivatisert, og aminer ble målt ved hjelp av reversert fase væskechromatografi koblet til et massespektrometer. Syv aminer ble bestemt: metylamin (MA), etylamin (EA), dimetylammin (DMA), dietylammin (DEA), monoetanolamin (MEA), 2-amino-2-metylpropanol (AMP) og piperazin (PIP). Deteksjonsgrensene varierte fra 10-100 ng/L. Vannprøver ble også levert til NIVAs laboratorium for bestemmelse av ulike kjemiske vannparametre: totalt organisk karbon (TOC), pH, totalt fosfor (TP), totalt nitrogen (TN), ammonium (SO₄), nitrat (NO₃), Cl, og klorofyll *a* (som et mål på biomassen av planktonalger).

Analysen av vannkjemien viste at vi hadde lyktes med å velge innsjøer med et bredt spekter av parametrene vi analyserte, både med henblikk på naturlig og menneskelig påvirkning. Dette inkluderer eutrofiering, graden av myrområder i nedbørsfeltet og forsurening.

Mangelen på målte konsentrasjoner av aminer i naturlig overflatevann skyldes delvis analytiske utfordringer knyttet til aminer i komplekse miljøprøver. På grunn av de analytiske utfordringene og usikkerhet knyttet til de målte konsentrasjonene av aminer, har NILU kontinuerlig videreutviklet analysemetodene som er benyttet til dette formålet. Dette gjelder tre studier som NIVA i samarbeid med NILU har gjennomført: baselineundersøkelsen (Grung et al. 2012), overvåking av TCM og denne undersøkelsen. Videreutvikling og forbedring av analysemetodene har vanskeliggjort tolkning og sammenligning av målte aminkonsentrasjoner i de tre undersøkelsene.

På grunn av usikkerheten knyttet til amin-analysene er det dessverre ikke mulig å rapportere robuste og kvalitetssikrede data for aminkonsentrasjoner i denne studien. Det betyr også at det ikke er mulig å rapportere årstidsvariasjoner for de prøvene som ble samlet inn til dette formålet. Vi håper fremtidig metodeutvikling vil løse de utfordringene som gjenstår. Spesielt vil det være nyttig med en ringtest for analyse av aminer i overflatevann.

Utfordringene knyttet til å måle aminkonsentrasjoner i overflatevann er svært uheldig, spesielt med tanke på at denne studien kunne ha gitt viktig informasjon knyttet til naturlig aminkonsentrasjoner i overflatevann og hvilke kilder dette kan knyttes til. Denne informasjonen er kritisk for en vurdering av mulige påvirkninger av aminkonsentrasjoner som eventuelt kan knyttes til amin-basert CO₂-fangst.

3. Introduction

Technologies designed to capture and store CO₂ from combustion flue gases (post combustion capture: PCC) are increasingly being considered for use in reducing CO₂ emissions, particularly at sites where electricity is being produced using fossil fuels. Aqueous amines are the most common solvents for PCC and have long been used as solvents in CO₂ removal (“sweetening”) processes for natural gas (Reynolds et al. 2012). PCC activities are known to result in a loss of amines from the absorber column, and as such, may represent a potential source of amines and amine degradation products (including nitrosamines and nitramines) to the environment (Reynolds et al. 2012), which is of concern with respect to potential toxic effects on aquatic ecosystems (Veltman et al. 2010).

CO₂ capture and storage has been proposed for Norwegian gas-fired power plants (Kårstø and Mongstad) as a means of reducing CO₂ emissions to the atmosphere, and amine-based PCC technology will be tested in Norway at Test Centre Mongstad (TCM). In 2011, in conjunction with TCM and the Norwegian Institute for Forest and Landscape (Skog og Landskap), NIVA carried out a baseline survey of concentrations of several amines in the Mongstad region (Grung et al. 2011).

Amines are widespread in the environment and have many natural as well as industrial sources (Poste et al. 2014). Biogenic amines can be formed through decarboxylation of amino acids (often through microbial processes), or by amination of ketones and aldehydes (Santos 1996). In the aquatic environment, many aquatic organisms are capable of producing and releasing amines (both primary amines as well as more complex compounds) to the surrounding environment. In the aquatic environment, studies in marine systems have indicated that aliphatic amines (such as methylamine, dimethylamine and diethylamine) often originate from biological sources (Facchini et al. 2008, Müller et al. 2009). Furthermore, many precursors to amines (including amino acids) are released by aquatic organisms while they are alive, or during decomposition of deceased plants and animals. These processes are likely to represent an important *in situ* source of amines to aquatic ecosystems.

Amines and amine precursors may also be delivered to freshwaters from their terrestrial catchments (while catchment export of both nitrate and nitrite may be of important in determining the potential for formation of nitrosamines and nitramines). Other potential natural sources of amines (and amine precursors) to freshwaters include sea birds and other migratory wildlife. These organisms could deliver amine and amine-related compounds to aquatic ecosystems through their faeces and urine both directly in the water and in the catchment. Furthermore, these organisms may be sources of both nitrates and nitrites, which may be of importance with respect to nitrosamine and nitramine formation.

There is a remarkably broad and complex range of potential sources of amines to the aquatic environment, including both natural as well as anthropogenic sources. This suggests that, in order to identify the potential for changes in delivery of these compounds (e.g. through amine-based CO₂ capture activities) and the resultant implications for aquatic ecosystem and human health, there is a strong need to identify the sources that are most prevalent and most important to determining eventual concentrations of these compounds in freshwaters.

This survey of amine concentrations in Norwegian lakes was designed to meet the following objectives:

1. To characterize amine concentrations in a diverse set of lakes, in order to gain insight into the range of concentrations encountered in Norwegian lakes. Lakes were selected to achieve gradients in water chemistry, the relative influence of various human activities, and other factors.
2. To identify potential determinants of amine concentrations in Norwegian freshwaters.

4. Materials and methods

4.1 Study Sites

We selected a set of 21 study lakes across southern Norway (Table 1, Figure 1) and one seawater site, which was included as a marine reference. The study lakes were chosen to represent the range of conditions that exist in southern Norwegian lakes. Many of the lakes included in the current study have been a part of monitoring programs at NIVA for many years (often decades), and as such, for several of the study lakes there are many existing data that can be used to contextualize the results of the current study. Three of the lakes included in the current study were also included in the 2011 baseline study carried out in the Mongstad region (Fonnebostvatnet (A7; M11 in 2011 study), Langevatnet (A8; M16 in 2011 study) and Storavatnet (A9; M52 in 2011 study)), in order to allow for temporal comparison of results.

Table 1. General characteristics of study lakes.

Site Code	Lake	County	Area (km ²)	Elevation (m a.s.l.)	Latitude (°)	Longitude (°)	NVEnr
A1	Spiradammen	Akershus	0.03	13	59.84	10.50	80670
A2	Lille Hovvatnet	Aust-Agder	0.22	503	58.61	8.04	10069
A3	Langtjern	Buskerud	0.23	516	60.37	9.73	7272
A4	Steinsfjorden	Buskerud	13.81	63	60.08	10.32	67985
A5	Atnsjøen	Hedmark	5.01	701	61.89	10.14	126
A6	Mjøsa	Hedmark	369.0	123	60.90	10.69	118
A7	Fonnebostvatnet	Hordaland	0.06	23	60.75	5.18	26234
A8	Langevatnet	Hordaland	0.05	16	60.77	4.71	26190
A9	Storavatnet	Hordaland	0.14	24	60.18	5.05	26964
A10	Litlevatnet	Møre og Romsdal	0.02	123	62.40	5.61	30910
A11	Svartdalsvatnet	Oppland	0.59	1018	62.28	8.84	34660
A12	Breidsjøen	Oslo	0.21	248	60.00	11.03	2514
A13	Østensjøvatnet	Oslo	0.33	107	59.89	10.83	2513
A14	Tunevatnet	Østfold	2.36	40	59.31	11.10	3451
A15	Vansjø	Østfold	35.62	25	59.38	10.86	291
A16	Frøylandsvatnet	Rogaland	4.69	24	58.74	5.67	1552
A17	Måkevann	Rogaland	0.28	272	58.32	6.38	21729
A18	Røyrvatnet	Rogaland	0.43	231	59.54	6.03	22548
A19	Nystølsvatnet	Sogn og Fjordane	1.27	715	61.34	6.46	1651
A20	Skjervatjern	Sogn og Fjordane	0.02	136	61.43	6.04	28412
A21	Akersvatnet	Vestfold	2.39	16	59.24	10.33	314
S1	Fedje Seawater	Rogaland	~	0	60.78	4.70	~

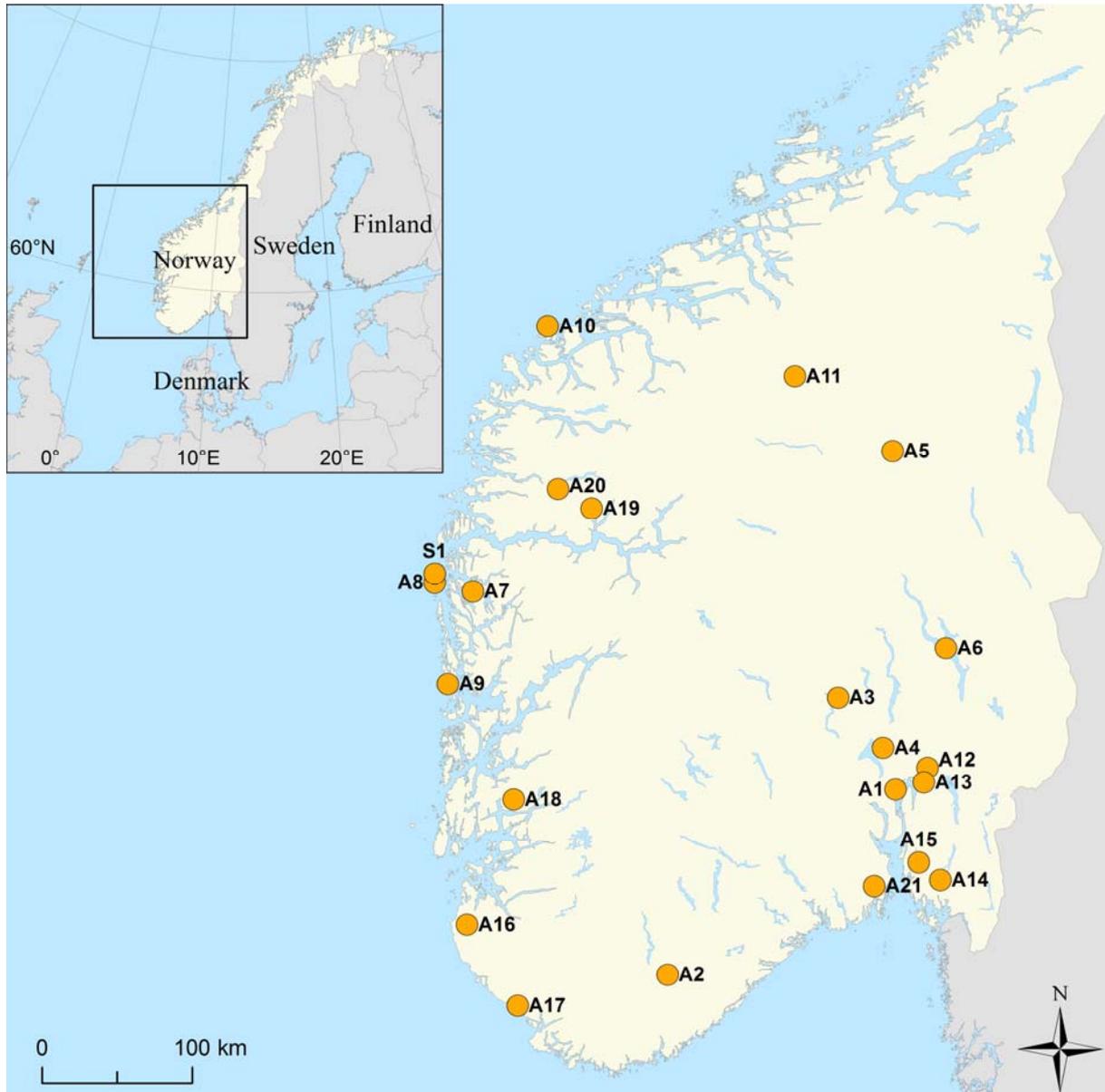


Figure 1. Map showing locations of study lakes. Sites in this map (and in all others that follow) are labeled with the codes defined in Table 1.

The set of lakes that we selected are diverse; they differ in their water chemistry and their exposure to various human activities, as well as several other factors (see Table 2). In particular, in our lake selection we have aimed to achieve the following gradients:

- A broad geographic distribution in southern Norway
- A range in lake elevation, lake area, lake depth and catchment area
- Differences in the prevalence of agriculture (both grain and animal), urbanization, and forested areas in lake catchments
- Differences in lake trophic status (including several eutrophic lakes)
- Differences in marine influence (i.e. proximity to the sea)
- Differences in humic content (lake colour) and pH
- Differences in macrophyte abundance
- Differences in the influence of seabirds

Table 2. Summary of factors influencing the study lakes (*impacted and **heavily impacted unless otherwise noted; agric: agriculture; urban: urbanization; eutro: eutrophication (**TP (total phosphorus)>25 µg/L, *10<TP<25 µg/L); macro: macrophytes; birds: bird influence; marine: marine influence (**high Cl and near sea); TOC: humic content (**TOC>10 mg/L, 5<TOC<10 mg/L); acid: lake acidity (*pH<5, *5<pH<6); forest: forested catchment).

Site	Lake	Agri	Urban	Eutro	Macro	Birds	Marine	TOC	Acid	Forest
A1	Spiradammen	*a	*	*	**			*		
A2	Lille Hovvatnet								*	*
A3	Langtjern							**	*	*
A4	Steinsfjorden	*a		*	*					
A5	Atnsjøen									
A6	Mjøsa	*a	*							
A7	Fonnebostvatnet	*b		**				**		
A8	Langevatnet			*e		*	**	*	*	
A9	Storavatnet						**		*	
A10	Litlevatnet			*e		**	**	*		
A11	Svartdalsvatnet ^c									
A12	Breidsjøen									*
A13	Østensjøvatnet		**	**	*	*	*	*		
A14	Tunevatnet	**a	*	**				*		
A15	Vansjø	*a		**				*		
A16	Frøylandsvatnet	**b		**						
A17	Måkevann ^d								*	
A18	Røyrvatnet								*	*
A19	Nystølsvatnet ^c								*	
A20	Skjervatjern							*	**	*
A21	Akersvatnet	**b		**				*		
S1	Fedje Seawater						**			

^a primarily grain agriculture; ^b primarily animal agriculture; ^c alpine lake; ^d relatively high concentration of nitrates for a low nutrient lake; ^e despite high TP concentrations, phytoplankton biomass is low in these lakes.

4.2 Sample collection

Field sampling included a broad survey of amines and physicochemical conditions at all study sites carried out between 26 July 2012 and 24 August 2012, as well as a seasonal study at five select study sites (Atnsjøen (A5), Fonnebostvatnet (A7), Langtjern (A3), Vansjø (A15), and Østensjøvannet (A13)), where additional sampling was carried out on four occasions (autumn (Oct–Nov 2012), winter (Jan–Feb 2013), spring (May–Jun 2013) and summer (Aug–Sep 2013)).

Water samples from all lakes were collected directly from the outflow (see Figure 2 for examples of outflow sampling sites). During autumn overturn, when the lake is fully mixed, sampling from a lake outflow should theoretically yield a sample that is fairly representative of the lake as a whole. However, during summer stratification, outflow water may be more representative of epilimnetic (upper water column) conditions.

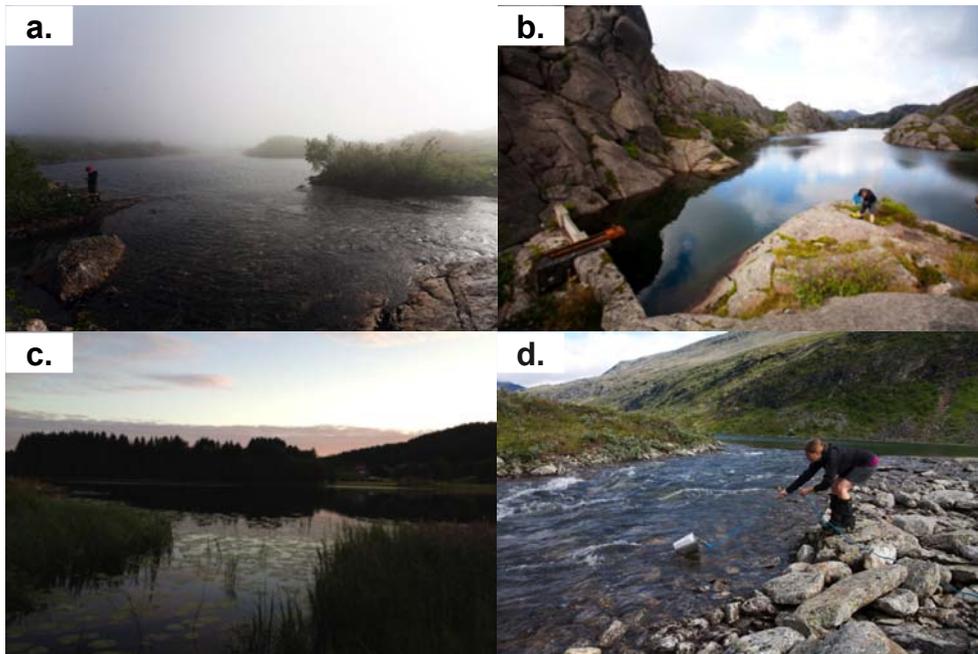


Figure 2. Examples of lake outflow sampling sites (a. Nystølsvatnet (A19), b. Måkevann (A17), c. Fonnebostvatnet (A7), and d. Svartdalsvatnet (A11)).

Water was collected using an acetone-cleaned stainless steel bucket, and transferred to sample containers (dark 1 L polyethylene bottles for chlorophyll *a* analysis, regular 1 L polyethylene bottles for analysis of major chemical components, and 2.5 L dark glass bottles for analysis of amines). Water samples were kept in a dark and cold ($\sim 4\text{--}10\text{ }^{\circ}\text{C}$) environment during transport, and water samples for analysis of amines were delivered to NILU within 48 hours of collection. Meanwhile, within 12 hours of sampling, water for chlorophyll *a* analysis was filtered onto GF/C filters (nominal pore size of $0.7\text{ }\mu\text{m}$) which were frozen until analysis.

4.3 Chemical analysis

4.3.1 General water chemistry

Analysis of major chemical components (TOC, pH, TP, TN, SO₄, NO₃, Cl and Na) and chlorophyll *a* (as a measure of phytoplankton biomass) was carried out at NIVA (see Table 3 for a summary of analytical methods and limits of detection). These parameters were included in order to characterize both the general water chemistry (including nutrient concentrations, humic matter/water colour, acidity and major ion concentrations) as well as trophic status of the study lakes.

Table 3. Summary of analytical methods and limits of detection (LOD) for surface water chemical analyses at NIVA.

Code	Parameter	Analytical method	LOD	Unit
pH	pH	Potentiometry	~	~
TOC	Total organic carbon	UV/persulphate oxidation to CO ₂ + IR-detection	0.10	mg C L ⁻¹
TN	Total nitrogen	Automated photometry	10	µg N L ⁻¹
TP	Total phosphorus	Automated photometry	1	µg P L ⁻¹
NO ₃	Nitrate	Ion chromatography	1	µg N L ⁻¹
Na	Sodium	Ion chromatography	0.02	mg L ⁻¹
Cl	Chloride	Ion chromatography	0.03	mg L ⁻¹
SO ₄	Sulphate	Ion chromatography	0.04	mg L ⁻¹
Chl <i>a</i>	Chlorophyll <i>a</i>	Acetone extraction + spectrophotometry	0.31	µg L ⁻¹

4.3.2 Amines

Analysis of seven amines of interest (methylamine, ethylamine, dimethylamine, diethylamine, monoethanolamine, 2-amino-2-methyl-propanol, and piperazine) was carried out at NILU. These seven amines are the same compounds that were measured in the 2011 baseline survey (Grung et al. 2012). These analyses were performed using NILU's in-house validated methods, which are based on using derivatization to improve the analyte behavior towards reversed phase chromatography (see Table 4 for a summary of general methodological approaches). Aliquots of the water samples were prepared by adding a buffer solution and the derivatizing agent (either dansyl chloride or benzene sulphonylchloride). After a defined reaction time, sample analysis was carried out on either Instrument 1 or 2 (Table 4).

Due to analytical challenges, uncertainty surrounding the measured amine concentrations, and further development of analytical methods, several iterations of the method (with differing limits of detection) were employed by NILU during the course of this project (Table 4), with some samples undergoing re-analysis using updated methods (Table 5). We also sent samples from a small number of lakes (collected in April 2014, including three from the current study and three from the baseline study (Grung et al. 2012)) to an independent lab for analysis of several amines in order to assess the reproducibility of measured amine concentrations, however, we are not entitled to report these results at this time (Table 5).

In method 4, three different blanks were included for quality control: one with only derivatization reagents and internal standards (which contain milliQ water and acetonitrile), one which was additionally diluted with milliQ water (to resemble a real sample) and one with reagents and HPLC-grade bottled water. The standard based on milliQ water showed high values of amines while the undiluted sample showed lower amines values. The latter was used for blank corrections.

Table 4. Summary of general approaches for the various methods used by NILU (based on personal communication from NILU).

NILU Method Number	General approach
1	Derivatizing agent: dansyl chloride; Instrument 1 (HPLC-ToF-MS)
2	Derivatizing agent: <i>unknown</i> ; Instrument 2 (UHPLC-ToF-MS)
3	Derivatizing agent: <i>unknown</i> ; Instrument 2; reagent corrected
4	Derivatizing agent: benzene sulphonylchloride; Instrument 2; New internal standards

Table 5. Summary of location of analysis and methods used for various sample sets (baseline study data are reported by Grung et al. 2012).

Sample set	Location of analysis and method number
Baseline study	Analyzed once at NILU using "Method 1"
2012 survey	Analyzed twice at NILU using "Methods 2 and 3"
Seasonal study	Analyzed once at NILU using "Method 3"
2014 follow-up sampling	Analyzed at NILU using "Method 4" and at external independent lab

5. Results and Discussion

5.1 General site chemistry

Based on the results of analysis of general water chemistry (Table 6) for samples collected from the 21 study lakes in the 2012 survey, we found that the sites chosen covered a wide range in major component water chemistry and spanned several gradients in the influence of both natural and anthropogenic drivers including eutrophication, prevalence of wetlands in the catchment, and acidification.

Table 6. Results from analysis of major chemical components and chlorophyll *a* (as a measure of phytoplankton biomass) in water from the study sites for the 2012 survey.

Site	pH	TP (ug/L)	TN (ug/L)	NO ₃ (ug/L)	TOC (mg/L)	Na (mg/L)	Cl (mg/L)	SO ₄ (mg/L)	Chl <i>a</i> (ug/L)
A1	7.97	13	450	1	5.9	7.6	8.4	17.8	1.8
A2	5.41	5	320	25	4.3	1.19	1.72	1.02	0.78
A3	5.24	5	270	1	11.7	0.51	0.38	0.55	1.40
A4	7.61	11	305	12	3.5	4.01	5.17	6.16	4.9
A5	6.61	4	103	11	1.3	0.36	0.25	0.71	2.5
A6	7.36	4	440	270	2.1	1.13	1.61	3.92	3.1
A7	6.32	33	605	<1	11.2	6.1	8.97	4	2.6
A8	5.06	16	255	1	5.2	19.3	35.1	4.99	1.5
A9	5.98	5	185	1	3	12.1	21.9	3.32	1.2
A10	6.93	18	370	1	7.8	21.4	37.9	3.61	1.8
A11	6.43	1	70	21	0.33	0.33	0.36	0.51	0.61
A12	7.37	3	215	23	3.1	1.76	1.83	2.73	1.30
A13	7.48	131	1140	6	7.5	23.7	38.8	9.78	80
A14	6.64	31	490	<1	5.3	10.1	15.5	5.71	12
A15	6.88	29	760	260	8.3	8.14	12.8	5.74	10
A16	7.55	25	950	380	4.4	10.3	18.2	6.52	18
A17	5.92	5	380	185	1.8	6.41	11.1	5.04	1.3
A18	5.92	2	165	47	2.1	1.62	2.33	0.94	0.7
A19	5.90	1	77	34	0.34	1	1.67	0.54	<0.31
A20	4.74	5	225	1	7.3	3.6	7.14	0.88	2.2
A21	7.59	56	1410	625	6.7	13.1	20.5	10.7	26
S1	8.09	10	195	2	2	2200	15900	2200	1.1

Some of the natural and anthropogenic (e.g. pollution) factors likely to be important determinants of water chemistry in the study lakes are summarized in Table 7 and are described in the text that follows.

Table 7. Select factors affecting the chemical composition of natural waters, with examples of chemical parameters affected, and sites influenced by these factors. Codes used are defined in Tables 1 and 3.

Factor	Type	Chemical signals	Site examples
Seasalts (atmospheric deposition)	Natural	High Cl, Na, SO ₄	Ma (A17), Sto (A9), Lit (A10)
De-icing road salts (from terrestrial catchment)	Pollution	High Cl, Na	Os (A13)
Humic runoff (from terrestrial catchment)	Natural	High TOC (low pH)	Ln (A3), Sk (A20), Fo (A7)
Acidification (deposition of S and N)	Pollution	Low pH, high SO ₄	Lil (A2), Ln (A3)
Eutrophication (nutrients from terrestrial catchment)	Pollution	High TP, TN, TOC, Chl <i>a</i>	Os (A13), Tu (A14), Va (A15), Fr (A16), Ak (A21), Fo (A7)

5.1.1 Na, Cl and SO₄

Lakes near the coast usually have high concentrations of chloride (Cl) and sodium (Na), as well as elevated concentrations of both magnesium (Mg) and sulphate (SO₄). This is due to natural seaspray that is entrained into the atmosphere, transported to the land and deposited in wet and dry deposition. In Norway the seasalt influence decreases with distance from the coast and is minor after 10-20 km inland. In the current study, several of the coastal study lakes had particularly high Na, Cl and SO₄ concentrations (Table 6, Figure 3).

Østensjøvatnet (A13) also had very high Na and Cl concentrations, despite having less of a marine influence than many of the coastal lakes. This is likely due to localized application of road salts, given that Østensjøvatnet is an urban lake (within Oslo) that is bordered by a large road.

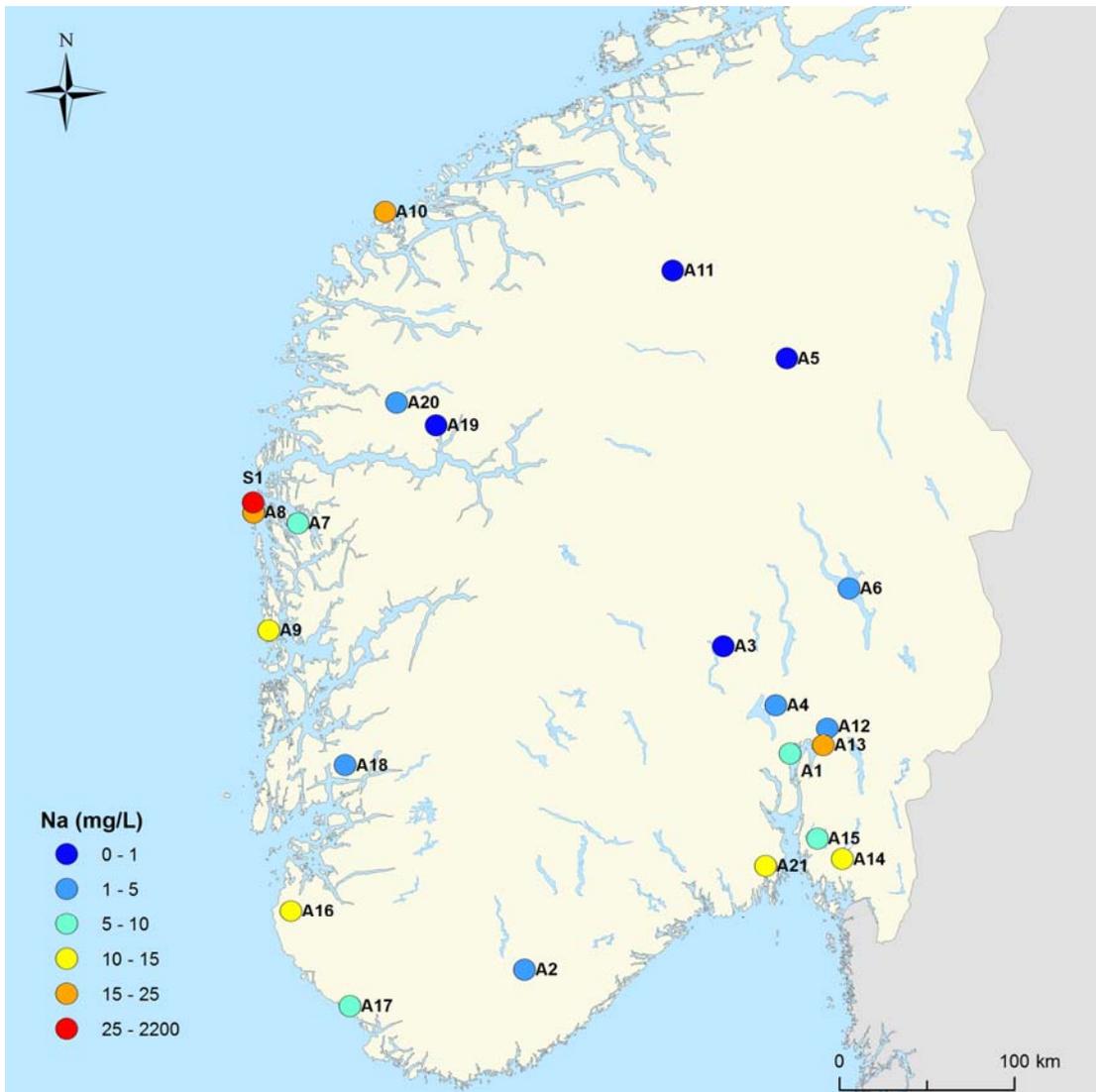


Figure 3. Na concentrations in the study lakes.

5.1.2 Organic matter

Organic matter in lakes can come from several natural or anthropogenic sources. Particularly important sources of organic matter to lakes includes humic-rich runoff from natural forests or peatlands (allochthonous carbon), or organic matter produced within the lake by algae (autochthonous carbon). High levels of dissolved organic matter are characterized by high concentrations of TOC as well as other nutrients such as TN. The dissolved organic matter in humic-rich runoff contains organic acids, which can act to lower the pH in highly coloured waters.

In the current study, TOC concentrations ranged from 0.33 to 11.7 mg/L (Table 6, Figure 4), with the highest concentrations occurring in lakes that have either substantial wetland areas in their catchment (e.g. Langtjern (A3) or Skjervatjern (A20)) or high levels of phytoplankton productivity (e.g. Østensjøvatnet (A13) and Vansjø (A15)).

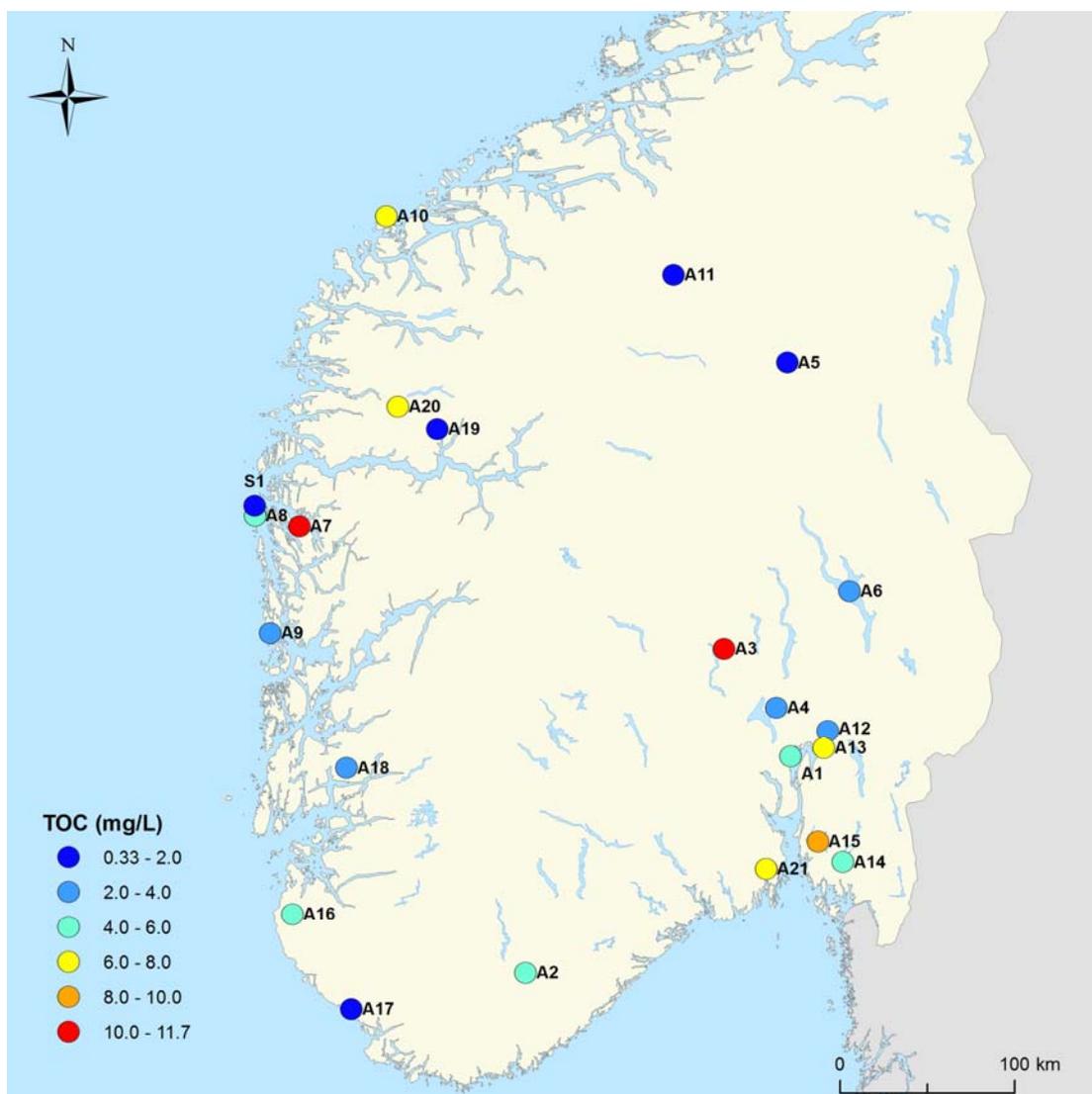


Figure 4. TOC concentrations in the study lakes.

5.1.3 Acidification

Deposition of sulphur and nitrogen components from the atmosphere (acid deposition) leads to acidification of soils, runoff and surface waters. Acidified waters have low pH and elevated concentrations of sulphate, and sometimes also nitrate, as well as high levels of inorganic aluminium species that are toxic to fish and other organisms.

The study lakes ranged in pH from 4.74 to 7.97 (Table 6, Figure 5; although the seawater reference site had a pH of 8.09). The lowest pH values were typically observed for poorly buffered humic lakes with a history of anthropogenic acidification (such as Lille Hovvatnet (A2) and Langtjern (A3)).

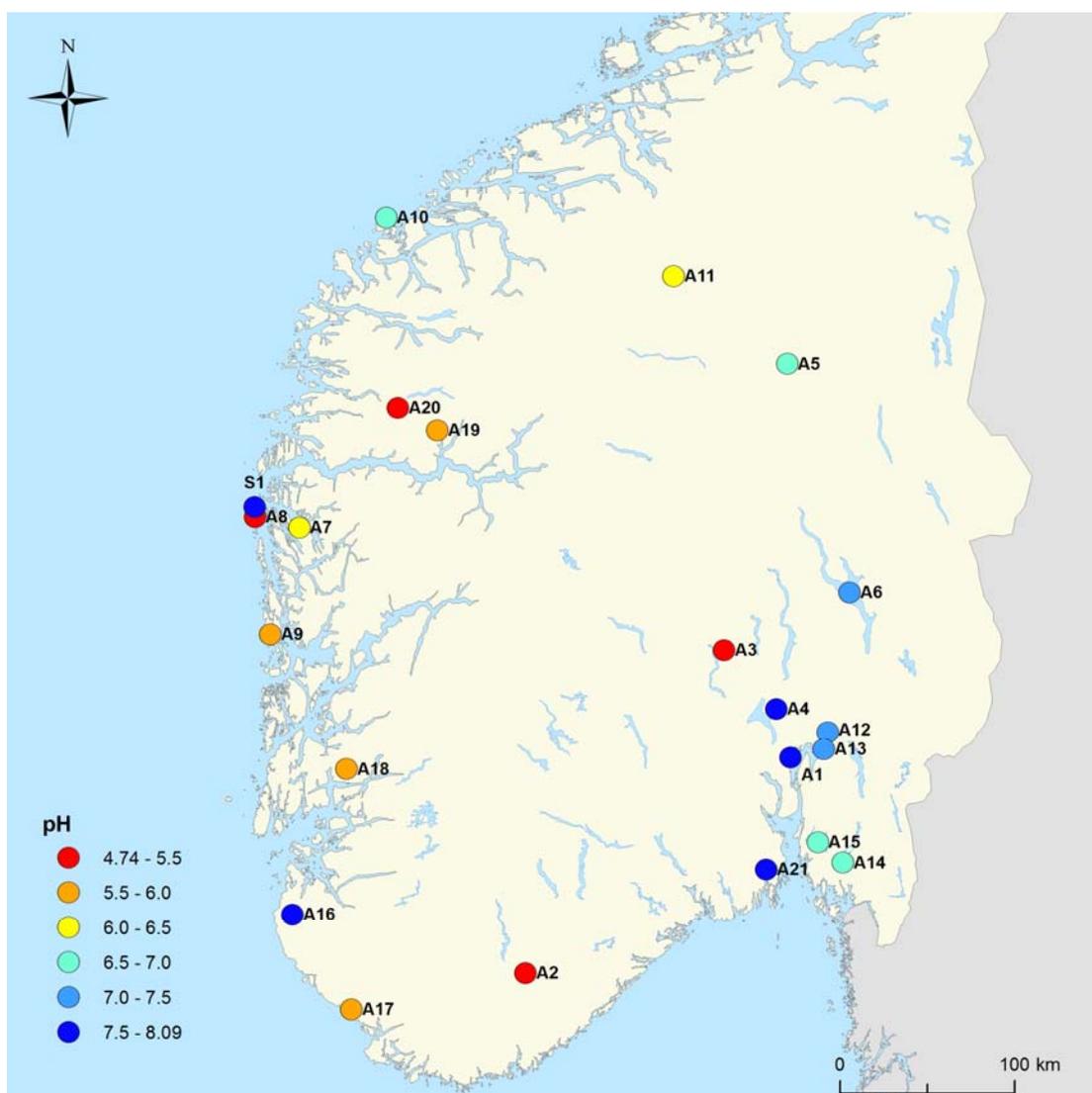


Figure 5. pH of the study lakes.

5.1.4 Eutrophication

Pollution by nutrients from agriculture and human wastewaters leads to elevated concentrations of phosphorus and nitrogen. Primary production in most Norwegian lakes is limited by phosphorus, and lakes with high concentrations of phosphorus typically have high biomasses of phytoplankton (reflected in concentrations of the pigment chlorophyll *a*) and /or higher aquatic plants (macrophytes). The high biomasses of primary producers can also lead to high TOC in these lakes.

Lakes are often categorized based on their trophic status (or productivity) based on their nutrient or chlorophyll *a* concentrations. “Oligotrophic” lakes are typically clear, unproductive lakes with low nutrient concentrations and phytoplankton biomass (often defined as a lake where TP < 10 µg/L and/or Chl *a* < 2.5 µg/L), “mesotrophic” lakes are intermediately productive (10 < TP < 35 µg/L and/or 2.5 < Chl *a* < 8 µg/L), “eutrophic” lakes are often defined as have TP concentrations between 35–100 µg/L and/or chlorophyll *a* concentrations between 8–25 µg/L. Lakes with TP and/or chlorophyll *a* concentrations in excess of these ranges are defined as “hypereutrophic”.

Of the sites included in the current study, based on TP and chlorophyll *a* concentrations, more than half of the study lakes can be classified as oligotrophic (Table 6, Figures 6, 7), while only a small subset of the lakes were eutrophic or hypereutrophic (e.g. Østensjøvatnet (A13) and Akersvatnet (A21)). The remaining sites can be classified as mesotrophic.

In particular, lakes heavily influenced by urbanization and/or agriculture tended to have the highest nutrient (TN, TP) and chlorophyll *a* concentrations (see Table 2 for a summary of agricultural and urban influences on the study lakes and their catchments). Meanwhile, the lowest concentrations were observed in dilute and remote lakes without significant inputs of organic matter (or nutrients) from their catchments, such as the alpine lakes Svartdalsvatnet (A11) and Nystølsvatnet (A19).

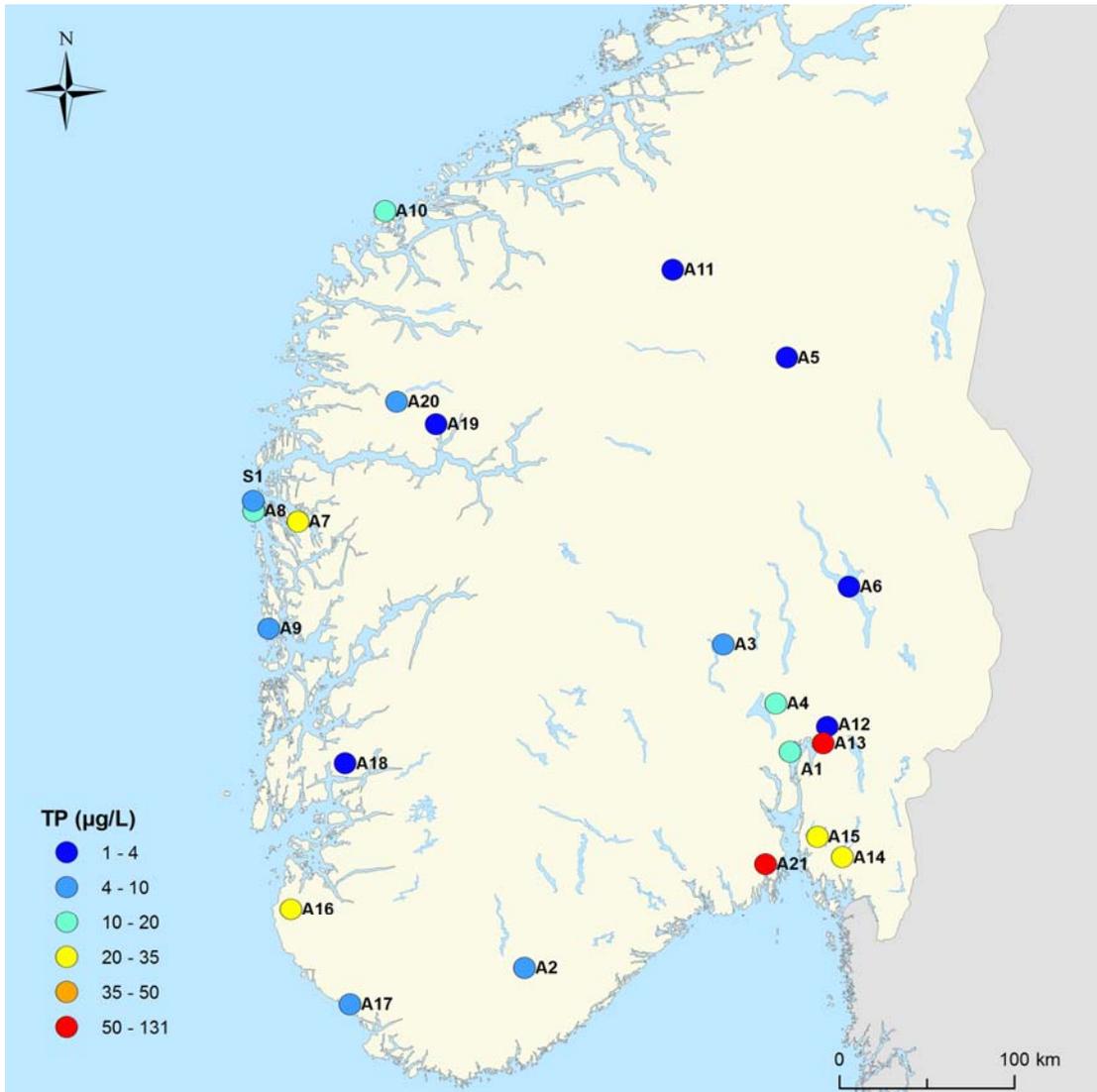


Figure 6. TP concentrations in the study lakes.

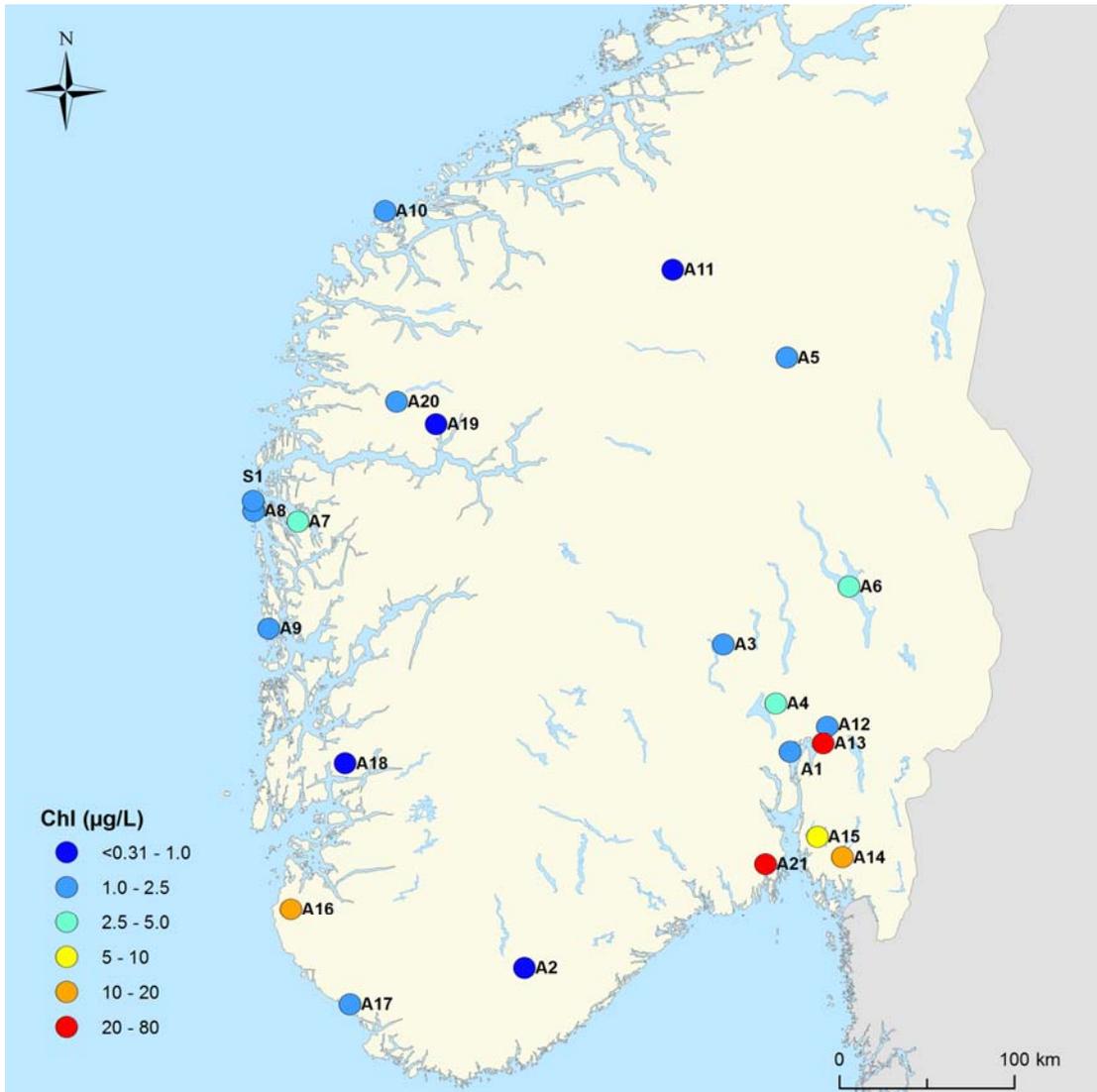


Figure 7. Chlorophyll *a* concentrations (as an indicator of phytoplankton biomass) in the study lakes.

5.2 Relationships between water chemistry parameters

Among the water chemistry parameters measured, there were significant positive correlations (Table 8) between chlorophyll *a* concentrations and total phosphorus and nitrogen concentrations (Figure 8a, b), with higher nutrient concentrations supporting higher biomasses of phytoplankton.

Concentrations of TP, TN and chlorophyll *a* were also positively related to concentrations of marine derived ions and negatively related to elevation (Figure 8c). This is likely due to the fact that lowland and coastal lakes in southern Norway are often situated in an agricultural landscape with higher population densities, thus are more prone to increases in nutrient loading and trophic status.

There were also significant positive correlations (Table 8) between Na, Cl and SO₄ concentrations, driven by the co-occurrence of these ions in water influenced by deposition of marine derived ions (from seaspray). We also found strong negative correlations between these three ions and elevation (Figure 8d), which can also be attributed to differences in marine influence between low-lying coastal areas and higher elevation lakes (which were typically further inland).

Table 8. Pearson's correlation coefficients (*r*) for correlations between water chemistry parameters for freshwater samples. Correlations shown are significant at the P<0.05 level. Values marked with an asterisk are significant at the P<0.01 level.

	pH	TP	TN	NO ₃	TOC	Na	Cl	SO ₄	Chl <i>a</i>
pH	1								
TP		1							
TN	0.47	*0.89	1						
NO ₃				1					
TOC		*0.64	*0.58	-0.45	1				
Na		*0.79	*0.69			1			
Cl		*0.74	*0.66			*0.99	1		
SO ₄	*0.65	*0.78	*0.79			*0.81	*0.77	1	
Chl <i>a</i>	*0.55	*0.87	*0.72			*0.57	0.53	*0.66	1
Elevation		*-0.71	*-0.67			*-0.80	*-0.77	*-0.82	-0.52

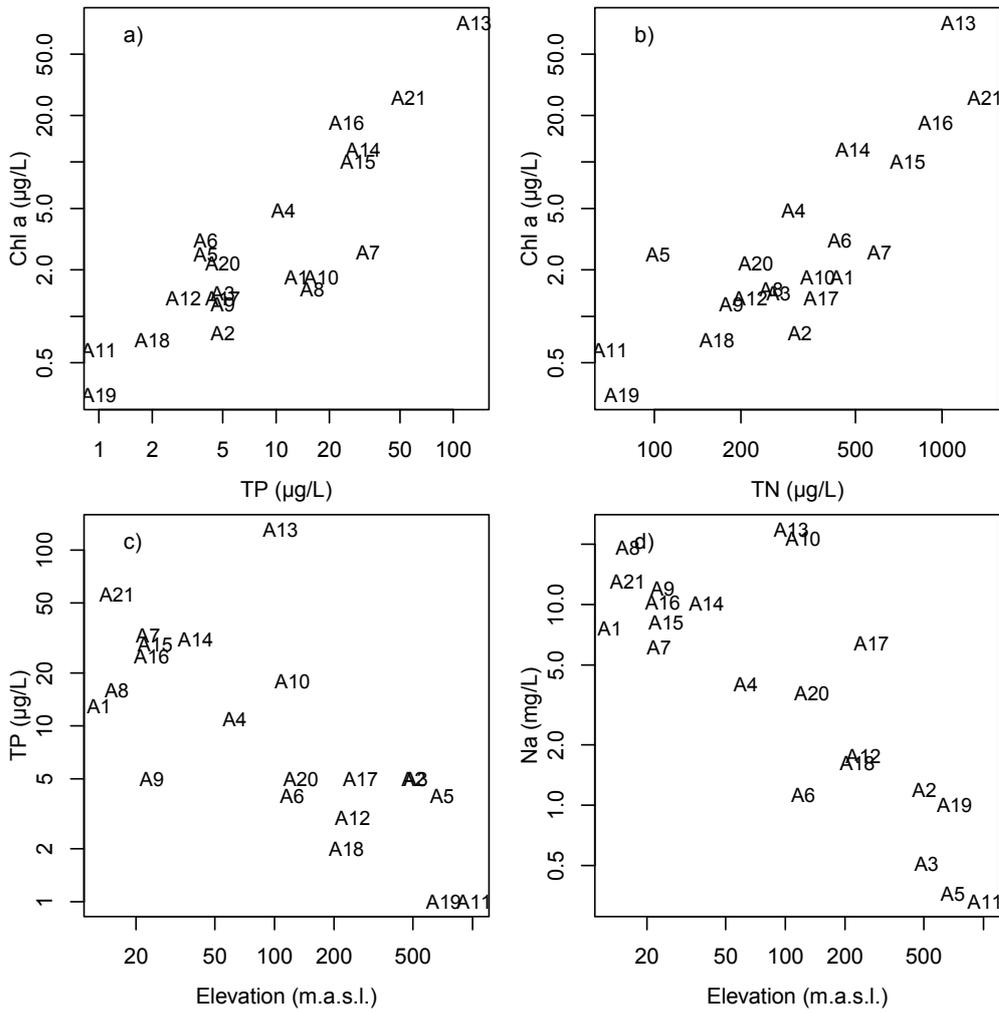


Figure 8. Select correlations between water chemistry parameters: a) chlorophyll *a* vs. TP, b) chlorophyll *a* vs. TN, c) TP vs. elevation, and d) Na vs. elevation. See Table 8 for Pearson’s correlation coefficients.

5.3 Amines in Norwegian lakes

5.3.1 Amines in surface waters: Methodological approaches

There are very few reported concentrations of amines in surface waters in the scientific literature, and as such, the current study was designed in order to provide critical information about the prevalence of amines in lakes. In particular, several remote lakes with low levels of human impact were included in order to provide an estimate of the range of background amine concentrations that exist in Norwegian freshwaters. Furthermore, we sought to test for relationships between lake characteristics, human and natural influences and amine concentrations through the pairing of analysis for select amines with characterization of general water chemistry and primary productivity.

The lack of available data on amines in surface waters is in part due to the analytical challenges associated with analysis of amines in complex environmental matrices, including surface waters. Several analytical approaches for the detection of amines have been presented in the literature, typically based on chemical derivatization followed by extraction of derivatives and detection (e.g. Sacher et al. 1997, Cai et al. 2003, Chang et al. 2012, Fu et al. 2012). These methods use a variety of derivatizing reagents as well as different methods for extraction (e.g. chemical or solid phase extraction), separation (e.g. gas chromatography (GC), liquid chromatography (LC) or electrophoresis) and detection (e.g. mass spectrometry (MS), fluorescence or laser-induced fluorescence). Chemical analysis of amines in environmental samples is particularly challenging due to low environmental concentrations, high volatility, low molecular weight, high polarity, instability and lack of chromophores (Chang et al. 2012).

5.3.2 Amine results from the current study

NILU sought to measure seven different amines in water from all study sites: methylamine (MA), ethylamine (EA), monoethanolamine (MEA), dimethylamine (DMA), diethylamine (DEA), 2-amino-2-methyl-propanol (AMP) and piperazine (PIP). Due to analytical challenges, uncertainty surrounding the measured amine concentrations, and development of analytical methods, as outlined in Section 4.3.2, several different methods were used at NILU for analysis of amines during the course of NIVA's collaborative amine work with NILU (including the baseline study (Grung et al. 2012), monitoring of TCM activities, and the current study). This has complicated the interpretation and comparison of measured amine concentrations for these studies.

The methodology employed at NILU for the amine-analyses was a combination of liquid chromatography (LC), which separates the compounds based on their inherent properties, followed by detection by mass spectrometry (MS). There are therefore two main factors that will influence the uncertainty in the instrumental aspect of the analysis; a) the ability of the chromatography to separate the compounds in time before the detection, and b) the ability of the mass spectrometer to distinguish between the mass to charge ratio (m/z) of the compounds of interest and interfering compounds.

When analysing compounds that are small or not so volatile, derivatisation of functional groups is often used to improve the chromatographic properties and/or to facilitate the volatility of the compound. For amine analyses, derivatisation was employed to improve the chromatographic separation, and to increase the m/z ratio for the amines, thereby increasing the specificity of the signal. The analytical challenge analyzing amines is that for most of these substances, the masses are so low that they are not particularly specific. The masses employed for detection can therefore have the same mass as fragmentation products of other compounds containing the same set of functional groups, and will therefore not be distinguishable from one another.

The methodology of amine analyses employed by NILU has evolved quite rapidly during the years since the first studies commenced (baseline study, Grung et al. 2012), and the method has improved substantially since then. Three major changes to the method employed for analysing amines have been the result of method development and a change of instrument.

- In an early phase, dansyl chloride was used as the derivatisation agent. Later, this was changed to benzenesulfonylchloride, which has shown to ease the process of derivatisation, increase the reaction product and produce more stable ions. All in all, this means that the sensitivity and specificity of the method has increased. The change of derivatisation agent meant that the m/z values measured had to be changed, and this seems to have minimised the tendency to analyse artefacts as part of the amines results.
- The LC part of the instrument was changed from a regular HPLC to a UHPLC (ultra-high performance liquid chromatography), which enabled improved chromatographic separation and therefore improved specificity of the method. At the same time, this allowed for a substantial reduction in the amount of time needed for analysis.
- The LC was coupled with a time-of-flight (ToF) mass spectrometer. The technique was developed decades ago, and although MS technology has not changed much during this time, recent rapid development of the electronic equipment used in the instrumentation has led to an improvement of the technique. The ToF instruments of today have a much better mass resolution (separation of one m/z from a nearby m/z) as well as mass accuracy (deviation from theoretical mass) than just a few years ago.

In an early phase of the amine-analyses at NILU (baseline study, Grung et al. 2012), the old HPLC instrument with ToF MS detection was used, along with dansyl chlorid as a derivatisation agent. In subsequent years, the method underwent several improvements (Table 4). The new LC/MS instrument with better sensitivity and chromatographic and mass resolution has improved the specificity of the method. The most current analytical method (since spring 2014) is theoretically therefore less hampered by the detection of artefacts than the earlier method. However, there is a high degree of uncertainty associated with the results of these amine analyses, and due to the wide variety of methods employed in the determination of amines at NILU, it is highly challenging to compare the results of different studies and different analytical techniques. In particular, the high amine concentrations observed in the baseline study as well as the current study may be driven by concurrent analysis of amine and artefact. The likely overestimation of these amine concentrations in the baseline and current study is also supported by recent results from our 2014 subset of samples (analyzed by both NILU and an independent lab), where amine levels were generally below detection.

Blank values are available for methods 2 and 4. Results from analysis of blanks revealed high concentrations of several amines, with the two methods yielding quite different results with respect to which amines were present at high concentrations (Table 9). The reasons for these elevated blank values are currently unclear and these challenges with obtaining low blank values adds to the uncertainty in the results of amine analyses in the current study,

Table 9. Concentrations (mean \pm s.d. in ng/L) of amines blank samples (for method 4, blanks reported were run on 27 June 2014).

	MA	EA	MEA	DMA	DEA	AMP	PIP
Method 2							
MilliQ Water	911 \pm 46	<40	993 \pm 109	8718 \pm 959	98 \pm 22	<100	589 \pm 71
Tap Water	725 \pm 36	<40	1048 \pm 105	3154 \pm 284	<50	<100	712 \pm 85
Method 4							
MilliQ Water	<10	160	140	200	1260	1280	<40
HPLC Water	<10	180	<130	210	1620	2320	<40
Reagents	<10	240	200	400	1460	<100	60

Due to this uncertainty in analyzed amine concentrations, we are unfortunately not able to report robust and quality assured data from amine analyses in the current study, nor are we able to assess seasonality in amine concentrations for samples collected for the seasonal study. However, in order to illustrate the challenges and changes in amine concentrations determined using the various analytical methods, we present data from the summer 2012 survey as analyzed using two different methods (Figure 9), and data from two of the survey lakes (A3 and A6) and four additional lakes that were sampled at several time-points and analyzed using multiple methods (Figure 10).

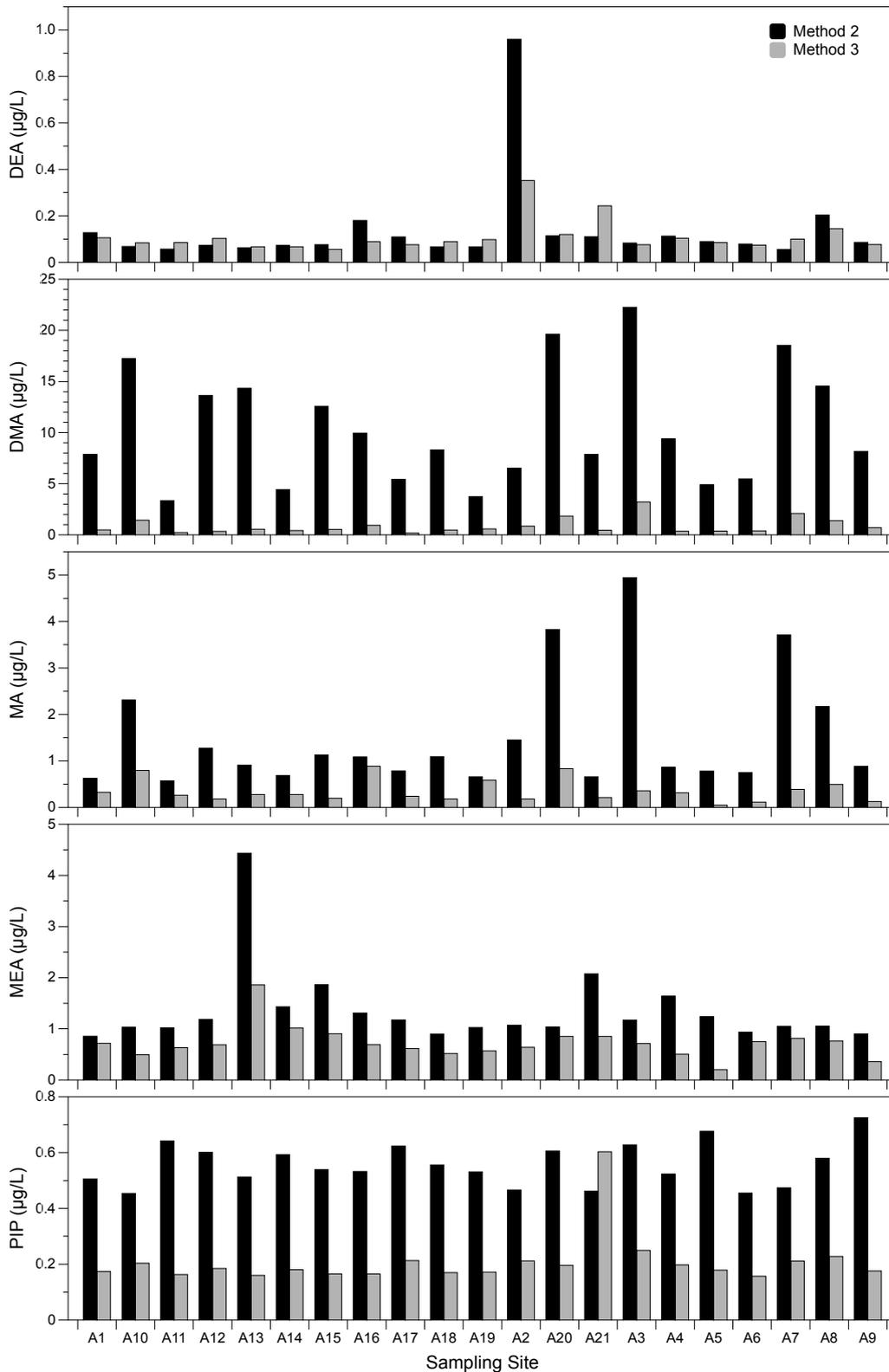


Figure 9. Concentrations of measured amines in samples collected during the summer 2012 survey using methods 2 and 3 (re-analysis of the same samples). EA and AMP concentrations were always below detection limits (<0.02 and <0.05 µg/L respectively). Concentrations are not blank corrected.

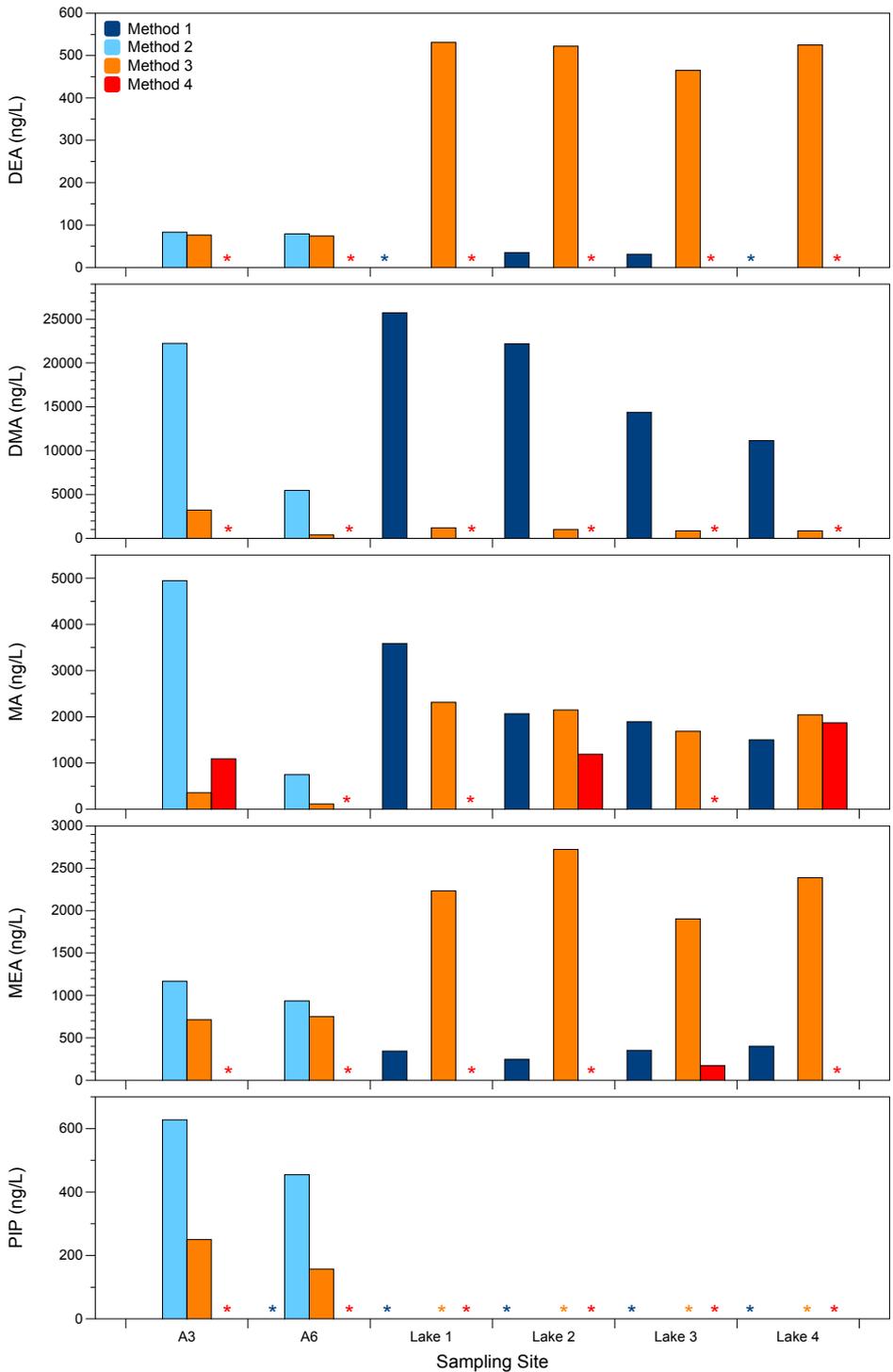


Figure 10. Concentrations of measured amines in samples collected from a subset of sites (including two sites from the 2012 survey, and four additional lakes). For A3 and A6, data for Methods 2 and 3 were from one re-analyzed sample from the summer 2012 survey. For Lakes 1–4, data for Method 1 are from samples collected in the baseline survey (Grung et al. 2012), while data for Method 3 are from samples collected in November 2013. All data for Method 4 are from samples collected in April 2014. Asterisks indicate that concentrations were below method detection limits. For method 4, detection limits for DEA, DMA, MA, MEA and PIP were 980, 270, 50, 130 and 40 ng/L respectively. Concentrations for methods 1 and 4 are blank corrected.

5.3.3 Relationships between amines and environmental parameters

We tested for relationships between concentrations of the seven measured amines and the water chemistry parameters included in the current study. We tested for relationships across all of the study lakes included in the summer 2012 survey to see whether these environmental parameters could explain the between-lake variability in concentrations of the measured amines. We also tested for relationships between environmental parameters and amine concentrations within the seasonal study lakes to assess whether seasonal variability in amine concentrations could be linked to seasonal changes in environmental conditions in these lakes. We found some very interesting results through these analyses (including evidence that concentrations of several amines were positively correlated to TOC concentrations, suggesting that catchment inputs of amines or amine precursors could be important in determining natural amine concentrations). However, due to the high degree of uncertainty surrounding the measured amine concentrations, we are not able to assess the validity of these observed relationships and seasonal patterns, and as such, they are not presented in this report. This is particularly unfortunate given the lack of such information in the literature and the strong need for knowledge on natural levels of amines in the aquatic environment, and potential environmental determinants of the presence of these compounds.

6. Conclusions

The study lakes chosen for analysis of amine concentrations were a good representation of the diverse range of conditions that exist in Norwegian lakes in terms of their water chemistry, exposure to various human activities and catchment land cover. The lakes provided gradients in several parameters, including: pH, trophic status, impact of acidification, and amount of humic matter (TOC). These gradients were used to assess the factors that influence amine concentrations in Norwegian lakes.

Despite a study design that would have provided key insight into some of the environmental and human factors that influence amine concentrations in lakes, our ability to draw conclusions regarding our questions of interest was strongly hampered by difficulties with the analytical methodology. Amines are particularly difficult to analyse in environmental samples, and these analytical challenges led to a high degree of uncertainty associated with the measured amine concentrations. Over the course of NIVA's collaborative amine work with NILU (including the baseline study (Grung et al. 2012), monitoring of TCM activities, and the current study), the rapid development of analytical methods at NILU has led to several different methods being used for analysis of amines over the course of these studies, complicating the interpretation and comparison of measured amine concentrations. Given the challenges associated with developing robust methodology for analysis of amines in complex environmental samples, including surface waters, future work should focus on improvement and validation of analytical methods. In particular, it would be useful to carry out an inter-lab comparison for analysis of amines in water samples.

The challenges we have encountered with obtaining reliable data on amine concentrations in water samples are extremely unfortunate, given that the data that this study aimed to provide would fill an important gap in the scientific literature and in our understanding of the determinants of natural background amine concentrations in surface waters, and of potential external and internal sources of amines to lakes. This information is particularly important for informing future work in the assessment of the importance and potential impact of future increases in amine loading to boreal lakes (e.g. due to amine-based CO₂ capture).

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8. Appendices

8.1 Abbreviations

AMP	2-amino-2-methyl-propanol
Chl α	chlorophyll α
Cl	chloride
DEA	diethylamine
DMA	dimethylamine
EA	ethylamine
LOD	limit of detection
MA	methylamine
MEA	(mono)ethanolamine
Na	sodium
NILU	Norwegian Institute for Air Research
NIVA	Norwegian Institute for Water Research
NO ₃	nitrate
PIP	piperazine
SO ₄	sulphate
TCM	CO ₂ Technology Centre Mongstad
TOC	total organic carbon
TN	total nitrogen
TP	total phosphorus

8.2 Personnel

The following people at NIVA participated in the project:

- **Merete Grung**, PhD – environmental toxicologist. Dr. Grung is a senior scientist with over 20 years of experience in analytical chemistry, environmental chemistry and environmental toxicology. Acted as the project leader.
- **Richard Wright**, PhD – environmental chemist. Dr. Wright is a senior research scientist with over 35 years of experience in hydrochemical research, including field studies, catchment-scale experiments and modelling. Acted as a senior advisor and provided quality assurance for the project.
- **Amanda Poste**, PhD – limnologist. Co-ordinated and carried out fieldwork, preparations for laboratory analysis, data analysis and played a central role in writing progress and final reports.
- **Espen Lund**, MSc – ecologist. Created maps related to the project, and collected background physicochemical data for lake selection.
- **Linda Marie Skryseth**, MSc – ecotoxicologist. Assisted with field sampling.
- **Tomas Adler Blakseth**, MSc – chemist. Blakseth is the acting research leader for the section for chemical analysis at NIVA and, along with colleagues in the NIVA lab, carried out chemical analysis and provided final data for major chemical components and chlorophyll *a* concentrations.
- **Andrew Harvey**, BA – assisted with fieldwork, and took the photos included in this report.

Amine analysis methods 1-4 were carried out at NILU, in the department for Environmental Chemistry (MILK). Analyses using NILU method 4 were carried out by **Pawel Rostkowski** (PhD).

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Norwegian Institute for Water Research

Gaustadalléen 21 • NO-0349 Oslo, Norway
Telephone: +47 22 18 51 00 • Fax: 22 18 52 00
www.niva.no • post@niva.no