NGU REPORT 2024.001

Organic soil geochemistry in Hedmark and
Møre and Romsdal counties, QC-report

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Organic soil geochemistry in Hedmark and Møre and Romsdal counties, QC-report

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Summary: This survey constitutes a continuation of corresponding surveys undertaken in Trøndelag county as part of the National Geochemistry Mapping Project. Organic soil samples (humus) were collected in Hedmark (n= 787) and More and Romsdal (n=431) counties at a grid of 6 x 6 km. The <2 mm size fraction of these samples was analysed by ICP-MS for 53 elements following Aqua Regia digestion. In this report, quality of analytical results for the organic soil are documented in tables, and descriptive statistics.

Table of contents

APPENDICES

APPENDIX 1. Random Plots

- APPENDIX 2. X-plots
- APPENDIX 3. Re-analyses
- APPENDIX 4. Field duplicates
- APPENDIX 5. Analytical duplicates
- APPENDIX 6. ECDF Hedmark
- APPENDIX 7. ECDF Møre and Romsdal

1. INTRODUCTION

As part of the National Geochemical Mapping Program of soil, mineral soil and O-horizon (i.e. organic soil) material, were collected in Hedmark (*n*= 787) and Møre and Romsdal (*n*= 431) counties during the field seasons of 2020-2021 and 2021-2022 at a density of 1 sample/36 km². An overview of the mapping progress is given in Table 1.

Survey	Year	Sampling density	Soil	Analysed Fraction	Lab
Finnmark and Troms ¹⁾	2011	$1/40 \text{ km}^2$	Till	< 0.063 mm	$ACME^*$
Nordland ¹⁾	2011	$1/40 \text{ km}^2$	Till	$<$ 2mm	$ACME^*$
North-Trøndelag and Fosen ^{2, 3)}	2013	$1/36$ km ²	Till Humus	$<$ 2mm	$ACME^*$
Southern Trøndelag ^{4, 5)}	2018/2019	$1/36$ km ²	Till Humus	$<$ 2mm	$ACME^*$
Hedmark ⁶	2020/2021	$1/36$ km ²	Till Humus	<2mm	Bureau Veritas (formerly ACME)
Møre and Romsdal ⁷	2021/2022	$1/36$ km ²	Till Humus	$\leq 2 \text{mm}$	Bureau Veritas

Table 1. *Progress in the national geochemical mapping program of Norway.*

¹⁾Reimann et al., 2011

2)Finne et al., 2014

3)Finne and Eggen, 2015

4)Flem et al., 2020

5)Flem et al., 2021

6)Flem et al. 2022

7)Acosta-Góngora et al. (2024)

*) At present, operating as Bureau Veritas Commodities Canada Ltd.

This report presents the quality control of organic soil sample analyses from Hedmark and Møre and Romsdal counties, and it represents a continuation of a national scale survey which has already covered the Trøndelag county (Finne and Eggen, 2015; Flem et al. 2021). The analytical data is available at https://www.ngu.no/ and https://geo.ngu.no/kart/geokjemi_mobil. The surveyed areas (sampling grid of 6km x 6 km) are shown in Figures 1 and 2.

Figure 1. Geology and sample distribution of the Hedmark county (sampling grid of 6 km x6 km). Bedrock map modified from Geological Survey of Norway (2021).

Figure 2. .Geology and sample distribution of the Møre and Romsdal county (sampling grid of 6 km x6 km). Bedrock map modified from Geological Survey of Norway (2021).

2.DESCRIPTION OF THE SURVEY AREA

2.1 Bedrock

2.1.1 Hedmark

Hedmark can be divided into four main bedrock types. The southernmost area is part of the basement, dominated by gneisses and granites (Fig. 1). The topography of this area is relatively uniform with low altitudes < 500 m. Towards the north, bedrock changes to deposits of CambroSilurian age, limestone and clay slate in alternating layers with numerous black shale horizons (Fig. 1). Further north, the bedrock consists of stalagmite from the late Precambrian, which extends far north and covers the majority of Hedmark. In the northern most part of Hedmark, the Caledonian mountain range has elevations that vary from 600 to > 2000 m. This area is dominated by the Trondheim fields, heavily converted Cambro-Silurian slate, penetrated by magmatic rocks in connection with the Caledonian mountain range folding (Fig. 1; e.g**.,** Dahl et al., 2017; Ramberg et al., 2008).

2.1.2 Møre og Romsdal

The Møre and Romsdal County is mainly characterized by granitic gneisses (Fig. 2), which are part of the Western Gneiss Region (WGR). The WGR consists primarily of granitic gneisses, which were formed between 1700 and 1600 Ma and derived from igneous sources. These were strongly deformed and metamorphosed during the Caledonian Orogeny (ca. 420–390 Ma).

During the Caledonian continent-continent collision with Laurentia (Greenland), the WGR was subducted to mantle depths, locally at more than 100 km, marked by the widespread occurrence of high-pressure eclogite lenses, particularly along the western parts that were subjected to the highest pressures (Cuthbert et al., 2000; Hacker and Gans, 2005). The subduction also resulted in fragments of this mantle getting stuck to the downgoing continental slab –found as lenses of olivine-rich mantle peridotite.

During the Caledonian Orogeny, the WGR was overthrust by nappes, which comprise of schists, amphibolites and marbles and continued deformation led to in-folding of the nappes into the gneissic substrate (Terry et al., 2000; Tucker et al., 2004).

The various rocks therefore represent diverse mineral resources, including marble (allochthonous nappes) and olivine (mantle peridotite fragments). Historically, mining for Fe and Ti was undertaken from mafic rocks associated with the gneisses.

3. METHODS

3.1 Field work

A nationwide 6 x 6 km grid covering the Hedmark and Møre and Romsdal counties was used for the selection of sample sites (Figs. 1 and 2). The exact location of each sample site within a grid cell was determined in the field based on accessibility, where each sample was collected

as close as practically possible to the centre of the cell (i.e., sampling site). Though most sites could be reached by car, some required long walking trips. A Quaternary geology base map was always on hand to avoid sampling on top of glaciofluvial and marine deposits. All samples were taken on undeveloped land. The size of the surveyed areas was approximately 27.300 km^2 (Hedmark) and 14, 300 km² (Møre and Romsdal). At each sample site, soil samples representing the O-horizon (i.e., organic horizon) were taken in the same way as described by Finne et al. (2014) and Finne and Eggen (2015).

The soil O-horizon was sampled using composite samples comprising a minimum of 5 subsamples taken with a steel spade within an area of approximately 100 m^2 . The spade was used to cut out a square piece of organic soil of about 15 x 15x 10 cm (example shown Fig. 3). Living plant material was removed from the top of the sample and any non-organic material was removed from the bottom so that only the uppermost 3 cm of the humus and litter layer was retained. In the case that the O-horizon was less than 3 cm thick, the number of subsamples were increased to obtain sufficient material. Sampling and all sample handling of the organic soil were carried out using Nitrile gloves. The samples were stored in white Hubco soil sample bags, $7'' \times 12{\text -}1/2''$, made of a poly/cotton blend cloth obtained from Forestry Suppliers, Inc., US. These contamination-free bags allow organic samples to partially air-dry during transport without starting to develop mold. At each sample site, the vegetation and the general landscape were documented in several photos.

3.2 Sample preparation/pre-analysis

All soil samples were air dried at 30 °C upon arrival at the laboratory of the Geological Survey of Norway (NGU) in Trondheim, within days after sampling. They were subsequently sieved using $a < 2$ mm nylon mesh (lumps were disaggregated by hand) and the passing fraction was retained for analysis. All samples were randomised before submission to the laboratory, and a project standard, field duplicates and sample duplicates were inserted in such a way that they were not recognisable by the laboratory following protocols described in Eggen et al. (2019).

Figure 3. Example of organic soil sample taken with a spade in a mountain landscape above the tree-line.

3.3 Laboratory analysis

The randomized sample series of the <2-mm fraction was shipped to Bureau Veritas Minerals (former Acme Labs) in Vancouver Canada for chemical analysis. The standard package 'VG105 + EXT Dry Plant Material Analysis' involving splits of 5 g was selected. The analytical packages involve a modified Aqua Regia digestion which consists of 1:1:1 v/v concentrated ACS grade HCl, HNO3 and de-mineralized H2O. The analyses were performed by using a Perkin Elmer Elan 6000/9000 inductively coupled plasma mass spectrometer (ICPMS) for 53 elements. Details on the analytical procedure can be found on Bureau Veritas home page, https://commodities.bureauveritas.com/metals-minerals/exploration-andmining/geoanalytical-services.

3.4 Quality control

It was agreed with the laboratory that all instrumental readings had to be reported, independent of detection limit (DL) or quantification limit (QL) set by the laboratory. Reporting limits used by the laboratory are usually set higher than the real quantification limit, as laboratory limits must cover long time operation conditions – possibly years. In addition, the data should not be rounded off, and at least one significant figure containing uncertainty had to be retained.

A project in-house reference material was prepared from natural humus material and analysed after every 20th sample in the randomized sample sequence.

For possible analytical comparison with the previous surveys undertaken in northern Trøndelag/Fosen (Finne and Eggen, 2014) and southern Trøndelag (Flem et al. 2020) a collection of samples was evenly distributed into the new sample series and re-analysed. The same laboratory, Bureau Veritas Minerals, Vancouver, Canada, have been used for all surveys.

The sampling and analytical design follows the unbalanced ANOVA design used in earlier surveys (e.g., Eggen and Finne, 2014; Eggen et al., 2017). The same information can be obtained from a balanced or an unbalanced sampling and analysis design, but an unbalanced design makes a more efficient use of resources (Reimann et al., 2008).

4. RESULTS

4.1 Analytical Quality Control, QC

4.1.1 In-house project standard

In Appendix 1, the analytical results for all samples (ordinary samples in addition to field duplicates and analytical duplicates, and the in-house project standard) are shown following the laboratory's analysis sequence order. The X-axis shows the random number given to the sample before shipping to the laboratory. Negative concentration values reported by the laboratory are replaced by a low positive value, (0.0000001 mg/kg). The laboratory detection limit is indicated by a red dashed line. The appropriateness of the in-house standard for this survey is good for most elements except Ta and S (large variability) and Th, W, Te, In, Re, Pd, and Pt (values at or below detection limit). Tantalum (Ta) might be out of analytical control (Appendix 1) as the in-house project standard shows a marked "undulating" trend in the random plot (Fig. 4) and most analyses have variations over $\pm 20\%$ of the median in X-plots (Appendix 2).

Figure 4. Random plot of Ta showing an analytical trend in the data. Dashed red line represents the detection limit

Fifty-nine splits of the in-house project standard were analysed along with the samples, one split after approximately every 20th sample. X-charts for all elements of the in-house standard are given in Appendix 2. The analytical results are plotted in the same order as they were analysed. For all elements, the median is indicated by a solid black line, whereas dashed lines are drawn at \pm 10% (blue) and \pm 20% (red) relative to the median. A statistical summary for the project in-house standard is given in Table A1. This includes the minimum, median, and maximum concentration values (among other parameters) for all elements. The elements: W, Te, In, Re, Be, Pd and Pt have most concentrations at or below the laboratory detection limit (DL). It is thus not possible to calculate the analytical repeatability for these elements. Most of the other elements show acceptable analytical repeatability with a robust coefficient of variation (CVR<15%) for the in-house project standard (Table A1). However, the elements Nb, Au, Tl, and Ta show a precision (CVR) in growing order from 20 to 40%, mainly due to proximity to DL (e.g., Tl) and/or sample inhomogeneity (e.g. Au, Ta), see Appendix 1.

4.1.2 Samples re-analysed from previous surveys

A selection of 22 organic soil samples from the North Trøndelag and Fosen survey (Finne and Eggen, 2014) was re-analysed; 1134, 1265, 1372, 1474, 1502, 1505, 1509, 1511, 1517, 1519, 1523, 1531, 1540, 1559, 1580, 1593, 1657,1662, 1686, 1728, 1762, 1762_D along with the Hedmark and Møre and Romsdal samples. The results from the laboratory analyses for all 54 elements are shown in Appendix 3. Most elements show a very satisfactory reproducibility. However, most of these show systematically lower values in the new analytical report relative to the previous one. In general, Au, B, Be, Bi, Hf, In, Pd, Pt, Re, S, Se, Sc, Ta, Tl, Th, V, and W show low precision probably due to nugget effects, grain size distribution, low concentrations, and laboratory batch effects. Other elements, such as Ag, Al, As, Ba, Cd, Ce, Ca, Co, Cr, Fe, Cs, Cu, Ge, K, Hg, La, Mg, Na, Mn, Mo, Ni, P, Pb, Rb, Sb, Sn, Sr, Ti, Zn, Zr and Y, show a generally high repeatability. However, one must nevertheless be careful when combining the data sets from the various surveys also for these elements.

4.2 Precision and Analysis of variance (ANOVA)

At approximately every $20th$ sample location, a field duplicate was collected. After drying and sieving, a split of it was prepared and analysed with the rest of the samples (an analytical duplicate of the field duplicate). Table A2 gives a precision estimate for all elements, with concentrations above DL, of the field and analytical duplicate pairs. In addition, the number of pairs above DL of the total of 60 pairs analysed are given. The estimated precision given by the coefficient of variation (CV) for the field duplicates (ordinary sample compared with the field duplicate) ranges from 3% (P) to % 31 (Au). The analytical duplicate pairs (the duplicate field sample and the analytical duplicate) show a much smaller range and better precision compared to the ordinary sample and field duplicate pairs (Tables A1 and A2).

The correlation between ordinary sample and duplicate sample, and the correlation between duplicate sample and analytical duplicate, are shown in Appendices 4 and 5. Poor correlation is usually due to low concentrations (e.g., Be; Appendix 4) or due to natural variation (e.g., Au; Appendix 5).

The field and analytical duplicates can be used to carry out an unbalanced analysis of variance (ANOVA) for those elements with \geq 4 duplicate pairs above DL. By unbalanced it is meant that unequal numbers of analysis occur at each level of design. In Table A3 the analysis of variance (ANOVA) is given for the 60 sites with a duplicate sample giving the distributed percentage

variabilities for all elements. The *p*-value given in Table A3 is for the F-test to determine if the variance at the "between" level are equal for the field and analytical duplicates. The field duplicate ANOVA indicates that the combined sampling and analytical variability is smaller than the between sites regional variability. Molybdenum, Mo, for instance, has an estimated regional variability of 92.1%, and 7.36% variability at site (local variability) and 0.55% analytical variability. However, elements such as Hf, K, Ge, Nb, Ca, Cu, U, Sc, Cr, Zr, Au, V, Ga, Fe, Al, Y, Ce, La and Be show higher local variability $(\geq 20\%)$. For some elements this mainly reflects the difficulties in determining these elements at low concentrations.

4.3 Data distribution

The geochemical distribution of each element for the Hedmark and Møre and Romsdal counties is presented in plots of the cumulative distribution function, ECDF (Appendix 6 and 7). The concentration is plotted along the X-axis and the cumulative probability is plotted along the Yaxis. These plots allow the direct visual recognition of breaks in the curve which may indicate different geochemical processes. Breaks in the uppermost 5-10 percentiles of the distribution are often used as thresholds for anomaly identification. All laboratory readings are shown including those below detection limit. Negative results are replaced by a low positive value. A statistical summary for data from both areas is provided in Tables A4 and A5.

5. SUMMARY

This report gives an evaluation of the quality of the data from the Hedmark and More and Romsdal geochemical organic soil survey. Few data are available for W, Te, In, Re, Pd, Be, and Pt due to too high detection limits. A sequence distribution trend detected for Ta indicate the presence of analytical artifacts for that element. Re-analysed samples from the Trøndelag surveys (Finne et al., 2014; Flem et al., 2020), show that 36 out of 52 elements have a general good repeatability between batches. However, one must nevertheless be careful when combining the data sets from the various surveys also for these elements. With careful use and working preferentially with quantiles, many elements in the national mapping program datasets, that do not have detection limit issues, can be merged and used as one dataset. However, it is the data user's responsibility to consider if the batch effects are within acceptable limits. Finally, the only objective of this report is to assess QA/QC parameters, so it is up to the user to carry out any exploratory data analysis (EDA) of the geochemical datasets.

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TABLES

Table A1. The in-house project organic soil standard

Laboratory detection limit and summary statistics for the organic soil standard. The minimum (MIN), median, mean maximum (MAX) are given. In addition, the interquartile range (IQR) and the robust coefficient of variation (CVR) are given as a measure of precision. The number of pairs (No. Pairs) used to calculate CVR is given, but this parameter is only reported for those elements having ≥ 4 pairs above DL.

Table A2. Precision of duplicates

The precision for field and analytical duplicates is presented in the table only for elements that have ≥4 pairs above DL. Abbreviations: CVR, robust covariation coefficient; No. Pair., Number of pairs above DL.

Table A3. ANOVA

Analysis of variance table (ANOVA) for the 60 duplicate sites giving the distributed percentage variabilities for all elements with ≥4 pairs above MDL. The p-value is for the F-test. All variables were log-transformed prior to the calculation.

Table A4. Hedmark data

Summary statistics for the regional survey data (n=862) giving the number of samples above detection limit (DL), median, mean, minimum (Min) and maximum (Max) concentration values, and the measured concentration at the 5, 25, 50, 75% quantiles. In addition, the standard deviation (Stdev) is given as a measure on variation. All data is given in ppm.

Table A5. Møre and Romsdal data

Summary statistics for the regional survey data (n=472) giving the number of samples above detection limit (DL), median, mean, minimum (Min) and maximum (Max) concentration values, and the measured concentration at the 5, 25, 50, 75% quantiles. In addition, the standard deviation (Stdev) are given as a measure on variation. All data is given in ppm.

Hedmark and Møre and Romsdal counties organic soil APPENDICES

APPENDIX 1: RANDOM PLOTS

Random plots for all samples with field and analytical duplicates in addition to the in-house organic soil standard and re-analyses previous surveys.

Samples from the **Hedmark** county are coloured **grey** whereas those from **More and Romsdal** are shown in **black.**

The laboratory method detection limit (DL) is indicated by a red dotted line.

Samples from the in-house standard are coloured in **green** and samples from a previous survey in **blue**.

Negative laboratory concentration values set to a low positive value to fit the log scale.

page 1 of 9

page 2 of 9

page 3 of 9

Analytical sequence

Analytical sequence

page 4 of 9

page 5 of 9

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16000

16500

Analytical sequence

17000

16000

17000
Analytical sequence

page 6 of 9

page 7 of 9

16500 Analytical sequence

page 8 of 9

APPENDIX 2: X-CHARTS

X-charts comprising splits of the in-house humus standard.

Analyses are plotted in the same order as the analytical sequence in the laboratory.

The median is indicated by a solid line, dashed lines are drawn at ±10% (blue) and ±20% (red) relative to the median.

page 2 of 9

16000 16500 17000

Serial

page 3 of 9

page 4 of 9

page 7 of 9

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0.55

0.60

0.65

0.70

Zr(mg/kg)

0.75

0.80

page 9 of 9

Appendix 3: Re-analysed samples from previous surveys

Appendix 3: Samples re-analysed from previous surveys analytical results (n= 22 samples ; no: 1134, 1265, 1372, 1474, 1502, 1505, 1509, 1511, 1517, 1519, 1523, 1531, 1540, 1559, 1580, 1593, 1657, 1662, 1686, 1728 and 1762) from the Nord Trøndelag and Fosen survey (Finne and Eggen, 2014) which were reanalysed along with the Sør Trøndelag samples in 2020 (Flem et al., 2020), and now for the third time with the present survey. The same laboratory, Bureau Veritas Minerals, Vancouver, Canada, has been used for all three sample collections. For elements with data close to detection limit (DL), this value (DL) was included in the plot as a horizontal line.

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page 2 of 9

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page 3 of 9

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page 6 of 9

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1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 Analytical sequence

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APPENDIX 4: Correla�on plots, field duplicates

The correlation between ordinary and field duplicate samples shown for all elements that have at least four duplicate pairs above DL. Each plot includes the covariance or correlation coefficient.

page 1 of 8

page 3 of 8

page 4 of 8

page 7 of 8

APPENDIX 5: Correlation plots, Analytical duplicates

The correlation between duplicate and analytical duplicate samples shown for all elements that have at least four duplicate pairs above MDL. Each plot includes the covariance or correlation coefficient.

page 1 of 8

page 3 of 8

page 5 of 8

page 8 of 8

APPENDIX 6: ECDF-PLOTs Hedmark data

Plots of the empirical cumulative distribution function (ECDF-plots) for all 53 elements analysed.

All laboratory readings are shown also for those below detection limit, negative reading is replaced by a low positive value (1×10^{-7}) .

The laboratory detection limit (DL) is indicated by a red dotted line for selected elements which have data below or close to this value (DL).

page 1 of 9

page 4 of 9

99.9

APPENDIX 7: ECDF-plots Møre and Romsdal data

Plots of the empirical cumulative distribution function (ECDF-plots) for all 53 elements analysed.

All laboratory readings are shown, including those below detection limit corresponding to negative readings which replaced by a low positive value (1×10^{-7}) .

The laboratory detection limit (DL) is indicated by a red dotted line for selected elements which have data below or close to this value (DL).

page 3 of 9

page 7 of 9

Li(mg/kg)

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