

# Mercury isotope analysis of sediments from Gunneklevfjorden – Method evaluation and a preliminary investigation of sources



# RAPPORT

**Hovedkontor**

Gaustadalléen 21  
0349 Oslo  
Telefon (47) 22 18 51 00

**NIVA Region Sør**

Jon Lilletuns vei 3  
4879 Grimstad  
Telefon (47) 22 18 51 00

**NIVA Region Innlandet**

Sandvikaveien 59  
2312 Ottestad  
Telefon (47) 22 18 51 00

**NIVA Region Vest**

Thormøhlensgate 53 D  
5006 Bergen  
Telefon (47) 22 18 51 00

**NIVA Danmark**

Njalsgade 76, 4. sal  
2300 København S, Danmark  
Telefon (45) 39 17 97 33

Internett: [www.niva.no](http://www.niva.no)

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Forfatter(e) Hans Fredrik Veiteberg Braaten, Nathan W. Johnson, Francois Clayer, Bjørnar Beylich, Jarle Håvardstun, Morten Thorne Schaanning, Marianne Olsen	Fagområde Forurensninger	Distribusjon Åpen
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Oppdragsgiver(e) Hydro Energi AS	Oppdragsreferanse Thor Oscar Bolstad
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<p>Summary</p> <p>Gunneklevfjorden in south Norway is an aquatic system where the pollution history includes decades of local industry releases and emissions. An accurate estimate of the importance of ongoing and historic sources is critical in evaluating the potential success of sediment remediation strategies. Here, we utilize a high-resolution analytical method to investigate the isotopic composition of mercury (Hg) from a selection of potential Hg sources compared to the composition found in the fjord sediments. The distinct differences in the isotopes signature (<math>\delta^{202}\text{Hg}</math> and <math>\Delta^{199}\text{Hg}</math>) from the potential sources suggests that Hg in Gunneklevfjorden sediment and suspended particles in the water column largely has originated from a source with a signature similar to the chlorine factory waste at Herøya. The results also support the previous assumption that resuspended sediment contributes to the outflow of Hg from the fjord.</p>
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*Hans Fredrik Veiteberg Braaten*  
Prosjektleder

*Marianne Olsen*  
Forskningsleder

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# **Mercury isotope analysis of sediments from Gunneklevfjorden**

Method evaluation and a preliminary investigation  
of sources

## Preface

Sources of mercury to the environment are of both natural and anthropogenic origin, with anthropogenic sources including both local and global. The brackish fjord Gunneklevfjorden in south Norway is a system where the pollution history includes decades of local point industry releases and emissions as well as diffuse sources. Determining the origin of mercury in Gunneklevfjorden sediments and biota may be challenging considering the range of possible sources. However, an accurate estimate of the importance of ongoing and historic sources is critical to evaluate the potential success of sediment remediation strategies. Here, we utilize a high-resolution analytical method to investigate the isotopic composition of mercury from the fjord.

The work was led by Hans Fredrik Veiteberg Braaten (NIVA), which has written this report in collaboration with Nathan W. Johnson (University of Minnesota Duluth) and Francois Clayer (NIVA). The field work was undertaken by Bjørnar Beylich, Jarle Håvardstun and Marianne Olsen (all NIVA). Morten Thorne Schaanning (NIVA) and Marianne Olsen contributed with essential historical knowledge and to the design of the sampling campaign. Marianne Olsen has quality assured the report.

All samples were pre-treated at NIVA in Oslo before shipped to the US for further analysis. Total mercury concentrations and mercury isotopic composition of samples was determined at the United States Geological Survey Water Science Center laboratory in Middleton, WI, US. Concentrations of a selection of other metals were determined at Pace Analytical laboratory in Minneapolis, MN, US.

Project principal has been Norsk Hydro with Thor Oscar Bolstad as contact person. Thanks to all involved for a good cooperation.

Oslo, 26.09.2019

Hans Fredrik Veiteberg Braaten

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## Summary

Gunneklevfjorden in south Norway has a long pollution history, including decades of local industry releases and emissions. Determining the origin of mercury (Hg) in Gunneklevfjorden sediments and biota may be challenging considering the range of possible sources. However, an accurate estimate of the importance of ongoing and historic sources is critical to evaluate the potential success of sediment remediation strategies, and the success of future remediation actions, including capping with clean materials, depends on source control to prevent recontamination of the surface sediment. Previous investigations include an estimated mass balance explaining the export of Hg from Gunneklevfjorden to Skienselva and Frierfjorden. The mass balance model includes the assumption that resuspended sediment contributes significantly to the transport of Hg out of the fjord. However, this assumption has not been verified. Here, we utilize a high-resolution analytical method to assess whether a selection of potential sources of Hg to Gunneklevfjorden have distinct Hg isotope fingerprints, and if these signatures resembles the signatures of surface sediment and suspended particles. Samples for analysis were collected as close as possible to a selection of known previous and potential ongoing Hg sources surrounding Gunneklevfjorden, to compare with sediment samples from Gunneklevfjorden and relevant potential reference areas (nearby lake Svanstulvatnet and from open sea).

Measurements of total Hg (THg) demonstrated large variations but showed that concentrations were highest in sediments from Gunneklevfjorden (0.3–307 mg kg<sup>-1</sup>) and in soil from the old chlorine factory waste site (60–75 mg kg<sup>-1</sup>). The sediment concentrations are in correspondence with results from previous investigations in Gunneklevfjorden. The reference sediment samples from Svanstulvatnet (<0.4 mg kg<sup>-1</sup>) and the North Sea (<method detection limit) had lowest THg concentrations, while samples from the Eramet Norway site (on-going production of refined manganese (Mn) alloys) had intermediate concentrations (<0.1–5 mg kg<sup>-1</sup>). Comparing the inverse concentrations of Hg (1/THg) with mass dependent and mass independent fractionation of Hg isotopes (MDF,  $\delta^{202}\text{Hg}$ , and MIF,  $\Delta^{199}\text{Hg}$ ) in our samples revealed the emergence of two distinct isotopic fingerprints among the samples having concentrations exceeding 1 mg kg<sup>-1</sup> THg. One group encompassed samples from the Gunneklevfjorden sediment and Herøya chlorine factory waste (THg>5 mg kg<sup>-1</sup> and  $\delta^{202}\text{Hg}$ >-0.6 ‰) and a second group encompassed samples from various Eramet locations (THg 1–5 mg kg<sup>-1</sup> and  $\delta^{202}\text{Hg}$  -1.7 – -2.5 ‰).

Other metals were analyzed to support the interpretation of the isotope results. Of other metals, samples from the Eramet site had significantly higher Mn concentrations (183.7±120.8 mg g<sup>-1</sup>) compared to other samples from Herøya (2.2–2.9 mg g<sup>-1</sup>) and Gunneklevfjorden (1.8±1.7 mg g<sup>-1</sup>). Concentrations of lead (Pb) showed less variation between sample locations, and a plot of Pb versus Mn concentrations for each sample taken at the various sites show a distinct separation of two groups of samples. This pattern is also demonstrated through a Principal Components Analysis (PCA), where vectors for Herøya and Gunneklevfjorden are very similar and grouped together whereas those for Eramet are diverging. This supports the idea that Herøya and Gunneklevfjorden have similar geochemical signatures, whereas Eramet represents a different signature.

By pairing MDF ( $\delta^{202}\text{Hg}$ ) and MIF ( $\Delta^{199}\text{Hg}$ ) isotopic signatures and comparing the values with known values for typical Hg sources from the literature, data confirm that samples from the Gunneklevfjorden area fall into two distinct isotopic groups. This includes samples with a signature typical for contamination of Hg from heavy metal refining and metallic Hg use (i.e. Gunneklevfjorden sediments and Herøya waste soil) and samples with a signature that are overlapping signatures reported of Hg from European coals (i.e. Eramet samples). The differences in both MDF and MIF between Gunneklevfjorden sediment/Herøya soil and samples from Eramet appear to be large enough that fractionation caused by natural processes is unlikely to explain the pattern. Our data suggests that Hg in sediment and suspended particles from Gunneklevfjorden largely has originated from a source with a signature similar to the chlorine factory waste at Herøya. The analytical method should be further assessed as a tool for tracking Hg into the food web, and as a potential tool for post-remediation monitoring.

## Sammendrag

Gunneklevfjorden i Porsgrunn kommune, Telemark, har en lang forurensningshistorie som inkluderer flere tiår med lokale utslipp av kvikksølv (Hg) til luft, vann og jord fra lokale industrikilder. Å bestemme kildene for Hg som finnes i sediment og biota (for eksempel fisk) i Gunneklevfjorden er komplisert, da Hg har mange potensielle kilder, både antropogene og naturlige, lokale og langtransporterte. En slik kildesporing er imidlertid viktig for å kunne estimere påvirkningen fra nåværende og historiske kilder ved planlegging av tiltak mot forurenset sjøbunn i Gunneklevfjorden, og effekten av tiltak på sikt. Tidligere undersøkelser og utredninger har inkludert en massebalanse-modell som beskriver eksport av Hg fra Gunneklevfjorden til Skienselva og Frierfjorden. Modellen legger til grunn en antagelse om at løst og re-suspendert materiale fra sjøbunnen bidrar vesentlig til eksporten av Hg ut av fjorden. I denne studien har vi benyttet en høy-oppløselig analytisk metode for å vurdere hvorvidt potensielle kilder til Gunneklevfjorden har ulike isotopsignaler for Hg, og om disse signaturene sammenfaller med signaturer i sediment og suspendert partikulært materiale i Gunneklevfjorden. Prøver ble samlet inn fra kjente og potensielle Hg-kilder i nærheten av Gunneklevfjorden, samt fra potensielle referanseområder.

Konsentrasjonene av Hg viste stor variasjon mellom de undersøkte prøvene. Nivåene var høyest i sedimentprøver fra Gunneklevfjorden ( $0,3\text{--}307\text{ mg kg}^{-1}$ ) og i prøver av avfall fra klorfabrikken på Herøya ( $60\text{--}75\text{ mg kg}^{-1}$ ). Konsentrasjonene fra Gunneklevfjorden er i samsvar med resultater fra tidligere undersøkelser. Referansesediment fra Svanstulvatnet ( $<0,4\text{ mg kg}^{-1}$ ) og Nordsjøen ( $<$ analytisk deteksjonsgrense) hadde lavest konsentrasjoner, mens prøver fra Eramet Norges område for produksjon av mangan (Mn) på Herøya varierte fra  $<0,1$  til  $5\text{ mg kg}^{-1}$ . En sammenligning av prøvenes inverterte Hg-konsentrasjoner ( $1/\text{Hg}$ ) med masseavhengig og masseuavhengig fraksjonering av Hg-isotoper (MDF,  $\delta^{202}\text{Hg}$ , og MIF,  $\Delta^{199}\text{Hg}$ ) avdekket to tydelige «isotopiske fingeravtrykk» i prøvene der konsentrasjonene overskred  $1\text{ mg kg}^{-1}$  Hg. Den ene gruppen inkluderte sedimentprøver fra Gunneklevfjorden og prøver av avfall fra klorfabrikken på Herøya ( $\text{Hg}>5\text{ mg kg}^{-1}$  and  $\delta^{202}\text{Hg}>-0.6\text{ ‰}$ ), mens den andre gruppen inkluderte ulike prøver fra Eramets område på Herøya ( $\text{Hg } 1\text{--}5\text{ mg kg}^{-1}$  and  $\delta^{202}\text{Hg } -1.7\text{ -- } -2.5\text{ ‰}$ ).

Analyse av andre metaller ble inkludert som støtteparametere i tolkningen av resultatene. Av andre metaller viste undersøkelsen at prøver fra Eramet som forventet hadde signifikant høyere konsentrasjoner av Mn ( $183,7\pm 120,8\text{ mg g}^{-1}$ ) sammenlignet med prøver fra Herøya ( $2,2\text{--}2,9\text{ mg g}^{-1}$ ) og Gunneklevfjorden ( $1,8\pm 1,7\text{ mg g}^{-1}$ ). Forskjellene i konsentrasjoner av andre metaller, for eksempel bly (Pb), mellom de ulike prøvegruppene var mye lavere, og et plot av Pb- mot Mn-konsentrasjoner for alle prøvene viste en separasjon av to distinkte prøvegrupper. Dette mønsteret ble også dokumentert gjennom en prinsippal komponent analyse (PCA), der vektorer for prøver fra den gamle klorfabrikken og Gunneklevfjorden var gruppert og forskjellige fra vektorene for prøvene fra Eramet. Et slikt resultat understøtter at prøvene fra den gamle klorfabrikken på Herøya og Gunneklevfjorden har lik geokjemisk signatur, og at prøvene fra Eramet skiller seg fra disse.



Ved å kombinere MDF ( $\delta^{202}\text{Hg}$ ) og MIF ( $\Delta^{199}\text{Hg}$ ) av Hg-isotoper og sammenligne verdiene med kjente verdier for typiske Hg-kilder fra litteraturen bekrefter våre data at prøvene fra område i og rundt Gunneklevfjorden tilhører to distinkte isotop-grupper. Dette inkluderer prøver med en signatur som er typisk for Hg-forurensing fra raffinering av tungmetaller og bruk av metallisk Hg (Gunneklevfjordens sediment og avfall fra klorfabrikken) og prøver med en signatur som overlapper med Hg-forurensing fra europeisk kullforbrenning (Erametprøver). Forskjellen i MDF og MIF mellom sediment fra Gunneklevfjorden/klorfabrikken og prøver fra Eramets område på Herøya fremstår som stor nok til at mønsteret ikke kan forklares ved fraksjonering av Hg-isotoper gjennom naturlige prosesser. Resultatene i denne rapporten indikerer at hovedkilden til Hg i sedimenter fra Gunneklevfjorden i stor grad er kilder med signatur lik den fra den gamle klorfabrikken på Herøya. Metoden som er benyttet bør vurderes videre som et verktøy for å spore Hg inn i næringskjeden, og som et verktøy for overvåking etter tiltak.

Tittel: Bestemmelse av kvikksølv-isotoper i sediment fra Gunneklevfjorden – metodeevaluering og undersøkelse av mulige kilder

År: 2019

Forfatter(e): Hans Fredrik Veiteberg Braaten, Nathan W. Johnson, Francois Clayer, Bjørnar Beylich, Jarle Håvardstun, Morten Thorne Schaanning, Marianne Olsen

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# 1 Introduction

## 1.1 Mercury isotopes in the environment

Mercury (Hg) is an environmental pollutant of global concern, posing a potential serious threat for human health and the environment. Sources of Hg to the environment are of both natural and anthropogenic origin, with anthropogenic sources including both local (e.g. industry releases and emissions) and global (i.e. long-range transported atmospheric Hg) (Streets et al., 2011). Determining the origin of Hg at a given site (e.g. lake, fjord system etc.) may be challenging considering the range of possible sources. However, high-resolution analytical methods have become a popular tool for accurately determining the isotopic composition (fingerprint) of Hg. In cases when Hg sources have different isotopic composition, the origin of Hg sources to sediment, water, and biota can often be unambiguously identified.

Hg exists naturally in the environment as seven stable isotopes which, when subject to certain chemical, physical, and biological reactions, are altered in their abundance relative to one another (Blum and Johnson, 2017). This relative abundance of heavy and light isotopes is termed ‘fractionation.’ The fractionation of Hg typically follows mass dependent fractionation (MDF) in which lighter isotopes will react faster and become enriched in the product; commonly  $\delta^{202}\text{Hg}$  is used to represent MDF (Blum and Bergquist, 2007). This tracer has been used in previous studies for the identification Hg sources in aquatic ecosystems (Wiederhold, 2015; Yin et al., 2014) including applications to track Hg contamination from mining activities (Foucher et al., 2009; Gehrke et al., 2011), coal combustion (Bartov et al., 2012), and chlor-alkali processes (Mil-Homens et al., 2013; Perrot et al., 2010).

Additionally, Hg can undergo processes such as nuclear volume and magnetic isotope effects which cause deviations from MDF predicted trends. This deviation from alterations to relative abundance based on mass difference is termed mass independent fractionation (MIF) and is typically represented as  $\Delta^{\text{xxx}}\text{Hg}$  (Bergquist and Blum, 2009). Most commonly MIF is observed in the odd-isotopes of Hg ( $^{199}\text{Hg}$  and  $^{201}\text{Hg}$ ), and odd-MIF occurs in samples from natural systems where it relates to photochemical reactions that Hg has undergone in the atmosphere or water column (Bergquist and Blum, 2007). MIF in even-isotopes (even-MIF) has been observed in  $^{200}\text{Hg}$  and  $^{204}\text{Hg}$  which are linked to atmospheric reservoirs and long-range transport of gaseous Hg (Blum and Johnson, 2017). The differences in fractionation patterns from these multiple Hg isotopes have allowed for improved source identification using various tracer approaches (MDF, odd-MIF) (Lepak et al., 2015).

## 1.2 Potential sources of mercury around Gunneklevfjorden

Gunneklevfjorden has been one of two major recipients for releases from different industries located on Herøya in Telemark (Olsen et al., 2015). It is estimated that between 20 and 30 tons of Hg are stored in the Gunneklevfjorden sediments, and surveys from the late 1970s revealed high concentrations of Hg and several other contaminants (Skei, 1978). Over the last few years, new surveys reveal much lower concentrations in surface sediment (Olsen et al., 2015), but still levels higher than relevant environmental quality standards (EQS). Surveys also reveal elevated concentrations of Hg in the fjord perch (*Perca*

*fluviatilis*) population, significantly higher than what is observed in nearby lakes without known local pollution sources (i.e. only influenced by long-range transported atmospheric Hg) (Braaten et al., 2017). Tracing of Hg isotopes in the Gunnekelevfjorden ecosystem could provide valuable insight for determining the importance of different sources of Hg to the Gunnekelevfjorden sediments and biota and for evaluating remediation alternatives.

Previous investigations include an estimated mass balance explaining the export of Hg from Gunnekelevfjorden to Skienselva og Frierfjorden (Olsen et al., 2015). In the mass balance, the following sources were estimated: atmospheric input, storm water/run off from urban area, storm water from Herøya, cooling water from Yara (this contributes some Hg mass, but net effect is to dilute and wash out Hg from the fjord), inflow from Skienselva and Frierfjorden (same effect as cooling water), and flux from sediment to water. Other potential sources that were not estimated include groundwater flows from Herøya and potentially episodic airborne dust from nearby Eramet Norway's production of refined manganese (Mn) alloys. All estimates for contributions from these sources were made based on measurements of Hg concentration and annual water flow, but the eventual fate of the Hg from these sources, in relation to the Hg found suspended in the Gunnekelevfjorden water or in surficial sediment, is presently unknown. The mass balance model includes the assumption that resuspended sediment contributes significantly to the transport of Hg out of the fjord. However, this assumption has not been verified.

### **1.3 Project aims and goals**

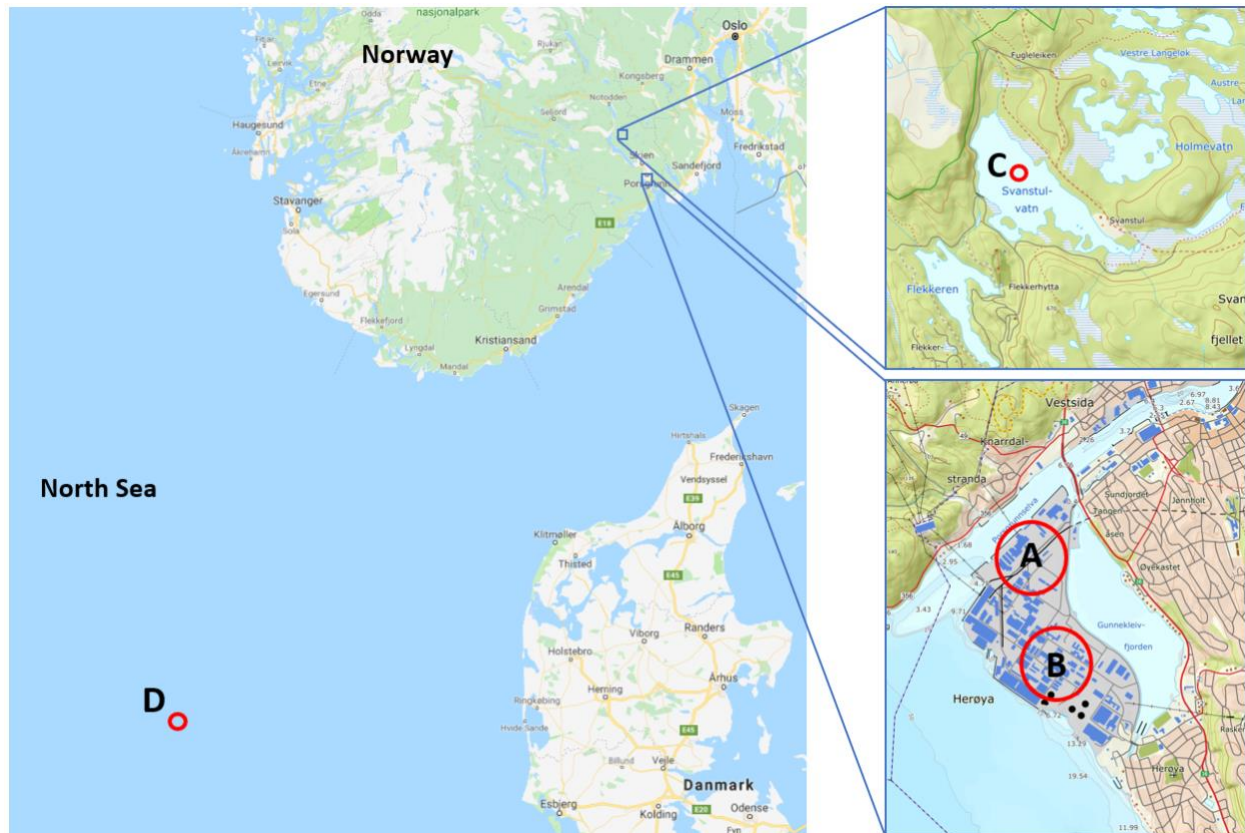
This work is one of several tasks aiming to strengthen the knowledge base for predicting effects from planned contaminated sediment remediation measures in Gunnekelevfjorden. Thus, an overall goal for this study is to enhance knowledge of potential Hg sources, as well as seek to verify the assumption about the significant contribution of resuspended particles to export of Hg, presented in the previous mass balance model. The main objective was to evaluate the use of a high-resolution analytical method to assess whether the potential sources of Hg to Gunnekelevfjorden surface sediment have distinct Hg isotope fingerprints, and if the signatures of Gunnekelevfjorden sediment and suspended particles resemble these signatures.

The approach included comparing the Hg isotope fingerprint of surface and suspended sediment in Gunnekelevfjorden with Hg isotope fingerprints in a selection of known previous and potential ongoing sources. Samples were collected as close as possible to a selection of known and potential Hg sources, to compare with sediment samples from Gunnekelevfjorden and relevant potential reference areas (nearby lake Svanstulvatnet and from open sea). The reference areas were selected to represent different sites with no known direct discharges of Hg. Samples were analyzed for Hg isotopes and also a suite of metals to enable a detailed discussion of differences between samples.

## 2 Materials and methods

### 2.1 Gunneklevfjorden

Gunneklevfjorden is a small (0.7 km<sup>2</sup>), shallow (max. depth 11 m), and brackish fjord located in southern Norway (**Figure 1**) that has a long history as recipient for industrial discharges and emissions. The fjord was heavily contaminated between 1947 and 1987, due to releases from a chlor-alkali plant at nearby Herøya of approximately 80 t of Hg to the fjord (Skei, 1989). Reported maximum concentrations of total Hg (THg, 492 mg kg<sup>-1</sup>) and methyl-Hg (MeHg, 269 µg kg<sup>-1</sup>) are found in intermediate sediment depths (10-20 cm), reflecting the historical discharges to the fjord. Natural recovery has resulted in reduced surface sediment (0-5 cm) concentrations of both THg (<10 mg kg<sup>-1</sup>) and MeHg (<5 µg kg<sup>-1</sup>) (Olsen et al., 2018). In general, concentrations of Hg (3.4 ng L<sup>-1</sup>) and MeHg (0.06 ng L<sup>-1</sup>) in the aqueous phase are low (Braaten et al., 2017). The sediments are recognized as the main Hg sources to the water column (Olsen et al., 2015; Olsen et al., 2019).



**Figure 1** A map showing an overview of sampling locations included in the present study. Capital letters indicate the four main sampling areas, including A: Eramet area at Herøya Industry Park (HIP), Porsgrunn; B: Old chlorine factory waste, HIP; C: Reference station Svanstulvatnet, Skien; D: Reference station North Sea. Sources: google maps and kartverket.no.

## 2.2 Field sampling

A total of 52 sediment samples (see **Appendix Table A1** for a detailed overview) were collected, freeze dried, and sent to the lab at the Wisconsin Water Science Center (USGS, Madison, WI, USA) for analysis of Hg isotopes. Samples were collected from several different locations (see **Figure 1** for an overview and **Appendix** for detailed locations) within the present operations of Eramet Norway (25 samples, **Appendix Figure A2**) and Herøya (2 samples, near chlorine factory waste) sites. In Gunneklevfjorden (**Appendix Figure A3**), samples were collected from three sediment traps approximately 1.5-2m above the sediment surface (at 5-6 m water depth) in a cross section in the middle area of the fjord (4 samples). The sediment traps were left in the fjord from December 2017 to May 2018. In addition, intact duplicate sediment cores were collected from four locations across the fjord, and sliced into samples representing surface sediment (0-5 cm, 2x4 samples) and deep sediment (20-25 cm depth, 2x4 samples). In addition, a few surface sediment samples from reference locations Svanstulvatnet (59.39425, 9.42537 (WGS84, latitude, longitude), 3 samples) and the North Sea (56.02930, 5.04626, 2 samples, **Figure 1**) were included in the study.

## 2.3 Analytical methods

Freeze dried homogenized sediments were weighed out and digested with concentrated aqua regia (3:1 hydrochloric acid (HCl):nitric acid (HNO<sub>3</sub>)) in 40 mL glass vials (90°C, 8 hours). Samples were subsequently diluted with ultrapure water (18.2 MΩ) to a final acid content of 50 % H<sup>+</sup>. Digests were analyzed for total Hg (THg) concentration via tin(II)chloride (SnCl<sub>2</sub>) reduction coupled to gold trap amalgamation and analysis via cold vapor atomic fluorescence spectroscopy (CVAFS) (USEPA, 2002) using an automated system.

Hg isotope measurements were performed using a Thermo Neptune plus Multicollector inductively coupled plasma mass spectrometer (MC-ICP-MS). The samples were diluted to a final concentration of 1 ng mL<sup>-1</sup> and an acid content <15%. Samples were bracketed with the NIST 3133 Hg standard, such that standards were concentration and matrix matched to samples. Hg solutions were introduced continuously to a custom-made gas liquid separator (Yin et al., 2016) simultaneously with 3% SnCl<sub>2</sub> in 10% HCl. Gaseous Hg produced from the reduction was released from the aqueous phase using a counter Argon (Ar) gas flow. Thallium (Tl, 40 ng mL<sup>-1</sup>) was also introduced using an Apex Q desolvating nebulizer to the gas liquid separator for mass bias correction. Instrument parameters were tuned for maximized Hg signal (1V ~ 1 ng mL<sup>-1</sup>) and stability. To ensure precision and accuracy a secondary Hg isotope standard (UM NIST RM 8610) was measured every 5 samples and certified reference sediment (NIST 1944) were used to verify digest efficiency. Hg concentrations and isotope values for secondary standards agreed with previously published values (Janssen et al., 2019; Lepak et al., 2015).

A separate aliquot (0.2 to 0.3 g) of homogenized and split sediment sample was digested in 12 mL of aqua regia (3:1 v:v HCl:HNO<sub>3</sub>) heated to 95 °C for 30 minutes after United States Environment Protection Agency (USEPA) method 3050B. Further, samples were filtered (0.45 um polyethersulfone), and diluted to 100 mL in 0.5 % HNO<sub>3</sub>. Metals were quantified at Pace Analytical Services by ICP-MS after USEPA method 6020B

following dilution of an aliquot of filtered digestate. All quality assurance (QA) for metals fell within reporting requirements.

Three of the analyzed samples (3A, 3B, and 18A) were problematic for the multi-collector ICP-MS and are undergoing a re-analysis. Since these samples belong to groups for which other samples were already quantified, it is expected that the forthcoming results will not change the interpretation significantly.

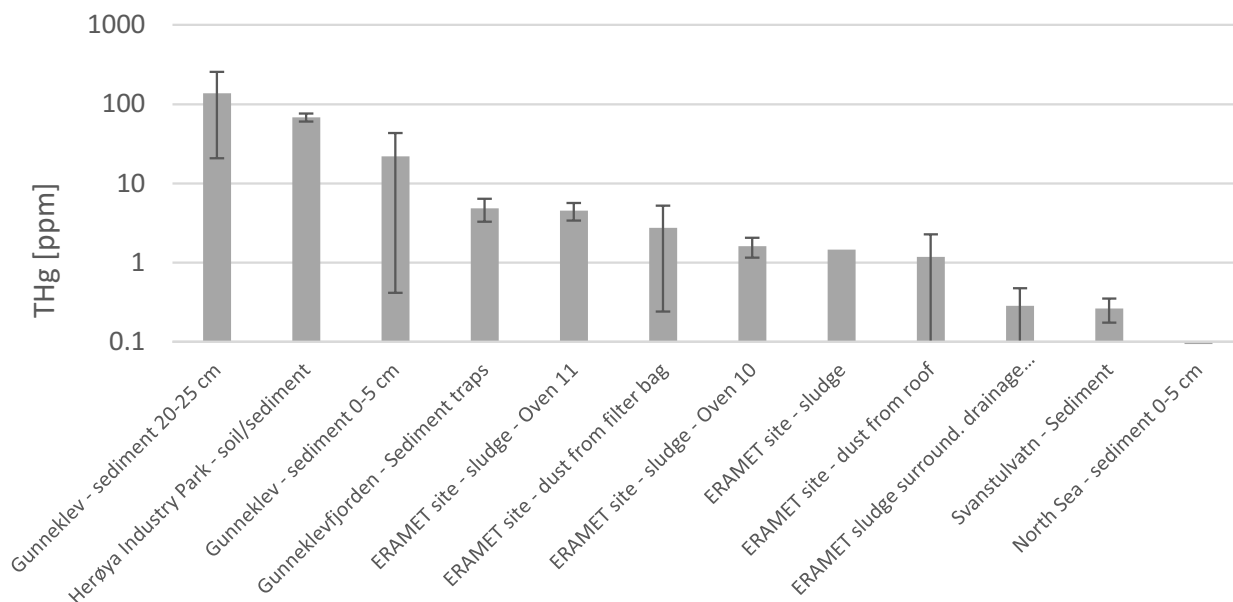
## **2.4 Statistical analysis**

Metal concentrations in the samples collected in the Gunneklevfjorden, and at Eramet Norway and Herøya sites were analyzed using a Principal Component Analysis (PCA). PCA allows to represent the variance in the dataset by a limited number of orthogonal vectors and assumes that compositional variation within the dataset is real and meaningful (Miesch, 1980). PCA identifies a smaller number of uncorrelated variables, called "principal components" (PC), from a large set of data. With this analysis, new variables, i.e., PC, are created as linear combinations of the observed variables. The goal of PCA is to explain the maximum amount of variance with the fewest number of PC. Prior to the PCA, the dataset was normalized by dividing each metal concentration by the average concentration of this metal.

## 3 Results and discussion

### 3.1 Total mercury sediment concentrations

Concentrations of THg in sediments from the sites in the Gunneklevfjorden area ranged from 0.3 mg kg<sup>-1</sup> at the Eramet site to 307 mg kg<sup>-1</sup> in deep sediments (**Figure 2**). The Gunneklevfjorden concentrations are in accordance with previous investigations (Olsen et al., 2018). The deep Gunneklevfjorden sediment (20 to 25 cm) was variable but had the highest average THg content. Deep sediment at locations 12 and 23 (mid-fjord: nearshore, SW and NE) exceeded 200 mg kg<sup>-1</sup>, but deep sediment at location 19 (mid-fjord offshore) exceeded 30 mg kg<sup>-1</sup>. Two samples from Herøya Industrial Park material (from old chlorine factory) had THg between 60 and 75 mg kg<sup>-1</sup>. Surface sediment (0 to 5 cm) THg in Gunneklevfjorden was also variable, ranging from < 10 mg kg<sup>-1</sup> at location 12 to more than 50 mg kg<sup>-1</sup> at location 23 (mid-fjord). Previous investigations showed a similar variation of THg concentrations in top sediments (0-5 cm) throughout the fjord, ranging from 0.2 to 142 mg kg<sup>-1</sup> (mean ± one standard deviation: 54.2±35.2 mg kg<sup>-1</sup>, n=22) (Olsen et al., 2018). Olsen et al. (2018) found that concentrations decreased from the surface down to peak concentrations at 10-15 cm sediment depth (491 mg kg<sup>-1</sup>). THg in the sediment traps material (6 samples from north, mid, south) were consistently between 3 and 7 mg kg<sup>-1</sup>.



**Figure 2** Total mercury (THg) concentrations (log scaled) in the Gunneklevfjorden area sediment samples. Error bars represent one standard deviation of the mean for each sample type.

THg in Eramet samples was highest in the sludge from the ovens (Oven 10 and 11), and variable in dust from the filter bag (P-SENT 11 was consistently ~5 mg kg<sup>-1</sup>, while P-SENT 10 was < 0.75 mg kg<sup>-1</sup>). Dust from the oven-house roofs was variable (0.3 to 3 mg kg<sup>-1</sup>) and was highest near oven houses #3 and #4. THg in

sludge surrounding the chambers (2, 5, 6, and 284) had between 0.1 and 0.5 mg kg<sup>-1</sup>. Reference sediment from Svanstulvatnet had THg < 0.4 mg kg<sup>-1</sup> and the North Sea sediment was below detection limits.

### 3.2 Isotope measurements in the Gunneklevfjorden area

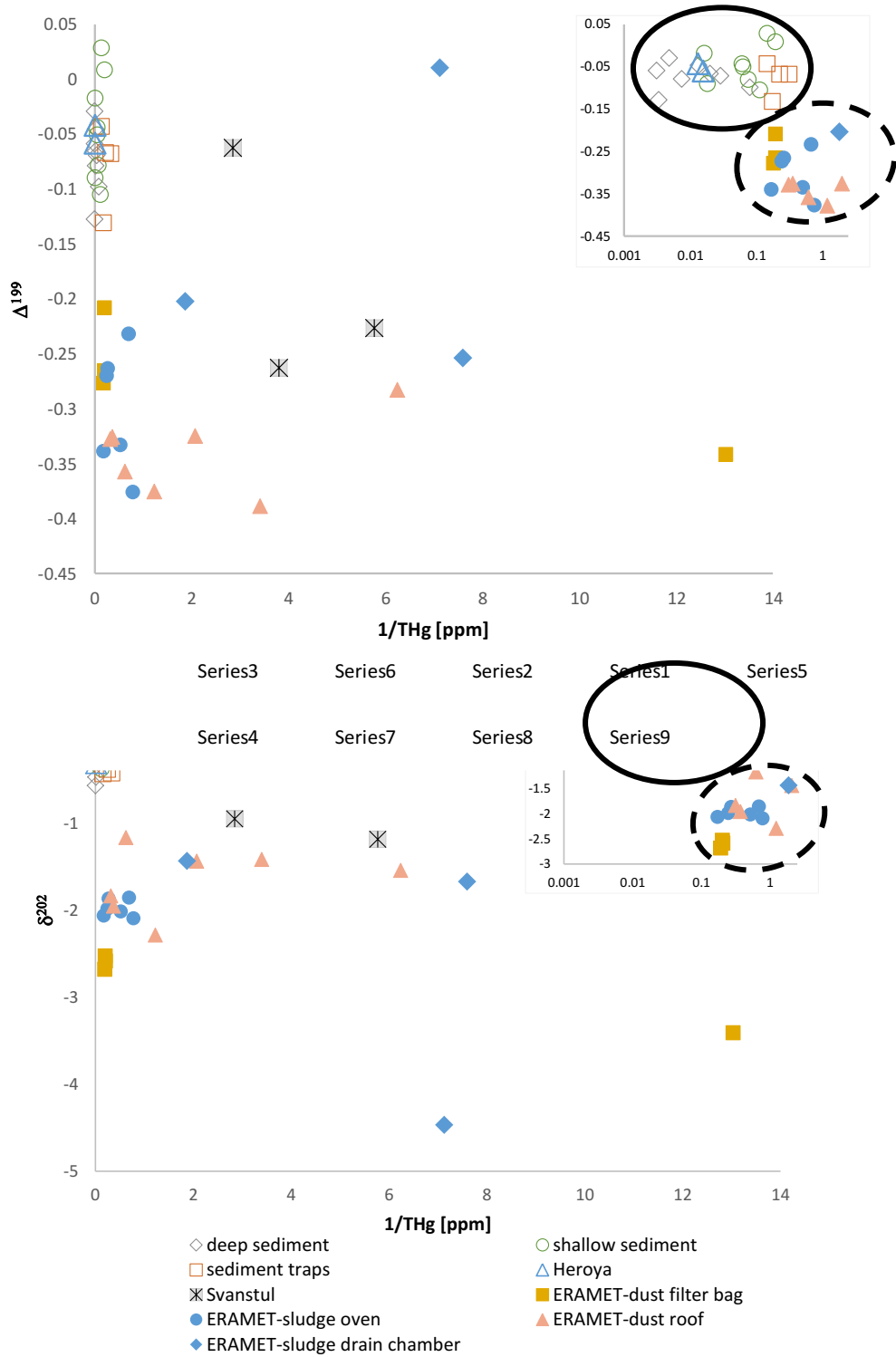
Isotopes of Hg with a molecular weight of 196 and 198 were not measured in sediment of the Gunneklevfjorden area due to their low natural abundance, leaving 5 stable isotopes (199, 200, 201, 202, 204) to describe MDF and MIF. Mixing plots of inverse concentrations (1/THg) and  $\Delta^{199}\text{Hg}$  and  $\delta^{202}\text{Hg}$  (**Figure 3**) show the emergence of two distinct isotopic fingerprints among those samples having concentrations exceeding 1 mg kg<sup>-1</sup> THg. One group encompassed samples from the Gunneklevfjorden sediment cores (shallow and deep and sediment traps) and Herøya chlorine factory waste, having > 5 mg kg<sup>-1</sup> THg and  $\delta^{202}\text{Hg}$  values above -0.6 ‰ (solid circles in **Figure 3**). The second group encompassed samples from various locations at Eramet and had THg concentrations between 1-5 mg kg<sup>-1</sup> and a more depleted isotopic composition ranging from -1.7 to -2.5 ‰ for  $\delta^{202}\text{Hg}$  (dashed circles in **Figure 3**). Some samples from Eramet dust and drainage chamber sludge had lower concentrations of THg (<1 mg kg<sup>-1</sup>, 1/THg>1) but maintained an isotopically depleted (more negative)  $\delta^{202}$  and  $\Delta^{199}$  signature. The only exception to this was the sample from near Chamber #284 (finished material) which had an extremely depleted  $\delta^{202}$  value (-4.5 ‰) but a relatively enriched  $\Delta^{199}$  (+0.1 ‰, similar to Gunneklevfjorden sediment).

Compared to the other samples from this study, reference sediment from Svanstulvatnet had, on average, intermediate  $\delta^{202}$  (-0.74 ‰) and  $\Delta^{199}$  (-0.18 ‰) (**Figure 3**). The signatures of these reference sediment are consistent with terrestrial Hg which has undergone MDF and MIF from landscape-scale volatilization processes. Previous observations of continental compartments such as soil and sediments that mainly received Hg from atmospheric deposition have shown predominantly negative MIF (summarized in Yin et al., 2014). However, the sediment from Svanstulvatnet is from a distant location to Gunneklevfjorden and may not be representative of the local watershed source. The surface sediment in Gunneklevfjorden had, on average, slightly higher THg concentrations and an enriched (higher) MIF and MDF signature. It is, at this point, difficult to discern whether these slight enrichments (relative to deep sediment, Herøya, and sediment traps) is due to another source (watershed-derived Hg, for instance) or due to physical, chemical, or biological processes that may have enriched the MIF and MDF in Hg in surficial sediment.

Negative  $\Delta^{199}\text{Hg}$  measured within many of the Gunneklevfjorden and Herøya samples (-0.12 to 0.03 ‰) are mostly within analytical error for the measurements. Odd-MIF values ( $\Delta^{199}$ ) are typically near zero in Hg derived from industrial usage particularly metallic Hg catalysts (Mil-Homens et al., 2013; Perrot et al., 2010; Sonke et al., 2010) which may be the case in these sites. The samples from Eramet consistently displayed significantly negative  $\Delta^{199}\text{Hg}$  values (<0.20 ‰). Negative  $\Delta^{199}\text{Hg}$  has been documented in two pools of environmental Hg: unprocessed coal (fly ash) (Sun et al., 2014) and atmospherically derived Hg (gaseous elemental, litterfall, and soils) (Blum and Johnson, 2017; Demers et al., 2013; Jiskra et al., 2015; Obrist et al., 2017). No significant even-MIF ( $\Delta^{200}\text{Hg}$ ) signals were observed in any of the samples from the Gunneklevfjorden area. This even-MIF signature is commonly associated with precipitation (+ $\Delta^{200}\text{Hg}$ ) and gaseous elemental Hg (- $\Delta^{200}\text{Hg}$ ) in the atmosphere (Blum and Johnson, 2017). The absence of this signal



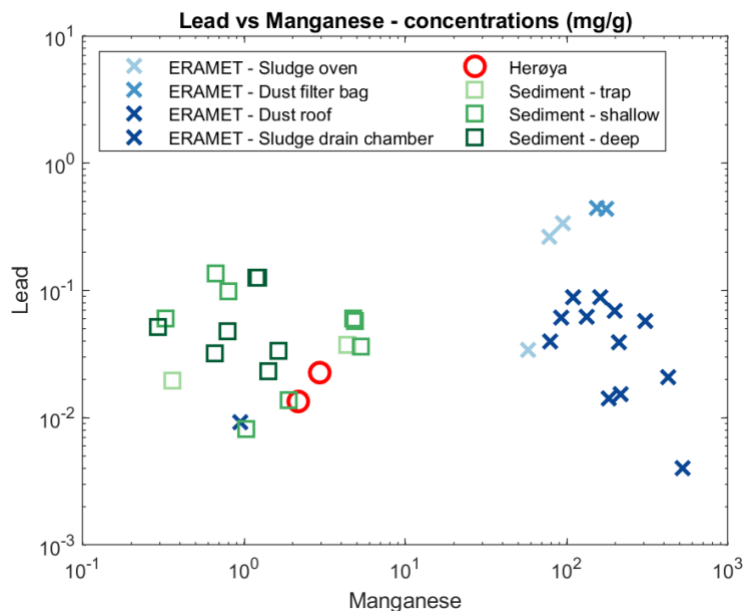
suggests little influence of atmospheric deposition as many soils and litterfall in remote regions typically display  $-\Delta^{200}\text{Hg}$  values (Demers et al., 2013; Jiskra et al., 2015).



**Figure 3** Mixing plots for  $\delta^{202}\text{Hg}$  (bottom panel, y-axis) and  $\Delta^{199}\text{Hg}$  (top panel, y-axis) in relation to inverse concentration of THg in sediments ( $1/\text{THg}$ , x-axis). Samples from Eramet (closed symbols) have distinctly lower THg concentrations and lower  $\delta^{202}\text{Hg}$  and  $\Delta^{199}\text{Hg}$  compared to samples from Herøya and Gunneklevfjorden sediments.

### 3.3 Other metals

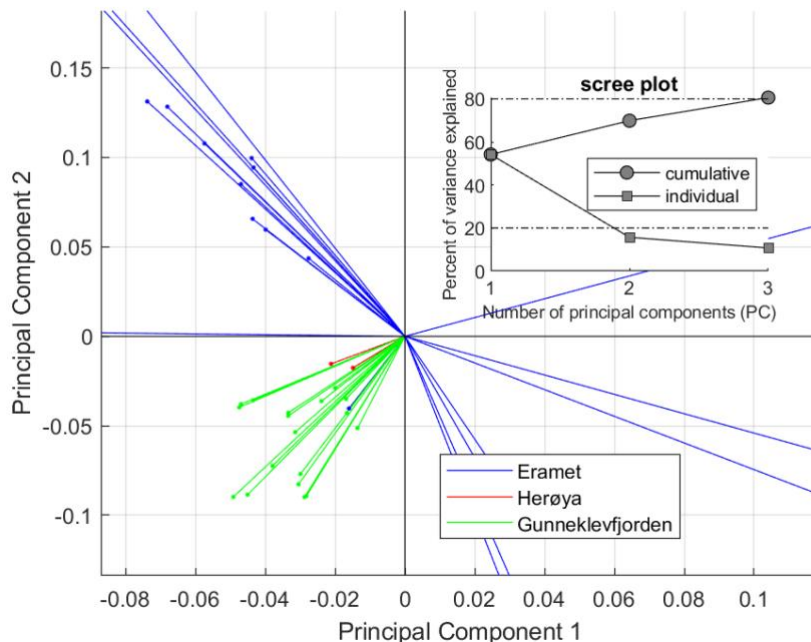
The samples from the Eramet area on Herøya had significantly higher (ANOVA  $p < 0.0001$ ) Mn concentrations ( $183.7 \pm 120.8 \text{ mg g}^{-1}$ ) compared to the samples from the waste site on Herøya ( $2.2\text{--}2.9 \text{ mg g}^{-1}$ ) and from Gunneklevfjorden ( $1.8 \pm 1.7 \text{ mg g}^{-1}$ ) (**Figure 4**). This is not surprising as the Eramet Norway on Herøya is site for production of refined manganese alloys. However, the similar Mn concentrations in samples from the waste site on Herøya and in sediments from Gunneklevfjorden suggest that Herøya and Gunneklevfjorden have a similar geochemical signature (see also **Figure 5**). The only exception to this clear cleavage is sample 14A (Eramet – Sludge chamber #2) which shows very low Mn content ( $0.9 \text{ mg g}^{-1}$ ) compared to the other samples taken at this site ( $77\text{--}522 \text{ mg g}^{-1}$ ). Concentrations of other metals, for example lead (Pb), shows much less variation between sample locations (no significant differences, ANOVA  $p = 0.1330$ ), and a plot of Pb versus Mn concentrations for each sample taken at the various sites show a distinct separation of two groups of samples (**Figure 4**), mainly riven by the difference in Mn.



**Figure 4** A xy-plot showing concentrations of lead (Pb, y-axis,  $\text{mg g}^{-1}$ ) versus concentrations of manganese (Mn, x-axis,  $\text{mg g}^{-1}$ ) for all samples in the Gunneklevfjorden area.

A PCA shows that two principal components were enough to explain almost 70% of the covariance in the dataset (**Figure 5**). Indeed, PC 1 and PC2 explained 54.1% and 15.7% of the variance in metal concentrations, respectively. The behavior of the dataset against those two PC is illustrated in **Figure 5**. This figure displays the individual vectors representing variance in the data against PC1 (x-axis) and PC2 (y-axis). Again, the vectors for Herøya and Gunneklevfjorden are very similar and grouped together

whereas those for Eramet are diverging (except for sample 14A). This further supports the idea that Herøya and Gunneklevfjorden have similar geochemical signatures.



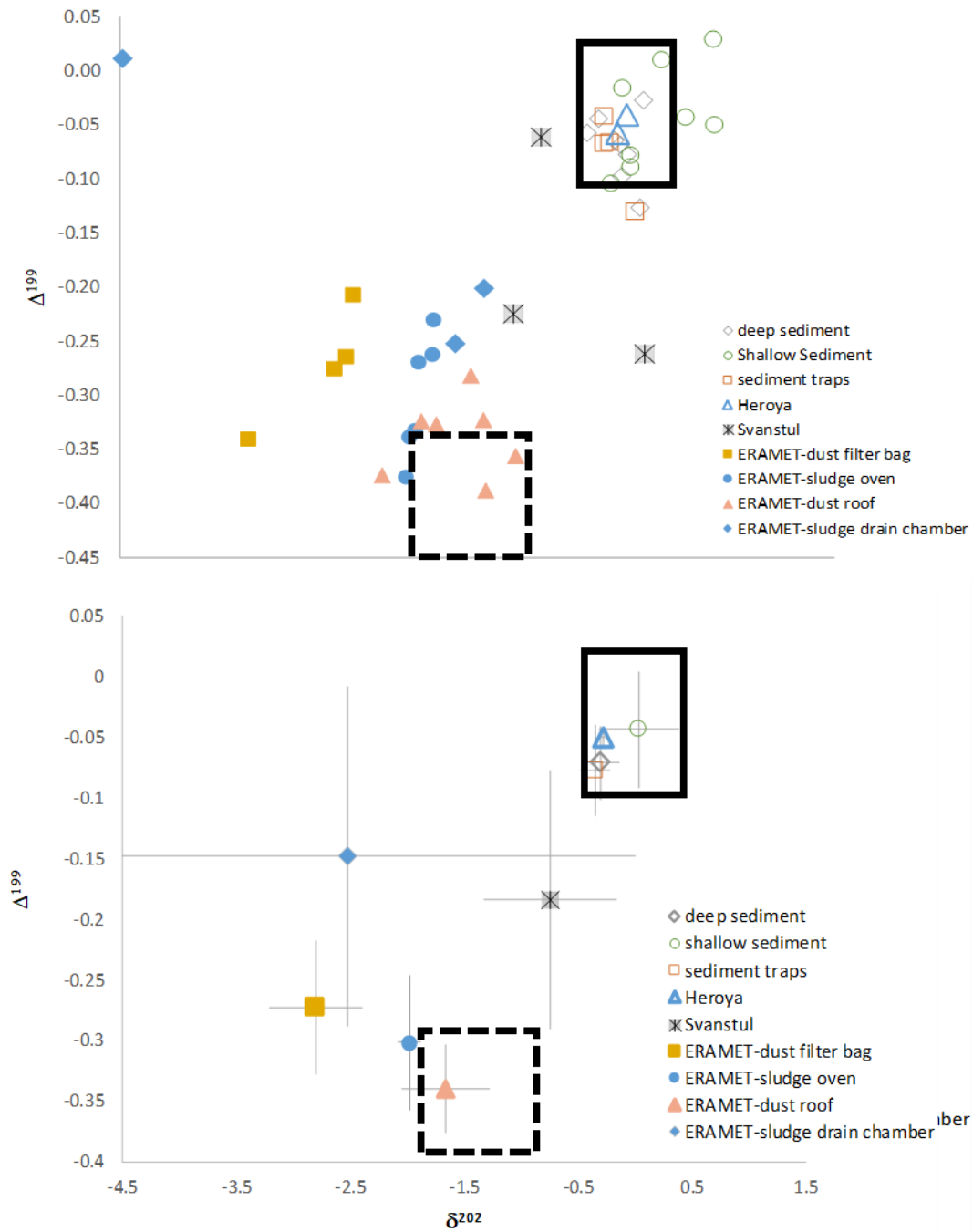
**Figure 5** Loading plot of the PCA showing the vectors for each site as well as the scree plot (inset) showing the % of variance explained by each principal component (PC).

### 3.4 Source apportionment evaluation

Paired  $\delta^{202}\text{Hg}$  and  $\Delta^{199}\text{Hg}$  signatures confirm that sediments fall into at least two distinct isotopic groups in the Gunneklevfjorden area (**Figure 6**). Sites that displayed  $\delta^{202}\text{Hg}$  above  $-0.6\text{‰}$  are largely absent of  $\Delta^{199}\text{Hg}$  signatures. These signatures fall within a range similar to sediment contaminated with Hg from heavy metal refining and metallic Hg usage such as chlor-alkali (Feng et al., 2010; Grigg et al., 2018; Mil-Homens et al., 2013; Perrot et al., 2010; Sonke et al., 2010). Sediments that displayed more depleted signatures of  $\delta^{202}\text{Hg}$  also showed negative  $\Delta^{199}\text{Hg}$ . The only exception to this grouping is site 19A which is the only isotopically depleted  $\delta^{202}\text{Hg}$  sample which has no  $\Delta^{199}\text{Hg}$ . European coals (Sun et al., 2014) are found to fall into a similar range for both isotopes as those found at Eramet.

The distinct differences in  $\delta^{202}\text{Hg}$  and  $\Delta^{199}\text{Hg}$  between samples from Eramet and those from the Gunneklevfjorden sediment and Herøya raises the possibility that the abundance of Hg isotopes could be used to differentiate sources of Hg to Gunneklevfjorden. Small differences in signatures, such as that observed between surficial sediment and deep sediment/sediment traps, could be due to environmental processes which cause fractionation. However, the large differences in both MDF and MIF between Gunneklevfjorden sediment/Herøya and samples from Eramet appear to be large enough that fractionation caused by natural processes is likely to be small by comparison. Some separation among

the samples from Eramet emerged in the  $\delta^{202}\text{Hg}$  and  $\Delta^{199}\text{Hg}$  data, and the overall signature of Eramet samples is not as clear as the Herøya-related signature. The large variation found within sample types at Eramet makes it difficult to tell whether Hg from distinct sources within Eramet could be traced using isotopic composition.



**Figure 6** Isotopic biplot for Gunneklevfjorden-area samples. Shown are all values (top panel) and mean  $\pm$  one standard deviation of measurements for each sample group (bottom panel). Boxes represent the ranges of European coals (dashed lines) and metallic (unbroken lines) Hg contamination.

The Hg isotopes quantified in this study make it clear that the total pool of Hg (isotopic ratios were measured in the THg from samples) is distinct among potential sources in the Gunneklevfjorden area and suggests that Hg in Gunneklevfjorden sediment and suspended particles largely has originated from a source with a signature similar to the chlorine factory waste at Herøya. This support the previous assumption that resuspension of sediment in Gunneklevfjorden is a significant source for particles in the water column and contributes to the Hg export out of the fjord (Olsen et al., 2015). Though this study does not present signatures for all potential Hg sources, there is no indication of other unknown major sources that have not been accounted for. It should be noted that this study does not quantify the contribution from the various sources.

## 4 Conclusion and future work

The present investigation of the potential for distinguishing Hg sources to the Gunneklevfjorden area by a high-resolution analytical isotopic fingerprinting method raises the possibility that the method can be utilized for this purpose. Comparing concentrations of Hg with MDF ( $\delta^{202}\text{Hg}$ ) and MIF ( $\Delta^{199}\text{Hg}$ ) revealed two distinct isotopic fingerprints: one group encompassed samples from the Gunneklevfjorden sediment and Herøya chlorine factory waste and a second group encompassed samples from various Eramet locations. This pattern is evident also in the samples' geochemical signatures (based on concentration distributions of other metals). The distinct differences in Hg isotopes ( $\delta^{202}\text{Hg}$  and  $\Delta^{199}\text{Hg}$ ) between samples from Eramet and those from the Gunneklevfjorden sediment and Herøya supports that the abundance of Hg isotopes could be used to differentiate sources of Hg to Gunneklevfjorden. The results suggest that Hg in bottom and suspended sediment from Gunneklevfjorden largely has originated from a source with a signature similar to the chlorine factory waste at Herøya. The interpretation of this is that suspended particles most likely originates from resuspended surface sediment, as previously assumed. Based on this study, Eramet does not seem to be a significant Hg source to Gunneklevfjorden surface sediment or to suspended particles.

By comparing the paired MDF and MIF isotopic signatures with known values from the literature, we find that the two groups resembles potentially different origins of Hg; the Gunneklevfjorden sediments and Herøya waste sediment have signatures typical for contamination of Hg from heavy metal refining and metallic Hg use, while the signatures of Eramet samples are overlapping with signatures related to contamination of Hg from European coals.

For future work, a next logical step would be to target measurements of the isotopic composition in the food web (where most Hg is MeHg) and also in the MeHg fraction of sediment, in order to help understand to what extent legacy sources are contributing to present Hg bioaccumulation potential. This would also be of value to assess the potential effects of sediment remediation actions to Hg concentrations in biota, and in fish in particular. It should be noted that the fraction of the THg in aquatic systems present as bioaccumulative MeHg is likely to be small (< 0.1 % in most contaminated systems but measured to 0.5 % in sediments in the Gunneklevfjorden meadow, Olsen et al., 2018). Hence, discerning the isotopic signature of the MeHg fraction in particular could help to understand which Hg sources may be responsible for contributing risk to ecological receptors (biota). The high Hg concentrations in Gunneklevfjorden are particularly suitable to the determination of isotopic fractionation of the Hg present as MeHg (Janssen et al., 2015). Since recently deposited Hg is typically more susceptible to methylation, it would also make sense to attempt to obtain a more precise signature of potential local watershed sources for Gunneklevfjorden. This could be done by collecting water and bedded or suspended sediment from major tributaries to the Gunneklevfjorden to help discern the isotopic signature of other recently deposited Hg sources. This additional information about the signature of other non-point Hg sources in the area would be especially valuable in tracing resuspended sediment from Gunneklevfjorden transported into Frierfjorden

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## Appendix A.

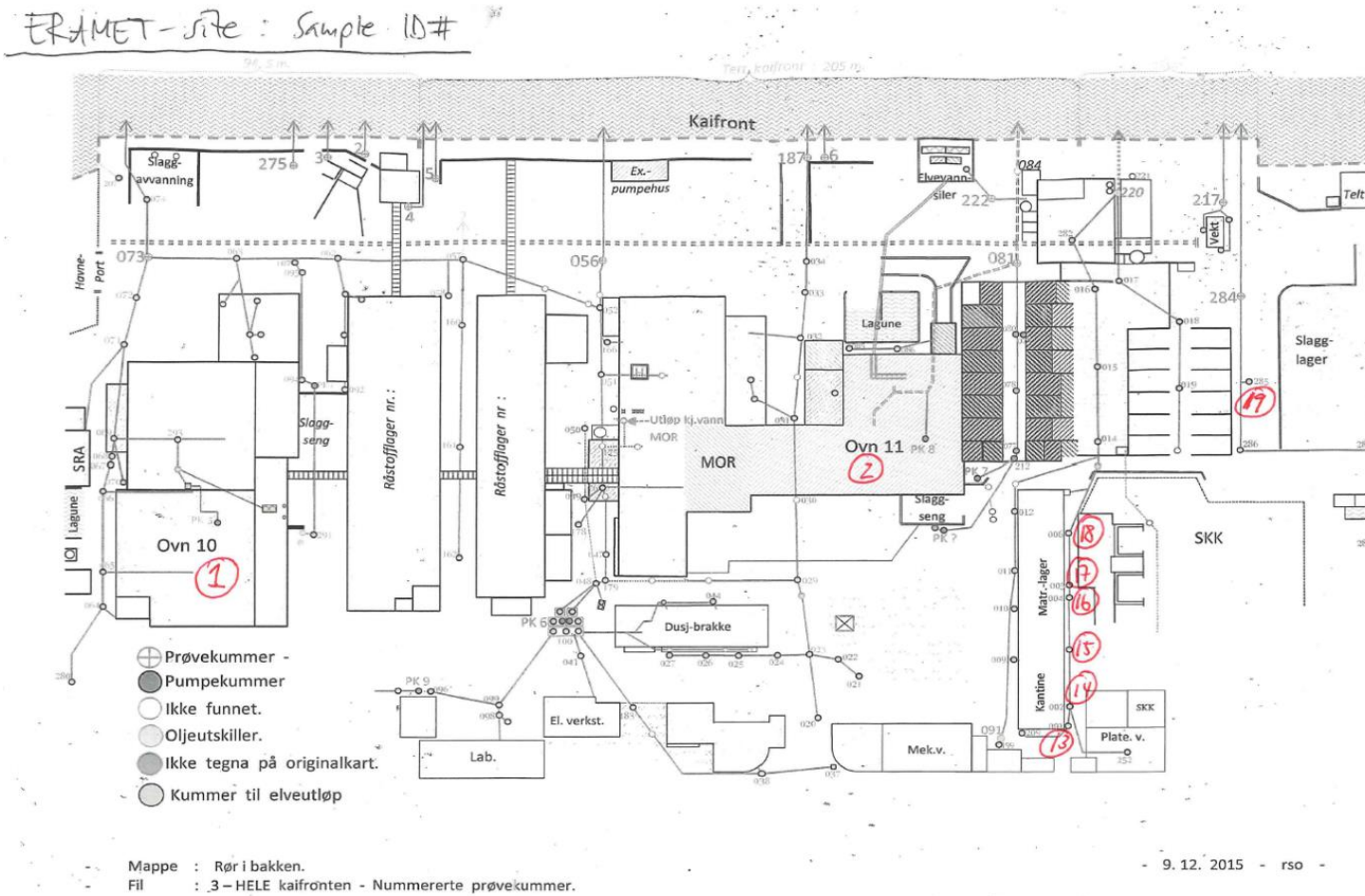
**Table A1** Sample locations and descriptions. The *description* column correspond to labels on Figures 1-3 in the report. The *specific location* column correspond to sampling IDs in Figure A1, A2 and A3.

UMD SampleID	Location	Sample Type	Description	Specific Location	Sampling Device
1A	ERAMET site	sludge - oven	ERAMET site - sludge - oven	Oven 10	Spoon
1B	ERAMET site	sludge - oven	ERAMET site - sludge - oven	Oven 10	Spoon
1C	ERAMET site	sludge - oven	ERAMET site - sludge - oven	Oven 10	Spoon
2A	ERAMET site	sludge - oven	ERAMET site - sludge - oven	Oven 11	Spoon
2B	ERAMET site	sludge - oven	ERAMET site - sludge - oven	Oven 11	Spoon
2C	ERAMET site	sludge - oven	ERAMET site - sludge - oven	Oven 11	Spoon
3A	ERAMET site	dust - filter bag	ERAMET site - dust - filter bag	P-SENT 10	Spoon
3B	ERAMET site	dust - filter bag	ERAMET site - dust - filter bag	P-SENT 10	Spoon
3C	ERAMET site	dust - filter bag	ERAMET site - dust - filter bag	P-SENT 10	Spoon
4A	ERAMET site	dust - filter bag	ERAMET site - dust - filter bag	P-SENT 11	Spoon
4B	ERAMET site	dust - filter bag	ERAMET site - dust - filter bag	P-SENT 11	Spoon
4C	ERAMET site	dust - filter bag	ERAMET site - dust - filter bag	P-SENT 11	Spoon
9A	ERAMET site	dust - roof	ERAMET site - dust - roof	Oven House #3	Spoon
9B	ERAMET site	dust - roof	ERAMET site - dust - roof	Oven House #4	Spoon
9C	ERAMET site	dust - roof	ERAMET site - dust - roof	Oven House #5	Spoon
10A	ERAMET site	dust - roof	ERAMET site - dust - roof	Oven House #1 and #2	Spoon
10B	ERAMET site	dust - roof	ERAMET site - dust - roof	Oven House #1 and #2	Spoon
10C	ERAMET site	dust - roof	ERAMET site - dust - roof	Oven House #1 and #2	Spoon
11A	ERAMET site	dust - roof	ERAMET site - dust - roof	Crushing of finished material	Spoon
11B	ERAMET site	dust - roof	ERAMET site - dust - roof	Crushing of finished material	Spoon
11C	ERAMET site	dust - roof	ERAMET site - dust - roof	Crushing of finished material	Spoon
14A	ERAMET site	sludge - drainage chamber	ERAMET site - sludge - drainage chamber	Chamber #2	n.a.

UMD SampleID	Location	Sample Type	Description	Specific Location	Sampling Device
17A	ERAMET site	sludge - drainage chamber	ERAMET site - sludge - drainage chamber	Chamber #5	n.a.
18A	ERAMET site	sludge - drainage chamber	ERAMET site - sludge - drainage chamber	Chamber #6	n.a.
19A	ERAMET site	sludge - drainage chamber	ERAMET site - sludge - drainage chamber	Chamber #284	n.a.
20A	Herøya Industry Park	Herøya - chlorine factory waste	Herøya - chlorine factory waste	old waste from chlorine factory	n.a.
20B	Herøya Industry Park	Herøya - chlorine factory waste	Herøya - chlorine factory waste	old waste from chlorine factory	n.a.
21A	Svanstulvatn	reference sediment - svanstul	Svanstulvatn - reference sediment - svanstul	Reference sediment	Grab
21B	Svanstulvatn	reference sediment - svanstul	Svanstulvatn - reference sediment - svanstul	Reference sediment	Grab
21C	Svanstulvatn	reference sediment - svanstul	Svanstulvatn - reference sediment - svanstul	Reference sediment	Grab
24A	Gunneklevfjorden	sediment trap	Gunneklevfjorden - sediment trap	North - 80 m from GU23	Sediment traps
24B	Gunneklevfjorden	sediment trap	Gunneklevfjorden - sediment trap	North - 80 m from GU23	Sediment traps
25B	Gunneklevfjorden	sediment trap	Gunneklevfjorden - sediment trap	Mid-fjord - close to GU20	Sediment traps
26A	Gunneklevfjorden	sediment trap	Gunneklevfjorden - sediment trap	South - between GU10 and GU11	Sediment traps
27A	Gunneklevfjorden	shallow sediment	Gunneklevfjorden - shallow sediment	GU12 - 0-5 cm	Sediment corer
27B	Gunneklevfjorden	shallow sediment	Gunneklevfjorden - shallow sediment	GU12 - 0-5 cm	Sediment corer
29A	Gunneklevfjorden	shallow sediment	Gunneklevfjorden - shallow sediment	GU19 - 0-5 cm	Sediment corer
29B	Gunneklevfjorden	shallow sediment	Gunneklevfjorden - shallow sediment	GU19 - 0-5 cm	Sediment corer
31A	Gunneklevfjorden	shallow sediment	Gunneklevfjorden - shallow sediment	GU23 - 0-5 cm	Sediment corer
31B	Gunneklevfjorden	shallow sediment	Gunneklevfjorden - shallow sediment	GU23 - 0-5 cm	Sediment corer
33A	Gunneklevfjorden	shallow sediment	Gunneklevfjorden - shallow sediment	GU02 - 0-5 cm	Sediment corer

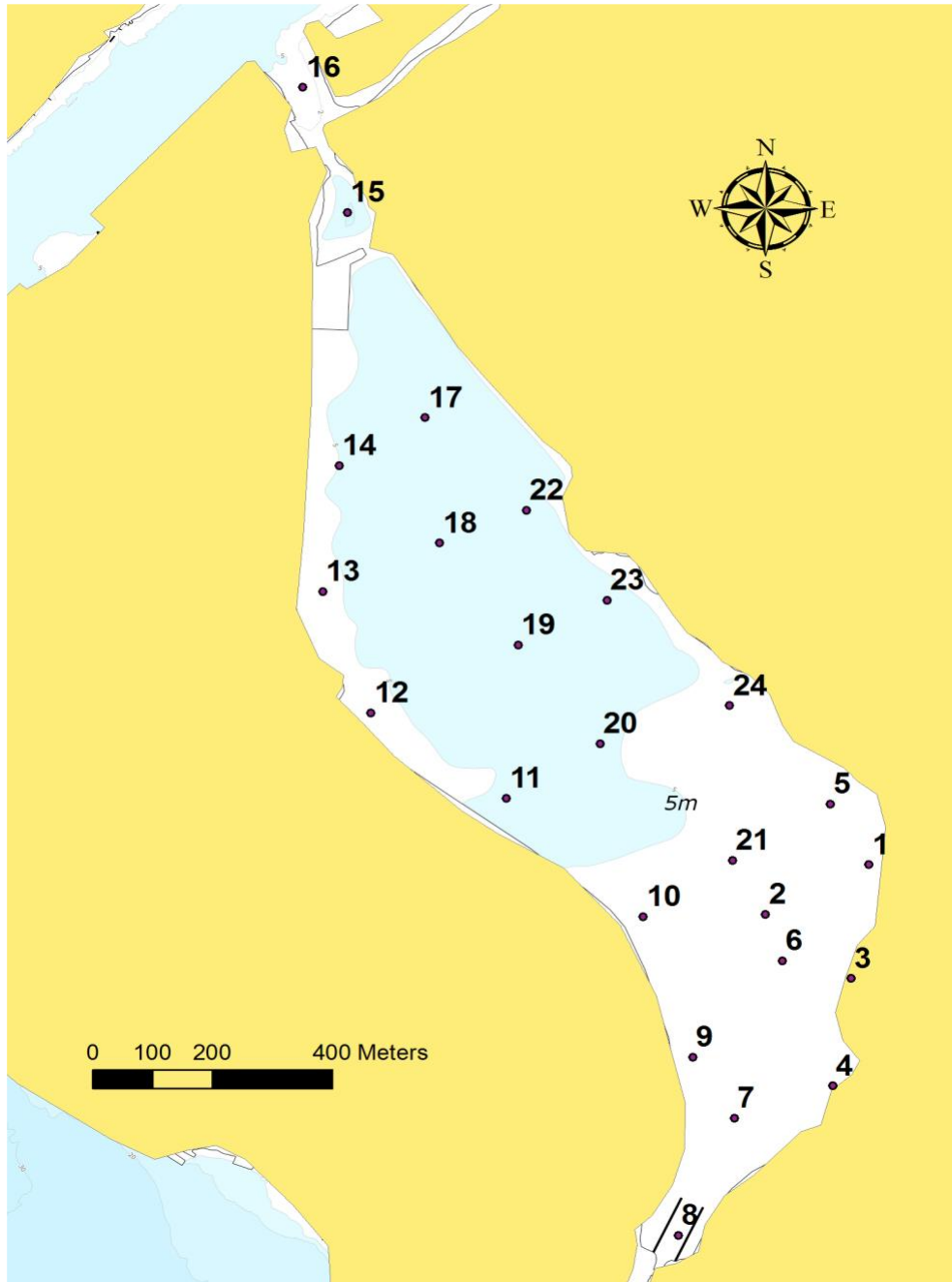
UMD SampleID	Location	Sample Type	Description	Specific Location	Sampling Device
33B	Gunnekelevfjorden	shallow sediment	Gunnekelevfjorden - shallow sediment	GU02 - 0-5 cm	Sediment corer
28A	Gunnekelevfjorden	deep sediment	Gunnekelevfjorden - deep sediment	GU12 - 20-25 cm	Sediment corer
28B	Gunnekelevfjorden	deep sediment	Gunnekelevfjorden - deep sediment	GU12 - 20-25 cm	Sediment corer
30A	Gunnekelevfjorden	deep sediment	Gunnekelevfjorden - deep sediment	GU19 - 20-25 cm	Sediment corer
30B	Gunnekelevfjorden	deep sediment	Gunnekelevfjorden - deep sediment	GU19 - 20-25 cm	Sediment corer
32A	Gunnekelevfjorden	deep sediment	Gunnekelevfjorden - deep sediment	GU23 - 20-25 cm	Sediment corer
32C	Gunnekelevfjorden	deep sediment	Gunnekelevfjorden - deep sediment	GU23 - 20-25 cm	Sediment corer
34A	Gunnekelevfjorden	deep sediment	Gunnekelevfjorden - deep sediment	GU02 - 20-25 cm	Sediment corer
34B	Gunnekelevfjorden	deep sediment	Gunnekelevfjorden - deep sediment	GU02 - 20-25 cm	Sediment corer
35A	North Sea	reference sediment N. sea	North Sea - reference sediment N. sea	E250 - 0-5 cm	Sediment corer?
36A	North Sea	reference sediment N. sea	North Sea - reference sediment N. sea	Ref. south (Z) - 0-5 cm	Sediment corer?

**Figure A1** Sample locations from ERAMET facilities at Herøya – part 1.





**Figure A3** Sample locations in Gunneklevfjorden.



**Figure A4** Location of Svanstulvatnet relative to Gunneklevfjorden.



**Table A2** Raw results for THg and multicollector isotope mass fractionation and mass independent fractionation. All isotope values reported in per mille (‰). Typical analytical standard deviation (derived from bracketing standards) is 0.03 ‰.

SAMPLE ID	LOCATION	SEDIM THG [MG/KG]	1/HGT	199 AVG	D200	D201	D202	D204	D199	D200	D201	D204
1A	ERAMET site	1.45	0.69	-0.699	-0.933	-1.621	-1.856	-2.824	-0.231	0.000	-0.226	-0.053
1B	ERAMET site	1.29	0.78	-0.903	-1.083	-1.897	-2.094	-3.044	-0.376	-0.031	-0.323	0.082
1C	ERAMET site	1.93	0.52	-0.840	-1.040	-1.778	-2.013	-3.045	-0.333	-0.029	-0.264	-0.039
2A	ERAMET site	3.67	0.27	-0.733	-0.947	-1.667	-1.866	-2.668	-0.263	-0.009	-0.264	0.117
2B	ERAMET site	5.80	0.17	-0.858	-1.049	-1.806	-2.062	-3.113	-0.339	-0.013	-0.256	-0.035
2C	ERAMET site	4.05	0.25	-0.770	-1.036	-1.706	-1.984	-3.002	-0.270	-0.039	-0.214	-0.039
3A	ERAMET site	0.73	1.37	need additional processing								
3B	ERAMET site	0.60	1.66	need additional processing								
3C	ERAMET site	0.08	13.03	-1.201	-1.740	-2.899	-3.410	-5.117	-0.342	-0.027	-0.335	-0.026
4A	ERAMET site	5.24	0.19	-0.952	-1.393	-2.268	-2.681	-3.931	-0.277	-0.046	-0.252	0.071
4B	ERAMET site	4.87	0.21	-0.916	-1.316	-2.196	-2.583	-3.886	-0.265	-0.018	-0.253	-0.029
4C	ERAMET site	4.92	0.20	-0.845	-1.260	-2.151	-2.527	-3.854	-0.208	0.009	-0.251	-0.081
9A	ERAMET site	2.70	0.37	-0.818	-0.986	-1.768	-1.956	-2.862	-0.325	-0.003	-0.297	0.059
9B	ERAMET site	3.17	0.32	-0.790	-0.939	-1.658	-1.837	-2.656	-0.327	-0.016	-0.277	0.086
9C	ERAMET site	0.82	1.23	-0.951	-1.170	-2.034	-2.288	-3.420	-0.375	-0.020	-0.313	-0.004
10A	ERAMET site	0.43	2.33	need additional processing								
10B	ERAMET site	0.29	3.40	-0.746	-0.758	-1.402	-1.418	-2.111	-0.389	-0.045	-0.335	0.006
10C	ERAMET site	0.97	1.03	need additional processing								
11A	ERAMET site	1.60	0.62	-0.651	-0.624	-1.194	-1.166	-1.761	-0.357	-0.038	-0.316	-0.019
11B	ERAMET site	0.48	2.07	-0.686	-0.723	-1.376	-1.438	-2.135	-0.324	-0.001	-0.294	0.012
11C	ERAMET site	0.16	6.24	-0.671	-0.729	-1.393	-1.543	-2.207	-0.282	0.046	-0.232	0.097
14A	ERAMET site	0.53	1.87	-0.562	-0.709	-1.264	-1.432	-2.132	-0.202	0.010	-0.188	0.006
17A	ERAMET site	0.13	7.60	-0.674	-0.852	-1.483	-1.670	-2.433	-0.253	-0.014	-0.227	0.060
18A	ERAMET site	0.33	3.05	need additional processing								
19A	ERAMET site	0.14	7.13	-1.115	-2.255	-3.371	-4.468	-6.745	0.011	-0.010	-0.012	-0.074
20A	Herøya Industry Park (HIP)	73.64	0.01	-0.102	-0.097	-0.211	-0.240	-0.387	-0.041	0.023	-0.031	-0.030



20B	Herøya Industry Park (HIP)	62.65	0.02	-0.136	-0.166	-0.254	-0.308	-0.453	-0.058	-0.011	-0.022	0.007
21A	Svanstulvatn	0.26	3.80	-0.283	-0.082	-0.410	-0.084	-0.073	-0.262	-0.040	-0.347	0.053
21B	Svanstulvatn	0.35	2.85	-0.302	-0.428	-0.836	-0.951	-1.596	-0.062	0.049	-0.121	-0.177
21C	Svanstulvatn	0.17	5.77	-0.525	-0.592	-1.107	-1.185	-1.794	-0.226	0.004	-0.216	-0.024
24A	Gunneklevfjo rden	5.53	0.18	-0.172	-0.087	-0.194	-0.162	-0.069	-0.131	-0.005	-0.072	0.173
24B	Gunneklevfjo rden	4.18	0.24	-0.162	-0.249	-0.437	-0.376	-0.612	-0.067	-0.060	-0.154	-0.050
25B	Gunneklevfjo rden	3.03	0.33	-0.173	-0.180	-0.399	-0.419	-0.701	-0.067	0.031	-0.084	-0.076
26A	Gunneklevfjo rden	6.54	0.15	-0.150	-0.253	-0.415	-0.426	-0.644	-0.043	-0.039	-0.095	-0.008
27A	Gunneklevfjo rden	4.80	0.21	0.023	0.058	0.043	0.057	0.197	0.009	0.030	0.000	0.112
27B	Gunneklevfjo rden	6.59	0.15	0.152	0.261	0.402	0.490	0.798	0.029	0.015	0.034	0.066
29A	Gunneklevfjo rden	14.90	0.07	0.076	0.245	0.270	0.502	0.779	-0.051	-0.007	-0.108	0.029
29B	Gunneklevfjo rden	12.55	0.08	-0.130	-0.116	-0.221	-0.205	-0.327	-0.079	-0.013	-0.066	-0.020
31A	Gunneklevfjo rden	15.45	0.06	0.023	0.181	0.131	0.266	0.318	-0.044	0.048	-0.068	-0.079
31B	Gunneklevfjo rden	8.51	0.12	-0.198	-0.220	-0.329	-0.369	-0.568	-0.105	-0.035	-0.052	-0.017
33A	Gunneklevfjo rden	53.15	0.02	-0.140	-0.089	-0.195	-0.198	-0.304	-0.090	0.010	-0.046	-0.008
33B	Gunneklevfjo rden	58.59	0.02	-0.085	-0.137	-0.257	-0.271	-0.357	-0.017	-0.001	-0.053	0.048
28A	Gunneklevfjo rden	306.95	0.00	-0.200	-0.317	-0.460	-0.565	-0.878	-0.058	-0.033	-0.035	-0.034
28B	Gunneklevfjo rden	73.61	0.01	-0.163	-0.289	-0.421	-0.468	-0.674	-0.045	-0.054	-0.069	0.025
30A	Gunneklevfjo rden	48.48	0.02	-0.149	-0.201	-0.302	-0.335	-0.527	-0.064	-0.033	-0.050	-0.027
30B	Gunneklevfjo rden	33.58	0.03	-0.141	-0.149	-0.271	-0.285	-0.456	-0.069	-0.006	-0.058	-0.032
32A	Gunneklevfjo rden	203.18	0.00	-0.053	-0.048	-0.043	-0.097	-0.186	-0.028	0.001	0.030	-0.041

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32C	Gunneklevfjorden	294.57	0.00	-0.157	-0.064	-0.179	-0.120	-0.161	-0.127	-0.003	-0.089	0.018
34A	Gunneklevfjorden	131.02	0.01	-0.136	-0.150	-0.264	-0.232	-0.250	-0.078	-0.033	-0.090	0.095
34B	Gunneklevfjorden	12.22	0.08	-0.168	-0.161	-0.283	-0.279	-0.375	-0.098	-0.020	-0.073	0.042
35A	North Sea	too low for detection with current digest										
36A	North Sea	too low for detection with current digest										

**Table A3a** A summary of a selection of the suit of metals analysed in the samples from Gunneklevfjorden, Herøya and ERAMET. All concentrations in mg g<sup>-1</sup>.

Sample ID	Al	Sb	As	Be	Cd	Ca	Cr	Co	Cu	Fe	Pb	Li	Mg	Mn
1A	5.60	0.0018	0.003	0.0002	0.0332	8.98	0.007	0.005	0.03	8.58	0.26	0.020	12.61	77.93
1C	9.82	0.0022	0.004	0.0005	0.0434	17.23	0.013	0.009	0.04	10.77	0.34	0.025	15.26	94.82
2A	2.30	0.0004	0.008	0.0002	0.0009	12.22	0.129	0.144	0.10	23.22	0.03	0.003	4.75	57.96
2B	6.98	0.0015	0.014	0.0004	0.6388	12.15	0.012	0.015	0.12	5.21	2.21	0.026	8.31	211.23
3A	15.49	0.0029	0.008	0.0007	0.0278	29.86	0.040	0.035	0.08	8.44	0.44	0.040	23.29	174.92
3C	13.45	0.0015	0.007	0.0006	0.0276	24.64	0.033	0.030	0.08	6.84	0.44	0.035	21.22	154.29
4A	8.39	0.0026	0.020	0.0006	0.2834	14.78	0.028	0.101	0.19	11.92	2.41	0.022	8.06	243.45
4B	6.74	0.0048	0.016	0.0005	0.2148	11.91	0.024	0.082	0.16	10.18	1.75	0.018	6.65	202.05
9A	7.13	0.0027	0.023	0.0005	0.0049	23.14	0.300	0.133	0.15	95.64	0.09	0.008	6.92	110.16
9B	5.78	0.0028	0.018	0.0004	0.0030	20.90	0.121	0.108	0.12	80.44	0.06	0.007	5.13	92.97
9C	8.59	0.0017	0.012	0.0005	0.0035	18.41	0.224	0.104	0.13	128.72	0.04	0.009	6.31	79.43
10A	4.18	0.0021	0.014	0.0002	0.0030	20.41	0.248	0.250	0.19	45.53	0.07	0.005	12.29	198.68
10B	3.66	0.0012	0.010	0.0002	0.0015	14.79	0.151	0.176	0.12	27.89	0.06	0.004	8.72	133.02
10C	4.67	0.0016	0.016	0.0002	0.0038	17.80	0.197	0.210	0.18	49.13	0.09	0.006	9.71	161.50
11A	5.66	0.0021	0.012	0.0005	0.0054	19.28	0.380	0.223	0.23	72.40	0.04	0.007	9.37	210.41
11B	7.20	0.0037	0.019	0.0006	0.0063	26.35	0.453	0.311	0.32	543.43	0.06	0.010	10.17	307.12
11C	2.52	0.0060	0.033	0.0002	0.0025	10.08	0.353	0.577	0.33	96.24	0.02	0.003	7.88	426.21
14A	2.34	0.0005	0.001	0.0004	0.0002	19.45	0.008	0.002	0.01	3.14	0.01	0.002	105.16	0.94
17A	13.22	0.0014	0.012	0.0012	0.0013	71.84	0.274	0.163	0.13	50.65	0.02	0.017	26.87	215.88
18A	3.34	0.0014	0.014	0.0002	0.0017	18.74	0.229	0.265	0.17	35.01	0.01	0.004	6.48	182.53

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<b>19A</b>	1.69	0.0030	0.028	0.0002	0.0002	7.40	0.479	0.648	0.36	85.73	0.00	0.002	3.15	522.19
<b>20A</b>	2.36	0.0005	0.006	0.0002	0.0001	96.41	0.026	0.006	0.02	8.12	0.02	0.005	3.07	2.94
<b>20B</b>	2.65	0.0005	0.004	0.0002	0.0001	83.80	0.018	0.004	0.01	6.58	0.01	0.007	2.65	2.17
<b>24A</b>	8.89	0.0007	0.005	0.0012	0.0005	10.75	0.023	0.008	0.04	15.17	0.04	0.011	7.34	4.36
<b>24B</b>	4.72	0.0005	0.003	0.0007	0.0003	5.74	0.026	0.004	0.02	7.32	0.02	0.005	4.48	0.36
<b>27A</b>	1.98	0.0004	0.002	0.0004	0.0002	27.10	0.008	0.002	0.01	2.73	0.01	0.002	209.68	1.03
<b>27B</b>	9.69	0.0005	0.008	0.0010	0.0005	2.83	0.026	0.008	0.06	21.06	0.06	0.019	9.04	0.33
<b>28A</b>	6.72	0.0005	0.004	0.0033	0.0006	33.48	0.038	0.003	0.03	7.53	0.03	0.003	139.90	1.64
<b>28B</b>	2.97	0.0005	0.002	0.0011	0.0002	27.43	0.019	0.003	0.03	4.29	0.02	0.002	96.55	1.42
<b>29A</b>	3.10	0.0004	0.002	0.0006	0.0003	25.77	0.010	0.003	0.01	5.06	0.01	0.004	74.59	1.88
<b>29B</b>	9.12	0.0004	0.005	0.0015	0.0007	19.42	0.027	0.008	0.04	15.15	0.04	0.012	82.87	5.27
<b>30A</b>	12.97	0.0005	0.011	0.0023	0.0029	27.10	0.091	0.010	0.17	25.95	0.13	0.021	42.01	1.22
<b>30B</b>	12.90	0.0011	0.010	0.0024	0.0025	10.45	0.083	0.009	0.15	24.68	0.13	0.022	60.43	1.19
<b>31A</b>	13.30	0.0005	0.010	0.0023	0.0010	32.52	0.038	0.012	0.06	24.77	0.06	0.017	25.77	4.76
<b>31B</b>	11.29	0.0005	0.008	0.0021	0.0010	40.51	0.039	0.009	0.06	20.07	0.06	0.014	63.39	4.88
<b>32A</b>	4.11	0.0005	0.008	0.0022	0.0015	72.22	0.018	0.002	0.05	8.41	0.05	0.002	106.80	0.79
<b>32C</b>	3.40	0.0005	0.005	0.0020	0.0007	86.21	0.008	0.001	0.04	4.22	0.03	0.002	128.41	0.66
<b>33A</b>	12.32	0.0005	0.009	0.0022	0.0017	4.43	0.056	0.009	0.11	20.05	0.14	0.016	10.78	0.67
<b>33B</b>	12.77	0.0005	0.008	0.0024	0.0023	7.35	0.066	0.009	0.12	20.52	0.10	0.019	12.90	0.80
<b>34A</b>	7.80	0.0005	0.006	0.0014	0.0010	55.86	0.040	0.007	0.22	16.02	0.13	0.013	34.41	1.19
<b>34B</b>	8.76	0.0005	0.007	0.0009	0.0004	2.94	0.022	0.008	0.05	18.57	0.05	0.017	8.29	0.29

**Table A3b** A summary of a selection of the suit of metals analysed in the samples from Gunneklevfjorden, Herøya and ERAMET. All concentrations in mg g<sup>-1</sup>.

Sample ID	Ni	K	Se	Ag	Na	Sr	Tl	Sn	Ti	V	Zn
1A	0.005	28.04	0.0019	0.0031	6.48	0.19	0.0036	0.05	0.03	0.001	1.94
1C	0.007	29.43	0.0020	0.0037	6.80	0.35	0.0114	0.04	0.07	0.003	2.40
2A	0.115	0.75	0.0004	0.0004	0.24	0.22	0.0006	0.01	0.23	0.018	0.55
2B	0.010	16.40	0.0068	0.0213	9.01	0.30	0.4911	0.03	0.04	0.005	41.73
3A	0.032	70.85	0.0103	0.0053	22.24	0.58	0.0184	0.09	0.15	0.008	3.46
3C	0.027	65.68	0.0095	0.0049	14.07	0.49	0.0115	0.02	0.12	0.006	3.39
4A	0.057	118.20	0.0060	0.0121	22.67	0.33	0.1105	0.01	0.11	0.013	18.57
4B	0.048	83.37	0.0046	0.0078	17.41	0.26	0.1006	0.02	0.09	0.011	15.36
9A	0.199	1.90	0.0021	0.0005	0.58	0.59	0.0005	0.02	1.31	0.020	5.68
9B	0.101	1.40	0.0011	0.0005	0.59	0.54	0.0006	0.01	1.13	0.020	2.54
9C	0.156	2.84	0.0013	0.0005	0.75	0.43	0.0008	0.02	0.51	0.015	6.03
10A	0.236	1.35	0.0005	0.0005	0.42	0.36	0.0002	0.03	0.50	0.036	2.45
10B	0.162	1.13	0.0004	0.0004	0.28	0.37	0.0005	0.04	0.30	0.025	0.82
10C	0.201	1.54	0.0005	0.0005	0.31	0.35	0.0014	0.03	0.32	0.031	3.54
11A	0.229	1.61	0.0006	0.0006	0.46	0.40	0.0019	0.02	1.13	0.028	1.21
11B	0.296	2.30	0.0005	0.0005	0.70	0.54	0.0028	0.07	1.24	0.036	1.35
11C	0.398	1.91	0.0006	0.0006	0.23	0.26	0.0009	0.05	0.81	0.071	0.64
14A	0.008	0.37	0.0005	0.0005	3.33	0.07	0.0002	0.01	0.07	0.004	0.09
17A	0.265	3.05	0.0010	0.0004	1.20	0.97	0.0002	0.42	0.82	0.028	0.17
18A	0.196	0.95	0.0005	0.0005	0.37	0.32	0.0006	0.02	0.70	0.025	0.19
19A	0.397	0.31	0.0005	0.0005	0.21	0.08	0.0001	0.04	1.09	0.061	0.04
20A	0.029	0.48	0.0005	0.0005	0.19	0.14	0.0001	0.07	0.13	0.007	0.06

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<b>20B</b>	0.018	0.62	0.0005	0.0005	0.20	0.11	0.0001	0.02	0.12	0.006	0.04
<b>24A</b>	0.018	2.32	0.0007	0.0007	19.16	0.15	0.0001	0.14	0.27	0.020	0.30
<b>24B</b>	0.015	1.39	0.0005	0.0005	15.17	0.09	0.0001	0.04	0.14	0.009	0.19
<b>27A</b>	0.007	0.26	0.0004	0.0004	2.45	0.08	0.0001	0.04	0.06	0.004	0.08
<b>27B</b>	0.018	3.18	0.0005	0.0005	4.50	0.03	0.0003	0.06	0.60	0.033	0.19
<b>28A</b>	0.041	0.34	0.0005	0.0011	4.10	0.07	0.0001	0.01	0.16	0.037	0.20
<b>28B</b>	0.021	0.31	0.0005	0.0030	2.79	0.08	0.0001	0.03	0.10	0.009	0.15
<b>29A</b>	0.010	0.57	0.0004	0.0004	5.00	0.07	0.0001	0.01	0.11	0.006	0.11
<b>29B</b>	0.022	1.86	0.0004	0.0004	10.23	0.11	0.0002	0.01	0.31	0.017	0.28
<b>30A</b>	0.074	2.28	0.0013	0.0037	10.96	0.13	0.0007	0.03	0.56	0.100	0.54
<b>30B</b>	0.060	2.29	0.0012	0.0037	10.68	0.10	0.0007	0.08	0.58	0.081	0.50
<b>31A</b>	0.035	2.37	0.0011	0.0005	13.08	0.20	0.0001	0.06	0.45	0.028	0.43
<b>31B</b>	0.030	1.95	0.0011	0.0010	11.14	0.19	0.0002	0.03	0.44	0.025	0.41
<b>32A</b>	0.023	0.57	0.0005	0.0010	9.80	0.14	0.0002	0.06	0.16	0.020	0.28
<b>32C</b>	0.021	0.51	0.0005	0.0005	10.00	0.13	0.0001	0.03	0.09	0.009	0.14
<b>33A</b>	0.031	2.45	0.0012	0.0021	10.85	0.11	0.0003	0.07	0.39	0.036	0.48
<b>33B</b>	0.043	2.51	0.0012	0.0024	9.81	0.10	0.0004	0.02	0.46	0.046	0.51
<b>34A</b>	0.031	1.83	0.0005	0.0012	7.87	0.15	0.0003	0.09	0.43	0.027	0.33
<b>34B</b>	0.017	2.91	0.0005	0.0005	4.02	0.03	0.0003	0.01	0.57	0.030	0.16

## NIVA: Norges ledende kompetansesenter på vannmiljø

NIVA gir offentlig vannforvaltning, næringsliv og allmennheten grunnlag for god vannforvaltning gjennom oppdragsbasert forsknings-, utrednings- og utviklingsarbeid. NIVA kjennetegnes ved stor faglig bredde og godt kontaktnett til fagmiljøer i inn- og utland. Faglig tyngde, tverrfaglig arbeidsform og en helhetlig tilnæringsmåte er vårt grunnlag for å være en god rådgiver for forvaltning og samfunnsniv.



Norsk institutt for vannforskning

Gaustadalléen 21 • 0349 Oslo  
Telefon: 02348 • Faks: 22 18 52 00  
[www.niva.no](http://www.niva.no) • [post@niva.no](mailto:post@niva.no)