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Chemical composition of black
shale from four locations,
greater Oslo area, Norway

REPORT

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Title: Chemical composition of black shale from four locations, greater Oslo area, Norway		
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Abstract: Chemical analysis of main, minor and trace elements have been made of Ordovician black (alum-) shale samples collected at two different localities and one waste pile in the greater Oslo area.		
This study is aimed at identification of differences between black shales from different localities by using main and trace element concentrations in whole rock for 'fingerprinting'. The study does not attempt to interpret the element data collected in terms of geological history and genesis.		
The results of the chemical analyses have been converted to oxides, treated statistically and plotted in several different types of scatter and triangular/ternary diagrams, which might be used for differentiation and classification of these seemingly homogeneous shales.		
Variation among samples from the same locality has been investigated. When comparing results of the same element in the samples collected at one of the localities (Slemmestad) there are significant differences with respect to several of the elements analyzed. This is highlighted in scatter diagrams.		
Based on different classification methods, e. g. used in classifying metamorphosed shales, the samples from each locality gather in separate clusters. Some of the plots differentiate the samples better than others, and the content of chemical elements can be recommended used in classifying shale samples from different building sites.		
Keywords: black shale	main elements	trace elements
Oslo area	whole rock geochemistry	

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NGU Analytical reports 2007.0458 for respectively:

- main elements by XRF in whole rock, including ignition loss LOI at 1 000°C
- trace elements by ICP after selective acid digestion
- total-C/total-S/total organic carbon (TOC) by Leco upon ignition
- ignition loss at 480°C

1. INTRODUCTION

The composition of black shale may vary locally, both regarding mineral content and geochemical whole-rock composition.

Ordovician black shale from three different localities in and around Oslo has been assessed previously using impregnation-fluorescence petrography on thin sections. Results from that study are presented in NGU report 2007.080 (Broekmans & Sæther 2007).

The current report presents results from whole-rock geochemical assessment of the same black shale materials. Samples have been analysed for main and trace elements by XRF, LOI, Leco for total-S and total-C, and ICP-AES.

2. ANALYTICAL METHODS

2.1 Sample origin

The samples of black shale were collected at two localities within the black shale formation, knowingly at Konowsgate 6-8 (Oslo municipality) from black shale tailings of local origin, and at Heimansåsen at Slemmestad (Røyken municipality), where black shale was sampled *in-situ* (two separate sampling sites labelled C and FE). The third site is a permanent pile of black shale tailings from a construction site in Akersgata (downtown Oslo), deposited on Precambrian basement rocks at Taraldrud/Ski municipality). The sample locations are shown in Figure 1. All samples analysed in this report were collected from the surface.

The representativeness of the collected samples might also be a problem with respect to content of potentially toxic trace components and geotechnical stability over time in contact with groundwater or concrete foundations. The representativeness of the samples with respect to content of chemical elements is addressed in this report.

2.2 Whole rock geochemical analysis

The Ordovician black shales of the Oslo area consist of very fine grained material with a typical grain size below 50 µm. The material is typically very fragile and easily disintegrates into a particulate material with 5-50 mm fragments. Whole rock geochemical analysis to assess the main and trace element composition of these black shales requires a suite of supplementary analytical methods. Details on sample preparation and instrumentation used are given below.

Adequate amounts of random black shale fragments were initially comminuted in a ceramic mortar and pestle to pass a 2 mm mesh sieve, then homogenized and split to ~35g fill an agate-lined vibratory disc-mill for final pulverization.

Powdered bulk material was fused at 1 000 °C in excess Li-tetra borate ($\text{Li}_2\text{B}_4\text{O}_7$) in a platinum crucible conform with the so-called 'Claisse'-method. Weight loss after fusion was recorded as loss on ignition (LOI-1000), and includes both organic (org-C), inorganic carbon (inorg-C; ie. carbonates) as well as water regardless of speciation.

The resulting fusion glass tablet was analyzed by XRF for the oxides Na_2O , K_2O , MgO , CaO , MnO , Al_2O_3 , total- Fe_2O_3 , TiO_2 , SiO_2 , and P_2O_5 . In addition, trace element oxides BaO , Cr_2O_3 , CuO , HfO_2 , NiO , PbO , SrO , V_2O_5 , ZnO , and ZrO_2 were analyzed in the same procedure.

Operating conditions of the PANalytical Axios Minerals® instrument were set at 50 kV and 50 mA, using a Rh target.

In a separate procedure, bulk-LOI was determined gravimetrically by weighing before and after ignition at 480 °C. Organic carbon (org-C) present in the sample oxidizes to CO₂ and loosely bonded water (eg. hydrate, zeolite, clay) evaporates. Obtained values include all of these species and are reported as single values under LOI-480.

Sulfur (total-S), organic carbon (org-C), and organic plus inorganic carbon (total-C) contents were determined by Leco® on separate aliquots. The sample material is heated in an oxidizing atmosphere until 1 450 °C, and the released volatiles are measured spectroscopically in the evading gases.

All element species from XRF are presented as oxides. Sulfur is present in the sample material in various sulfides whereas oxidized sulfur only occurs in very minor amounts in sulfates. Thus, sulfur is tabulated as S. Trace elements by XRF as listed above are not further considered for main element analysis.

Lower limits of detection are specified per species analyzed in Appendix 1. Raw and un-normalized cumulative sum totals of individual whole-rock analyses (XRF + LOI-1000 + Leco) are typically within 100.0 ± 1.00 wt%. Total iron content is reported as Fe₂O₃-total.

A separate amount of powder was dissolved in excess 7N HNO₃ by autoclaving in a microwave oven. Subsequently, the solution was evaporated and redissolved in dilute nitric acid. The final solution was then introduced into the ICP-AES instrument. Concentrated nitric acid at 7N strength is a strong and oxidizing acid that readily dissolves many minerals, including sulfides and carbonates. In contrast, silicate minerals are incongruently dissolved essentially leaving a (alumino-) silicate framework, whereas quartz dissolves hardly at all.

Main element concentrations also analyzed by XRF (Na, K, Mg, Ca, Mn, Al, Fe, Ti, Si and P; see above) were recalculated from the content of oxides using the factors in Table 1 below to facilitate direct comparison between XRF and ICP-AES analytical methods.

Additional trace elements by ICP-AES (Ag, As, Ba, Cd, Co, Cr, Cu, Mo, Ni, Pb, V, Zn, and B, Be, Ce, La, Li, Sc, Sr, Y, Zr) are presented ‘as is’, in parts per million (ppm=mg/kg).

An appropriate number of official and in-house standards was inserted with all analytical procedures. Blind duplicates were inserted throughout the entire sample series, and were pulverized and digested from separate splits.

2.3 Data assessment and presentation

Data have been collated in a spreadsheet using entirely standard Microsoft Office Excel® software, 2007 edition. Triangular plots were produced using Rock Works Grapher® software (by Øystein Nordgulen at NGU).

Main element oxide contents from whole-rock analysis by XRF are presented in wt%. Oxide content data are supplemented with volatile contents determined as LOI-1000, whereas total-C and total-S from Leco is already included in the ignition loss. Cumulative sum totals are within 100 ± 1.00 wt%, with only two exceptions, Slemmestad 5 and 7.

Main element oxides by XRF are compared with data on the same elements by ICP-AES.

Triangular/ternary plots present the relative position of individual samples for the species in each corner. To that purpose, the sum of the species presented in the plot are cumulated per sample and taken as 100 %. Subsequently, the relative proportion of each species is calculated per sample (i.e. 0.0-1.0) as indicated along each side of the triangular plot, determining the sample's position plotted in the triangle.

3. RESULTS

3.1 Main elements

Results from whole rock analysis of main elements by ICP-AES, XRF, loss on ignition at 1 000°C (LOI) and total-S and total-C by Leco are presented in Table 2. Data are arranged per sample and are given in wt% per component oxide.

The first column ("ICP") gives data by ICP-AES after partial digestion in excess acid. Thus, the non-dissolved residue is excluded from the analysis, which is why cumulative sum totals ("CUM SUM") have not been calculated.

The second column ("XRF") lists data on the same oxides as determined by XRF after bulk digestion in excess flux, supplied with data from LOI and Leco. Total-C was measured by Leco but is already included in LOI through thermal decomposition of carbonate present. Total-S is present in sulfides finely dispersed throughout the rock and is hence presented as S, not as oxide. Cumulative sum totals including XRF, LOI and total-S have been calculated and are well within 100.0 ± 1.0 wt%, with only two exceptions (Slemmestad 5 and 7).

The third column ("% ICP/XRF") lists the recovery in percent (dimensionless) of partial digestion in acid by ICP-AES, relative to whole rock by XRF. Values over 100% recovery may be due to calculation artifacts ('rounding off', especially important with low values to begin with; compare e.g. Konowsgate 1 and 2), and essentially represent the same analytical values.

The summary table on the lower right gives calculated values for average (AVG), standard deviation (STDEV) and coefficient of variation (CoV) in percent (rounded off to the next nearest integer) of the recovery rates for all samples. A low standard deviation indicates that *absolute* variation between samples from the same locality are small, whereas the coefficient of variation CoV indicates whether such variation is *relatively* large or small. In general, there is consistency in the oxide content of duplicates and samples from the same localities. However, the content of Fe varies a lot among the Slemmestad samples, whereas the Ca content varies among samples from Konowsgate.

Data from Table 2 are collated graphically in Figures 2, 3, and 4. Each locality is presented by a different marker (see legend).

The triangular plot in Figure 2 shows total alkalis as $\text{Na}_2\text{O} + \text{K}_2\text{O}$, versus $\text{MgO} + \text{Fe}_2\text{O}_3$ on the lower right, versus Al_2O_3 on top. The four different localities seem to define a short trend towards a composition richer in $\text{MgO} + \text{Fe}_2\text{O}_3$, but are still quite close to each other.

The triangular plot in Figure 3 shows MgO versus CaO on the lower right, versus total iron as Fe₂O₃ on top. Here, three locations plot along the MgO-Fe₂O₃ edge, the fourth (Konowsgate) is clearly richer in CaO and plots more towards the center of the triangle. This reflects significant differences with respect to content of carbonates and sulfides (presumably the main host for Fe) in the samples. Samples from Konowsgate and Taraldrud forms two different clusters separate from the Slemmestad F and C samples; samples from Konowsgate are highest in CaO and the Taraldrud samples are highest in MgO. Slemmestad samples are highest in Fe₂O₃, probably from sulfide minerals, possibly also organics.

The triangular plot in Figure 4 shows total iron as Fe₂O₃ versus CaO at lower right, versus Al₂O₃ on top. Once more, three locations plot along the Fe₂O₃-Al₂O₃ edge, the fourth (Konowsgate) is again higher in CaO. This is essentially the same view as Figure 3.

3.2 Trace elements

Trace elements contents determined by ICP-AES are collated in Table 3. Data are arranged in two groups: predominantly chalcophile elements (Ag, As, Ba, Cd, Co, Cr, Cu, Mo, Ni, Pb, V, and Zn, upper part), and lithophile elements (B, Be, Ce, La, Li, Sc, Sr, Y and Zr, lower part). All data are reported in ppm (mg/kg). Maximum and minimum contents are indicated in two rows directly under the actual data. Under these, calculated values for average (AVG), standard deviation (STDEV) and coefficient of variation (CoV) are given in separate rows. Values lower than 2× the lower limit of detection (LLD) are indicated with <[2×LLD].

Data from Table 3 are plotted as scattergraphs in Figures 5, 6, and 7. While these have no geochemical significance, they do serve as a kind of fingerprint to differentiate between sample locations.

Element species are plotted along the horizontal axis in alphabetical order grouped as chalcophile/lithophile, and concentrations are plotted along the vertical axis, in ppm. For all trace element species reported here, the LLD essentially plots on the horizontal axis. All vertical axes present the same scale 0-400 ppm for easy 1:1 comparison. However, Figure 6 has a second vertical axis from 0-4 000 ppm to fit the Slemmestad-FE samples. The data for Slemmestad C are again plotted on a secondary axis of standard 0-400 ppm length.

Figure 5 shows the trace element scatter patterns from Konowsgate, with highest peaks for Ba, Cu, Ce, and Sr.

Figure 6 from Slemmestad shows a Ba peak too, but for the Slemmestad-FE samples, this peak is ~20× as high as for all other samples analyzed in this report. Rather than a peak at Cu, there is a bulge for Cu-Mo-Ni in both Slemmestad-FE and Slemmestad-C (note different axes!), and again a peak for V, Ce, and to a lesser extent for Sr (note axis scales!).

Figure 7 from Taraldrud again shows scatter peaks for Ba, Mo, V, Ce and Zr. Sr is still elevated yet considerably lower than in the two previous figures, whereas also B is elevated.

Summarizing the above, each sample location does indeed have a different fingerprint as illustrated by the very different scattergrams.

The ternary plot in Figure 8 shows Mo versus V at right versus As on top. All sample locations plot comparatively far from each other, including Slemmestad-FE and Slemmestad-C.

The ternary plot in Figure 9 shows Ni versus V at right versus Cr on top. All sample locations plot along the Ni-Cr edge, and Konowsgate nearly coincides with Slemmestad-FE.

The ternary plot in Figure 10 shows Zn versus Cu at right versus Pb on top. All sample locations plot comparatively far from each other, especially Slemmestad FE and C.

Finally, the ternary plot in Figure 11 shows Mn versus Ba at right versus Sr on top. All sample locations plot close to the Ba corner, only Konowsgate plots more towards Mn and again far from Sr.

4. DISCUSSION

4.1 Main elements

The two samples from Taraldrud (Table 2, lower right) seem to be fairly homogeneous with respect to content of all of the main elements showing low standard deviation and coefficient of variation.

The samples from Konowsgate and Slemmestad show the largest standard deviation with respect to the content of CaO and Fe₂O₃. The coefficient of variation is largest in samples from Slemmestad for all elements except CaO and Fe₂O₃.

This reflects significant differences with respect to content of carbonates and sulfides (presumably the main host for Fe) in the samples.

Fe is presented as total-Fe₂O₃, whereas this type of sample material is known to contain some pyrite and other sulfides. This overestimates the wt% of Fe₂O₃ by a certain, unknown, but rather small amount as this includes excess O of the Fe that occurs bonded to S. This artifact will be greatest in the sample containing most S, notably Slemmestad 5-8, of which 5 and 7 have the largest cumulative sum total (see above).

By plotting the results of the main element-oxides in the ternary diagrams (Fig. 2, 3, and 4) it seems like most of the separation is in the MgO-CaO-Fe₂O₃-space. Thus the difference in content of main elements associated with silicates is less than the variation in carbonates and in Fe derived from sulfides in these twelve samples of black shale.

4.2 Trace elements

The data in Table 3 show values below detection limit for Ag in all samples analyzed and close to or under the detection limit for B. The reproducibility of all other elements is within 5-10% except for Ba. The concentrations for some elements in the alum shales analyzed (e.g. Ba, Cu, Ni, and Zn) vary up to one whole order of magnitude. This reflects how the respective elements are bound to shale minerals and the mineralogical composition of a given alum shale horizon.

The clustering of samples from the same locations in the ternary diagrams seem to be best separated in Mo- V-As-, Zn- Cu-Pb-, and Mn-Ba-Sr-space (Figs. 8, 10 and 11). Separation is less distinct in Ni-V-Cr-space (Fig. 9).

5. CONCLUSIONS

Chemical analysis of main, minor and trace elements of twelve Ordovician black (alum-) shale samples collected in the greater Oslo area quantifies the variation between samples at the same localities. The samples collected at each of the localities are significantly different with respect to many elements analyzed. The variation is largest with respect to the content of FeO and CaO at one location (Slemmestad). This is highlighted by the use of scatter diagrams. Further mineralogical assessment requires x-ray diffraction analysis and microscopic studies.

By plotting the data in triangular diagrams, a classification method used in classifying metamorphosed shales, the samples fall into distinct categories. Some of the plots presented differentiate the samples better than others, and these can be recommended used in classifying shale samples from different building sites.

6. REFERENCES

- Broekmans, M. A. T. M. and Sæther, O. M. 2007. Impregnation-fluorescence petrography of black shale from the greater Oslo area. NGU-report 2007.080, 21 p.
- Weast, R. C. (1987; editor): Handbook of chemistry and physics. Section B: The elements and inorganic compounds. 68th edition. CRC Press, Boca Raton, FL 33431: B68-146.

APPENDIX 1: FIGURES

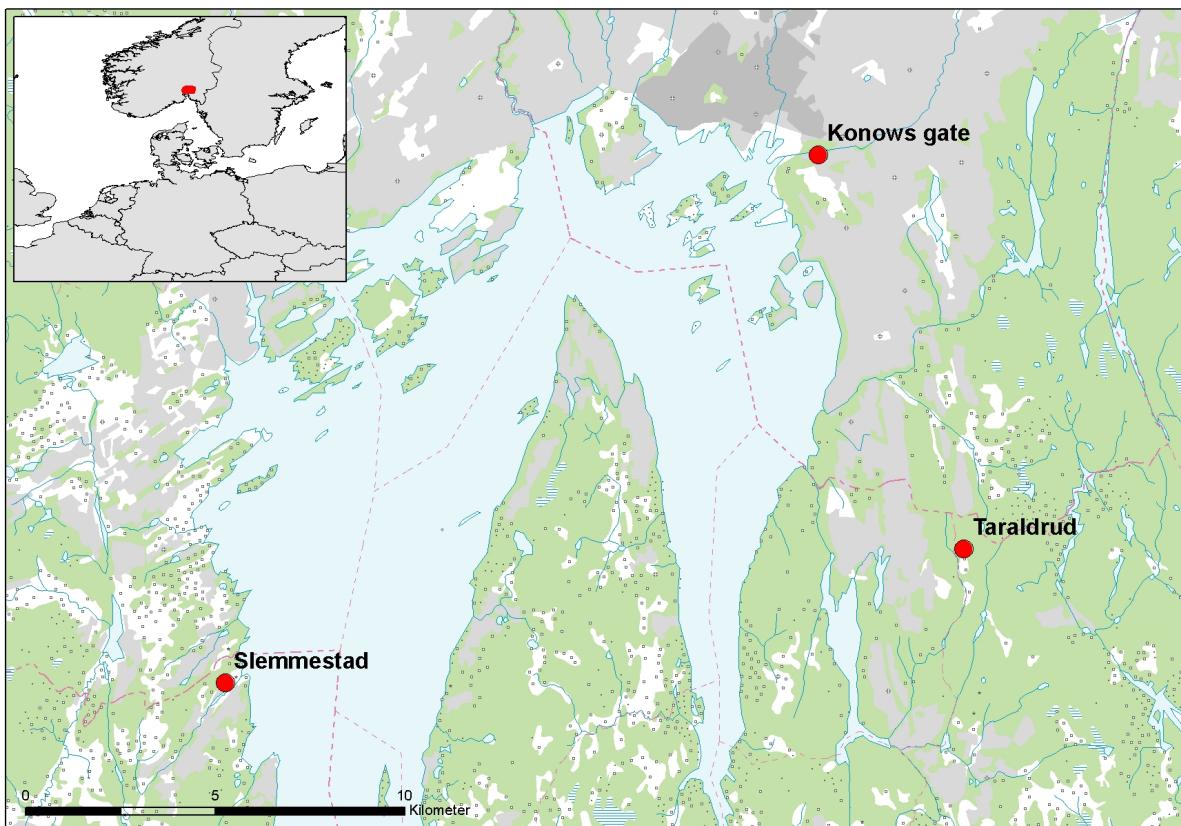


Figure 1: Sampling locations for black shale. Downtown Oslo located near top edge.
(source: <http://earth.google.com>)

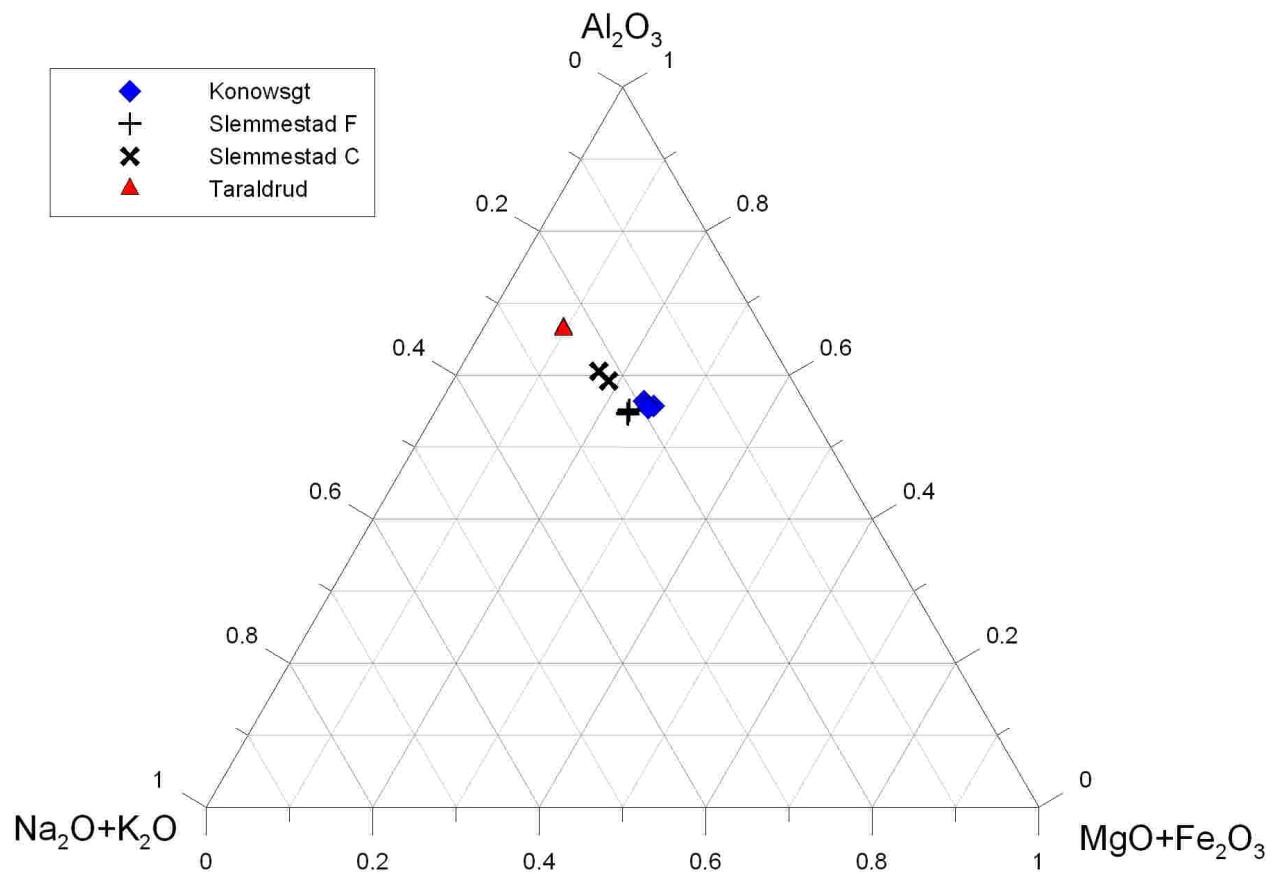


Figure 2: Ternary plot of $\text{Na}_2\text{O} + \text{K}_2\text{O}$ vs. $\text{MgO} + \text{Fe}_2\text{O}_3$ vs. Al_2O_3 , by XRF. Sum contents in wt% for the 2+2+1 species first normalized to 1.00, then individual proportions calculated.

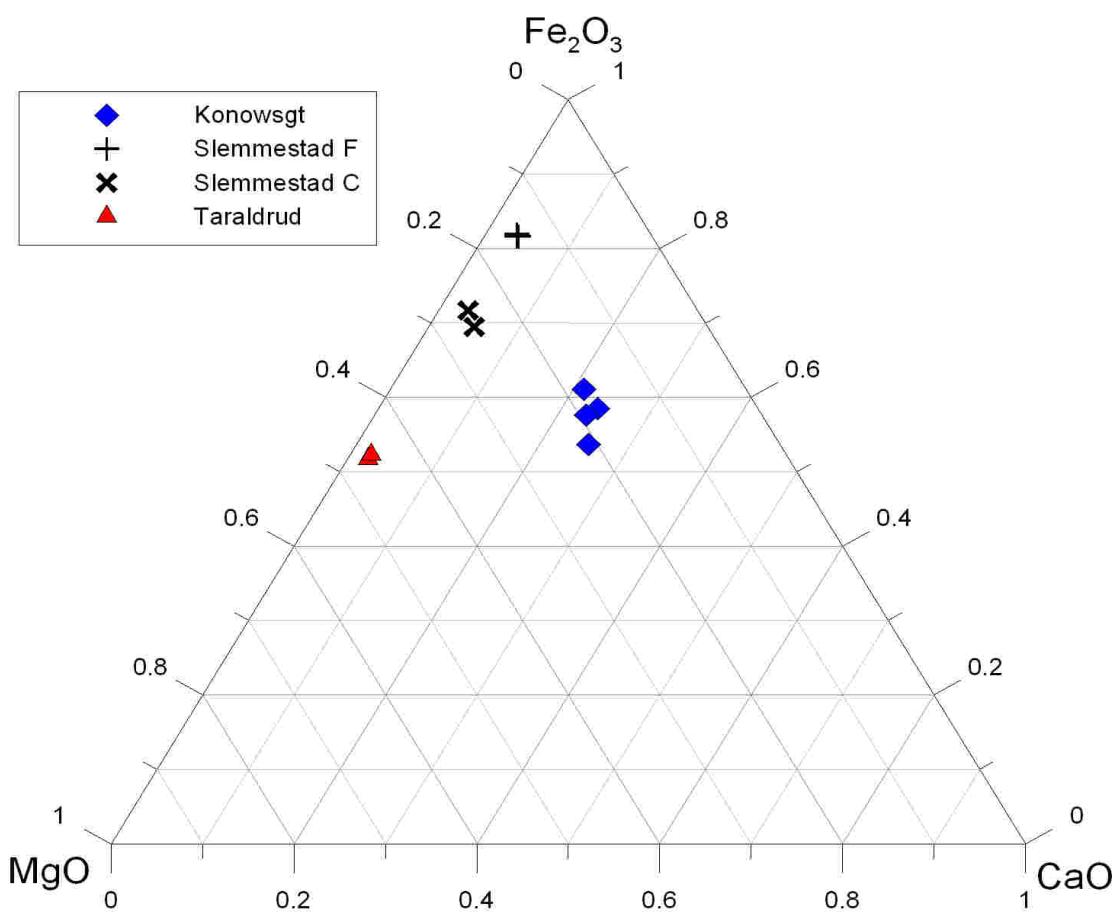


Figure 3: Ternary plot of MgO vs. CaO vs. Fe_2O_3 , by XRF. Sum contents in wt% for the three species first normalized to 1.00, then individual proportions calculated.

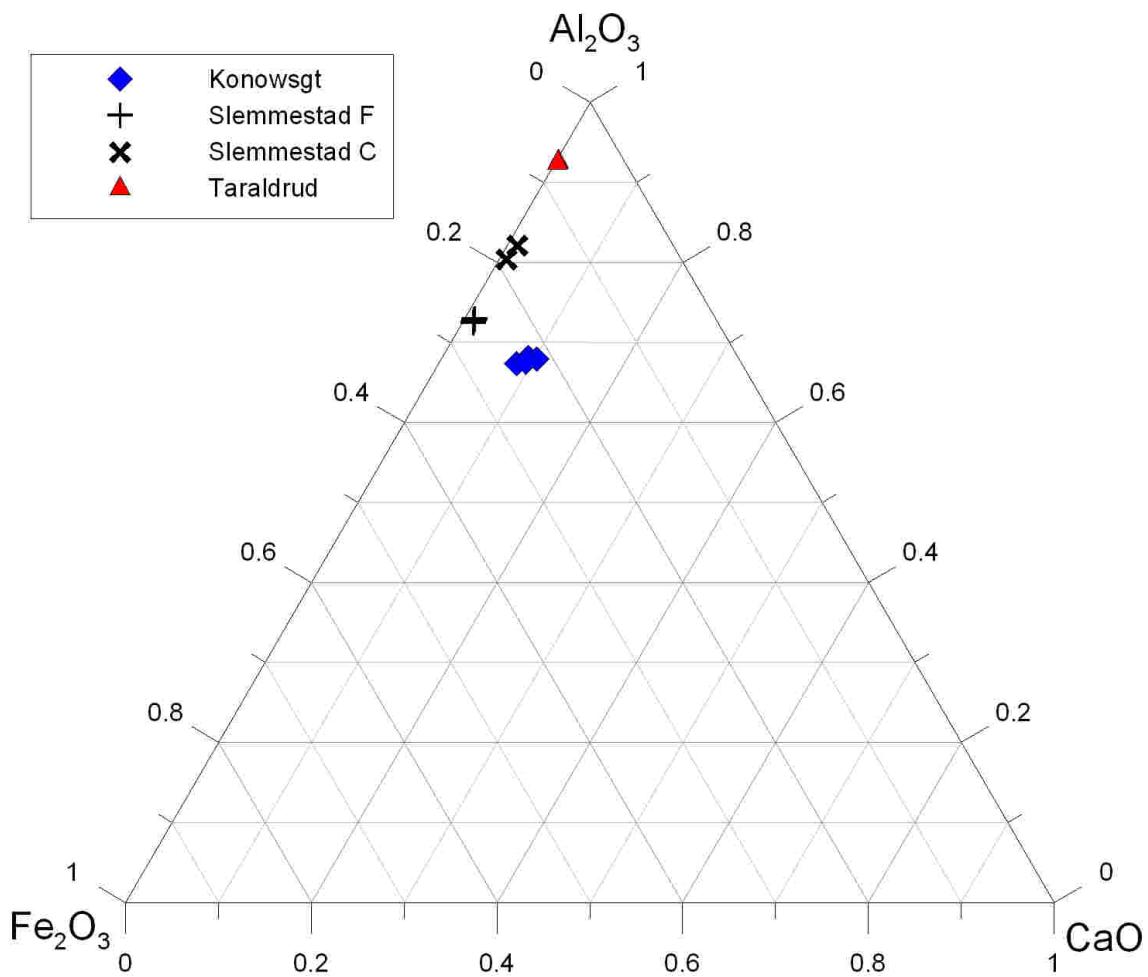


Figure 4: Ternary plot of Fe_2O_3 vs. CaO vs. Al_2O_3 , by XRF. Sum contents in wt% for the three species first normalized to 1.00, then individual proportions calculated.

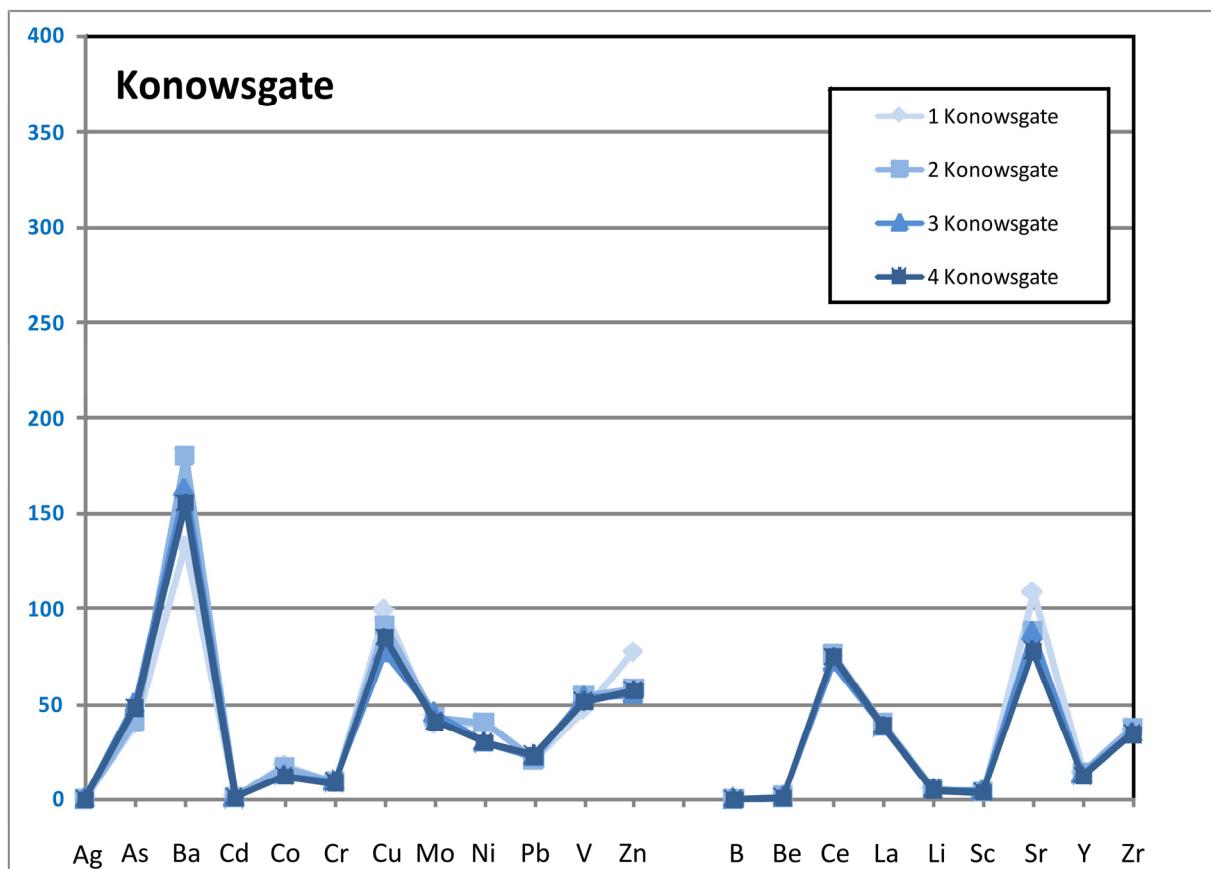


Figure 5: Scattergraph of metallic and lithophile trace elements in black shale from Konowsgate, plotted in alphabetical order. ICP-AES after acid extraction, values in ppm.

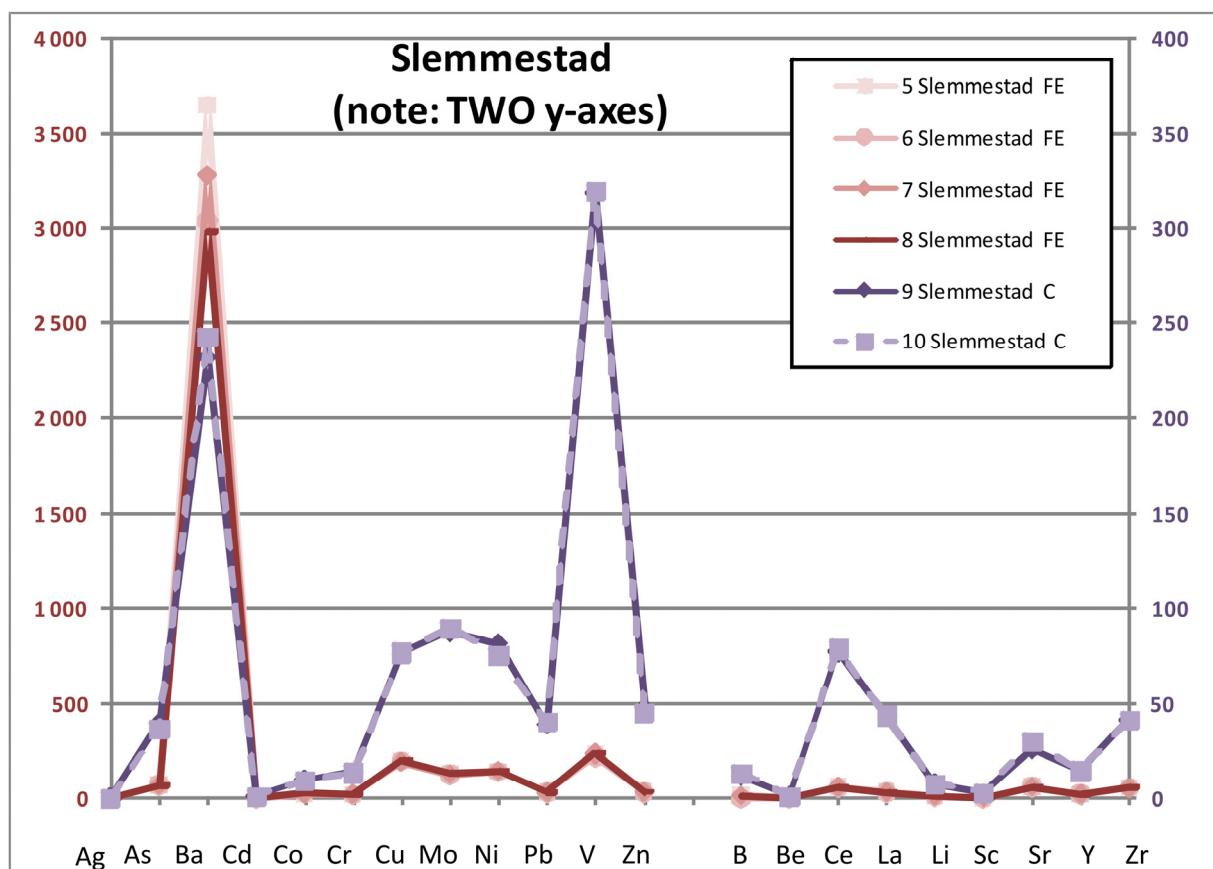


Figure 6: Scattergraph of metallic and lithophile trace elements in black shale from Slemmestad, plotted in alphabetical order. ICP-AES after acid extraction, values in ppm.

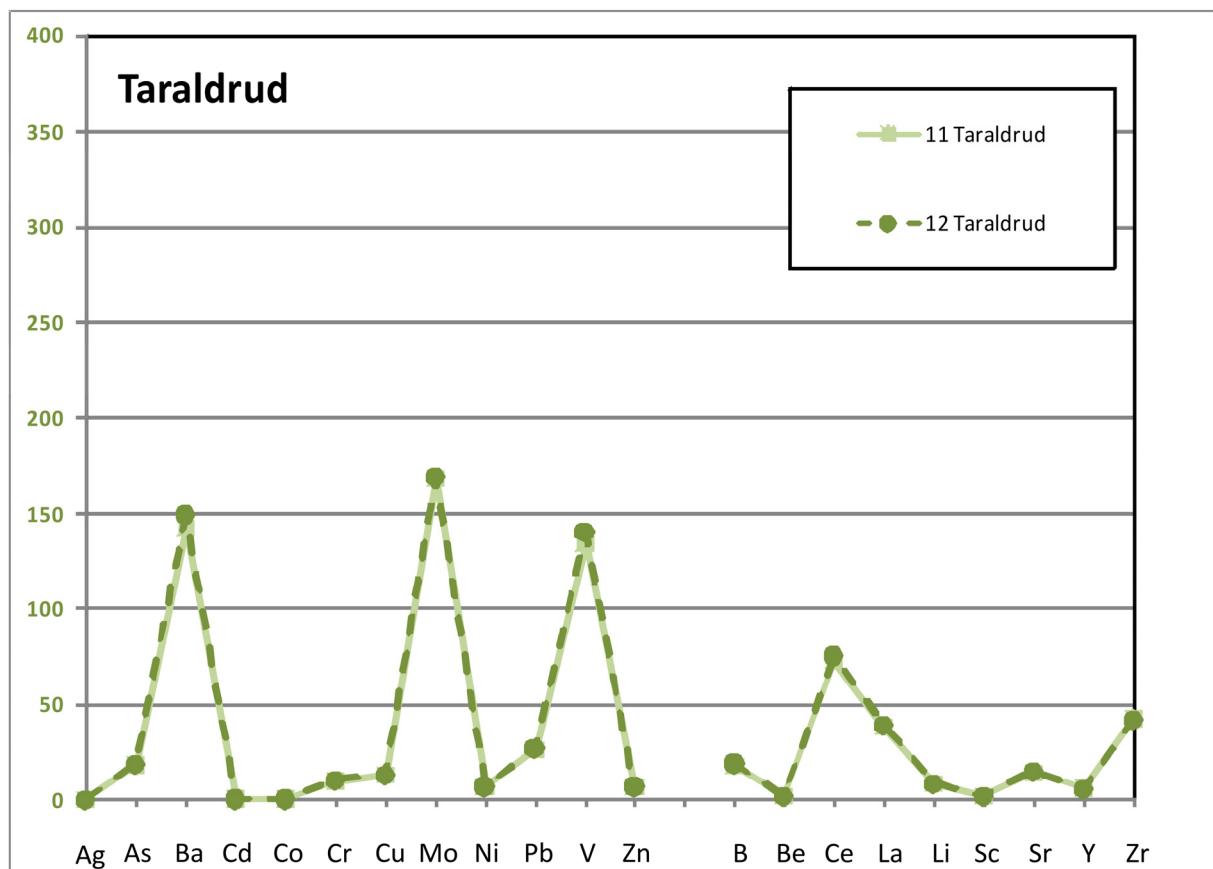


Figure 7: Scattergraph of metallic and lithophile trace elements in black shale from Taraldrud, plotted in alphabetical order.
ICP-AES after acid extraction, values in ppm.

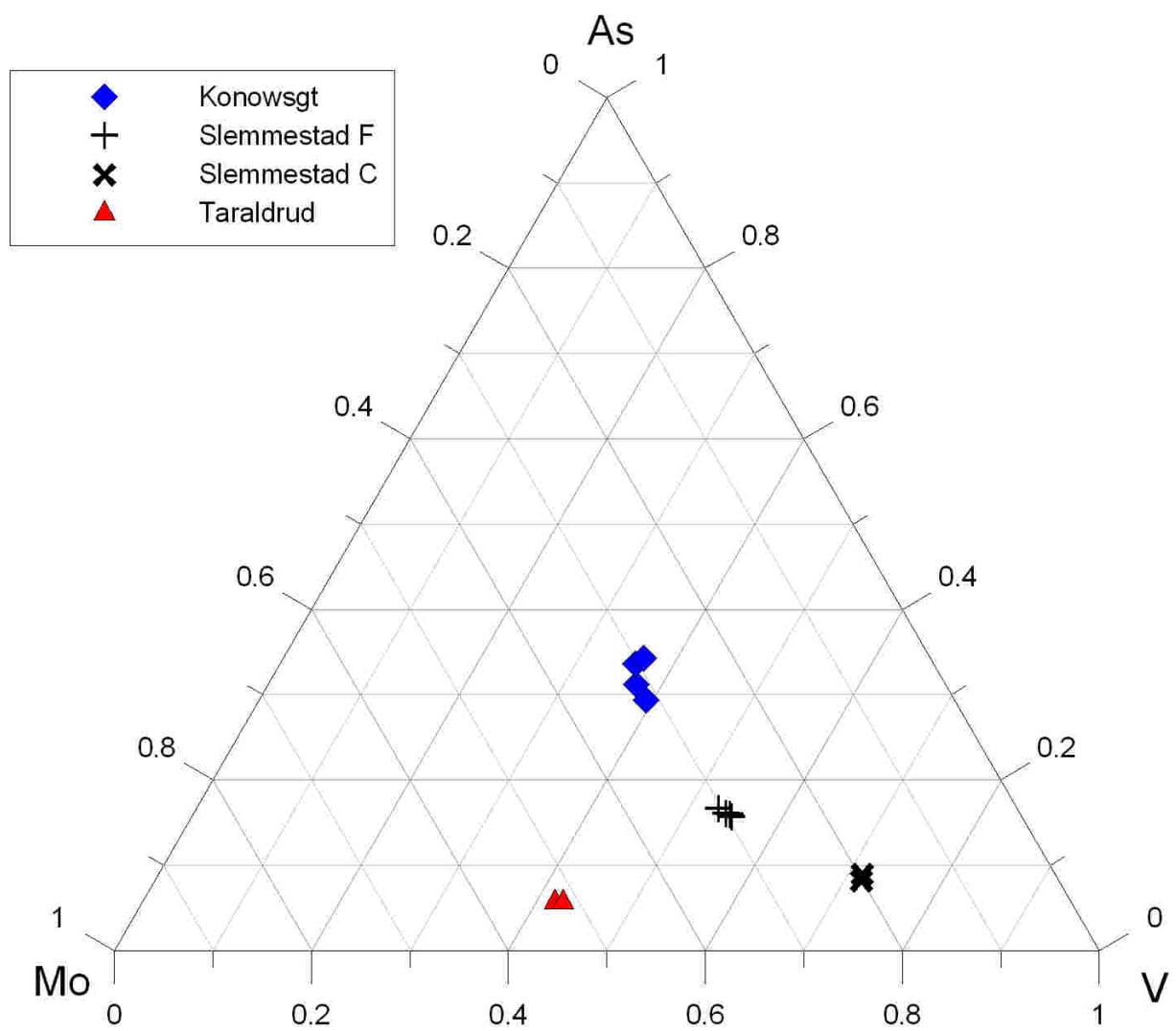


Figure 8: Ternary plot of Mo vs. V vs. As, by ICP-AES after selective acid digestion.
Sum contents in ppm for the three species first normalized to 1.00,
then individual proportions calculated.

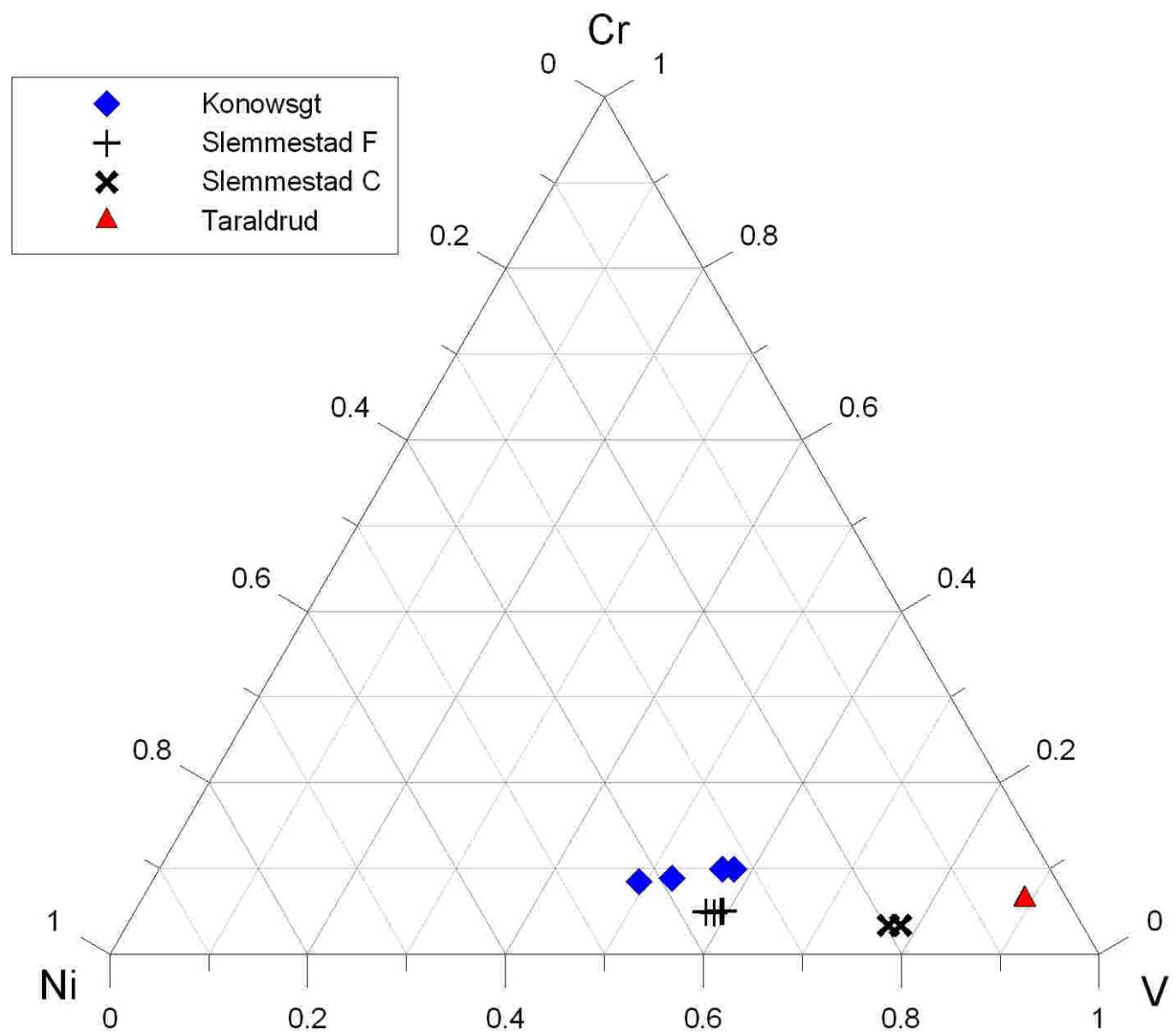


Figure 9: Ternary plot of Ni vs. V vs. Cr, by ICP-AES after selective acid digestion.
Sum contents in ppm for the three species first normalized to 1.00,
then individual proportions calculated.

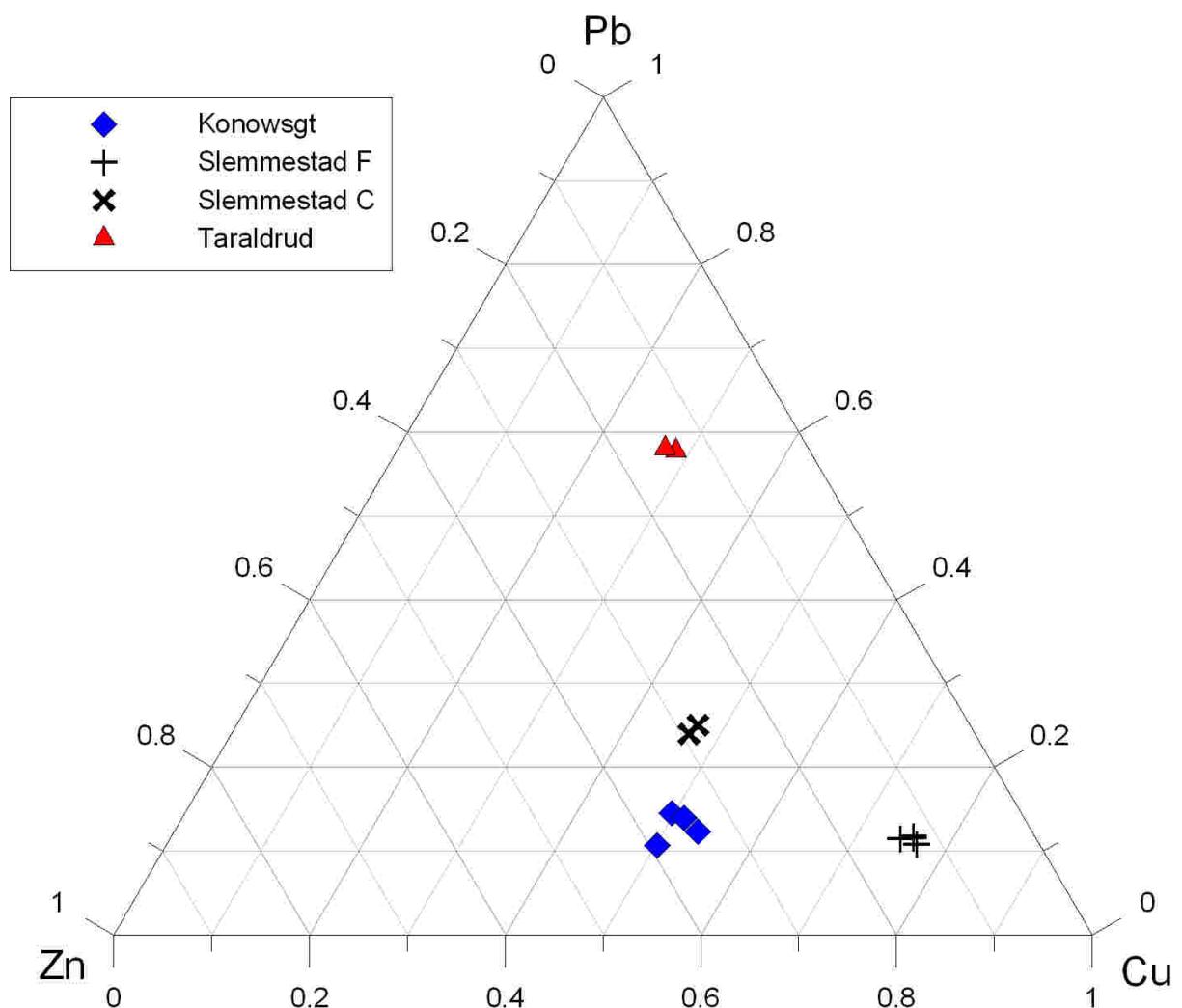


Figure 10: Ternary plot of Zn vs. Cu vs. Pb, by ICP-AES after selective acid digestion.
Sum contents in ppm for the three species first normalized to 1.00,
then individual proportions calculated.

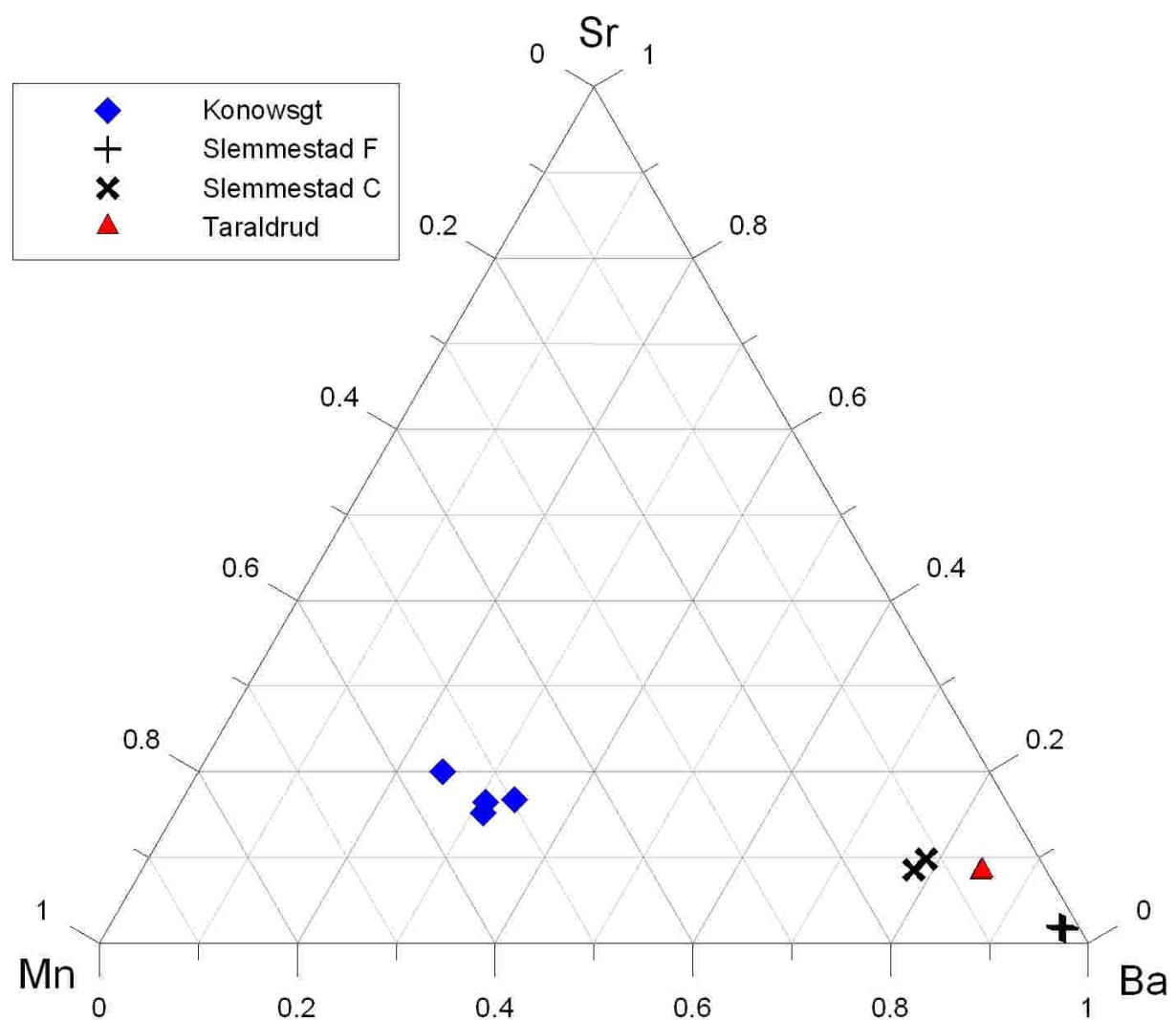


Figure 11: ternary plot of Mn vs. Ba vs. Sr, by ICP-AES after selective acid digestion.
Sum contents in ppm for the three species first normalized to 1.00,
then individual proportions calculated.

APPENDIX 2: TABLES

Table 1: Conversion factors for recalculation of element to oxide weight percentages.

element	oxide	factor
Na	Na ₂ O	1.3480
K	K ₂ O	1.2047
Mg	MgO	1.6581
Ca	CaO	1.3992
Mn	MnO	0.3265
Fe*	Fe ₂ O ₃	1.4297
Al	Al ₂ O ₃	1.8894
Si	SiO ₂	2.1391
Ti	TiO ₂	1.6683
P	P ₂ O ₅	2.2916
C**	CO ₂	3.6641

*: to represent total iron content as Fe₂O₃-total

**: for recalculation of [total-organic]-C to CO₂

Table 2: Main elements in wt%, in whole rock by XRF and by ICP-AES after selective acid digestion. Ignition loss LOI determined in conjunction with XRF. Total-S determined in separate procedure with Leco. Recovery of contents by ICP-AES relative to XRF calculated in % (dimensionless). Further explanation in text.

SampleLabel	unit	notes	Konowsat 20070320S1	% ICP/XRF	ICP	XRF	% ICP/XRF	ICP	XRF	% ICP/XRF	ICP	XRF	% ICP/XRF	ICP	XRF	% ICP/XRF
SiO2	wt%		0.10	54.30	0	0.07	54.80	0	0.09	55.10	0	0.07	55.40	0		
Al2O3	wt%		1.50	15.10	10	1.68	15.20	11	1.50	14.60	10	1.52	14.30	11		
Fe2O3-total	wt%		4.36	5.02	87	4.36	5.47	80	4.70	4.69	100	4.37	4.90	89		
TiO2	wt%		0.01	0.88	1	0.01	0.88	1	0.01	0.84	1	0.01	0.83	1		
MgO	wt%		0.54	1.50	36	0.66	1.59	42	0.97	1.83	53	0.82	1.63	50		
CaO	wt%		1.89	2.06	92	1.82	1.90	96	2.56	2.22	115	2.21	1.97	112		
Na2O	wt%		0.07	0.32	22	0.06	0.32	19	0.07	0.28	25	0.07	0.28	25		
K2O	wt%		0.48	4.82	10	0.53	4.71	11	0.54	4.70	11	0.54	4.66	12		
MnO	wt%		0.04	0.04	93	0.04	0.04	100	0.03	0.03	88	0.03	0.03	111		
P2O5	wt%		0.07	0.08	89	0.07	0.09	77	0.08	0.07	111	0.08	0.08	104		
SUM TOTAL	wt%		9.06	84.12		9.29	85.00		10.54	84.37		9.71	84.07			
S-total	Leco			2.72		2.80			2.68			2.77				
LOI1000	wt%			12.80		12.70			13.20			13.50				
CUM SUM	wt%			99.64		100.50			100.25			100.34				
Konowsat 20070322S2																
SiO2	wt%		0.06	53.50	0	0.11	51.60	0	0.06	53.20	0	0.10	57.30	0	0.09	57.40
Al2O3	wt%		2.15	16.40	13	1.98	15.80	13	3.98	16.20	25	2.21	15.80	14	1.62	17.30
Fe2O3-total	wt%		5.15	5.86	88	5.10	5.68	90	4.99	5.77	86	4.99	5.73	87	3.23	3.53
TiO2	wt%		0.02	0.86	2	0.01	0.84	2	0.01	0.85	2	0.01	0.84	2	0.01	0.97
MgO	wt%		0.35	1.05	34	1.03	0.35	34	0.35	1.05	33	0.35	1.03	34	0.24	1.30
CaO	wt%		0.27	0.25	110	0.29	0.25	115	0.26	0.25	104	0.26	0.26	98	0.20	0.18
Na2O	wt%		0.08	0.68	12	0.08	0.68	12	0.08	0.68	12	0.06	0.69	12	0.06	0.81
K2O	wt%		0.72	5.76	13	0.66	5.68	12	1.45	5.71	25	0.73	5.68	13	0.57	5.60
MnO	wt%		0.01	<0.01		0.01	<0.01		0.01	<0.01		0.01	<0.01		0.01	<0.01
P2O5	wt%		0.17	0.18	93	0.18	0.19	95	0.33	0.19	178	0.16	0.20	82	0.02	0.03
SUM TOTAL	wt%		8.98	84.54		8.76	81.74	11	11.52	83.90	14	8.86	81.93	11	6.03	87.53
S-total	Leco			3.88		3.79			3.74			3.82		2.54		2.27
LOI1000	wt%			14.90		14.90			15.20			15.20		9.61		9.71
CUM SUM	wt%			103.32		100.43			102.84			100.95		99.68		99.23
Konowsat 20070323S1																
SiO2	wt%		0.09	55.20	0	0.11	54.40	0	0.09	55.10	0	0.10	57.30	0	0.09	57.40
Al2O3	wt%		0.56	16.70	9	1.60	16.40	10	2.21	15.80	14	1.62	17.30	9		
Fe2O3-total	wt%		0.56	1.28	44	0.58	1.29	45	0.58	1.29	89	0.58	1.29	87	3.23	3.53
TiO2	wt%		0.01	0.89	1	0.01	0.88	1	0.01	0.88	1	0.01	0.88	1	0.01	0.97
MgO	wt%		0.23	1.14	20	0.24	1.12	21	0.24	1.12	45	0.24	1.12	21	0.21	1.30
CaO	wt%		0.07	0.06	118	0.07	0.06	129	0.06	0.06	104	0.06	0.06	106	0.07	0.07
Na2O	wt%		0.07	0.67	10	0.07	0.65	11	0.07	0.65	23	0.07	0.65	11	0.07	0.65
K2O	wt%		0.47	5.33	9	0.48	5.28	9	0.48	5.28	11	1	7	14	6	42
MnO	wt%		0.01	<0.01		0.01	<0.01		0.01	<0.01	98	10	10	below LLD	9	0
P2O5	wt%		0.02	0.03	80	0.02	0.03	77	0.02	0.03	95	15	16	94	44	78
SUM TOTAL	wt%		3.08	81.29	4	3.19	80.10	4	3.19	80.10	4	3.19	80.10	4	3	3
Slommestad 20070320S1																
SiO2	wt%		0	0	24	0	0	0	0	0	28	0	0	0	0	14
Al2O3	wt%		10	0	5	14	6	41	10	0	0	0	0	0	0	3
Fe2O3-total	wt%		89	8	10	85	4	44	5	0	32	1	0	1	2	4
TiO2	wt%		1	0	8	1	0	0	0	0	28	8	29	21	1	3
MgO	wt%		45	8	18	45	8	18	12	11	106	7	124	12	8	6
CaO	wt%		104	12	11	104	12	11	3	13	10	2	19	10	1	9
Na2O	wt%		23	3	13	23	3	13	11	1	116	7	124	12	8	6
K2O	wt%		11	1	7	11	1	7	14	6	106	7	124	12	8	6
MnO	wt%		98	10	10	98	10	10	below LLD	below LLD	98	10	10	below LLD	9	0
P2O5	wt%		95	15	16	95	15	16	94	44	47	47	47	78	2	3
S-total	Leco		1.47			1.47			1.48			1.48				
LOI1000	wt%		17.50			17.50			18.00			18.00				
CUM SUM	wt%		100.26			100.26			99.59			99.59				

Table 3: Trace elements in ppm by ICP-AES after selective acid digestion. Coefficient of variation CoV calculated as percentage of STDEV relative to AVG. Further explanation in text.

Sample# unit	Ag*	As	Ba	Cd*	Co	Cr	Cu	Mo	Ni	Pb	V	Zn
LLD	in ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
1 Konowsgt 20070320S1	<4	38,8	133	1,0	17,5	8,1	98,5	39,1	40,0	20,9	46,6	76,8
2 Konowsgt 20070321S1	<4	40,5	180	0,7	16,7	9,2	91,2	43,3	40,1	21,0	54,3	58,2
3 Konowsgt 20070322S2	<4	50,1	163	0,7	12,4	9,2	76,9	45,1	29,6	22,4	53,8	55,1
4 Konowsgt 20070323S2	<4	48,0	155	0,7	11,9	8,9	84,5	40,7	29,8	22,9	51,2	57,2
5 SlommestadFE20070320S1	<4	69,1	3650	1,1	23,4	19,8	197,0	128,0	141,0	30,3	236,0	36,5
6 SlommestadFE20070320S2	<4	69,1	3040	1,2	23,7	18,5	194,0	126,0	142,0	30,0	220,0	31,9
7 SlommestadFE20070320S3	<4	67,0	3280	1,2	23,2	19,6	191,0	126,0	141,0	29,0	234,0	31,4
8 SlommestadFE20070320S4	<4	69,1	2990	1,1	24,1	19,1	201,0	129,0	145,0	28,3	233,0	33,0
9 SlommestadC20070320S1	<4	39,9	232	1,1	9,6	14,0	75,8	87,4	81,3	38,9	319,0	47,4
10 SlommestadC20070321S1	<4	36,6	243	1,1	9,1	13,6	76,5	89,5	75,2	40,6	320,0	45,0
11 Taraldrud20070320S1	<4	18,1	142	0,2	0,4	9,7	12,9	168,0	6,5	26,1	134,0	6,2
12 Taraldrud20070321S1	<4	18,5	149	0,2	0,4	10,0	12,7	169,0	6,7	27,0	140,0	6,8
MIN in ppm		0	18,1	133	0,2	0,4	8,1	12,7	39,1	6,5	20,9	6,2
MAX in ppm		0	69,1	3650	1,2	24,1	19,8	201,0	169,0	145,0	40,6	320,0
Avg in ppm			<4	47,1	1196	0,9	14,4	13,3	109,3	99,3	73,2	28,1
STDEV in ppm			0	18,6	1518	0,3	8,6	4,8	69,3	48,7	55,6	6,4
CoV in %			-	39,4	127	40,9	59,6	35,8	63,4	49,1	76,0	22,7
												50,9
												51,3

Sample# unit	B	Be	Ce	La	Li	Sc	Sr	Y	Zr	
LLD	in ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	
1 Konowsgt 20070320S1	<10	1,1	75,3	39,0	5,3	2,9	108,0	14,0	36,1	
2 Konowsgt 20070321S1	<10	1,1	75,7	39,7	5,7	3,4	87,8	14,1	37,6	
3 Konowsgt 20070322S2	<10	1,0	71,4	38,1	4,8	4,3	87,3	12,9	34,8	
4 Konowsgt 20070323S2	<10	0,9	74,4	38,6	5,1	3,6	77,6	12,2	34,1	
5 SlommestadFE20070320S1	14,2	1,8	59,3	33,1	12,7	3,9	60,1	20,0	54,8	
6 SlommestadFE20070320S2	<10	1,8	57,1	32,0	12,1	3,7	58,9	20,9	54,1	
7 SlommestadFE20070320S3	12,8	1,8	57,5	31,7	12,3	3,8	59,2	19,8	53,7	
8 SlommestadFE20070320S4	12,7	1,8	57,5	31,6	12,3	3,9	61,2	20,6	54,8	
9 SlommestadC20070320S1	11,6	1,0	76,9	43,3	7,5	2,6	25,6	14,2	41,3	
10 SlommestadC20070321S1	13,0	1,0	78,4	43,5	7,6	2,7	30,3	14,5	41,3	
11 Taraldrud20070320S1	17,7	1,5	72,7	38,0	8,2	1,7	13,9	5,8	42,5	
12 Taraldrud20070321S1	18,9	1,5	75,3	39,2	8,1	1,8	14,9	5,8	41,8	
MIN in ppm		11,6	0,9	57,1	31,6	4,8	1,7	13,9	5,8	34,1
MAX in ppm		18,9	1,8	78,4	43,5	12,7	4,3	108,0	20,9	54,8
Avg in ppm		14,4	1,4	69,3	37,3	8,5	3,2	57,1	14,6	43,9
STDEV in ppm		2,8	0,4	8,6	4,3	3,1	0,8	30,5	5,2	8,2
CoV in %		19,3	27,2	12,5	11,4	36,4	26,4	53,5	35,6	18,6

APPENDIX 3: ORIGINAL DATA

XRF ANALYSER
GEOLOGISK MATERIALE
ANALYSEKONTRAKTNR. 2007.0458



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 Telefaks: 73 92 16 20



4.2 g Li₂B₄O₇ + 0.6 g prøve smeltet til glassplate

Analyseprogram : HOVEELEMENT

Analysene er utført på glødete prøver (v/1000 °C)

INSTRUMENT TYPE : PANalytical Axios 4 kW x-ray spectrometer (Rh-røntgenrør)

Analyse-data er regnet tilbake fra glødet- til mottatt-prøve

NEDRE BESTEMMELSESGRENSER FOR HOVEELEMENTER angitt i %

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	MgO	CaO	Na ₂ O	K ₂ O	MnO	P ₂ O ₅
0,5	0,02	0,01	0,01	0,04	0,01	0,1	0,01	0,01	0,01

BaO*	Cr ₂ O ₃ *	CuO*	HfO ₂ *	NiO*	PbO*	SrO*	V ₂ O ₅ *	ZnO*	ZrO ₂ *
0,025	0,02	0,01	0,02	0,01	0,01	0,04	0,02	0,01	0,02

*) Omfattes ikke av akkreditering

ANALYSEUSIKKERHET : Analyseusikkerheten er beregnet fra regresjonsanalyse av internasjonale standarder, hvor det er benyttet en veid regresjonsmodell.

Usikkerheten er gitt ved: **USIKKERHET = $\pm K_{Element} \cdot \sqrt{0,1 + C_i}$ [%]** [1] men dog aldri < 1 % relativ

hvor C_i er den rapporterte konsentrasjon i %, $K_{Element}$ er gitt for de enkelte element i tabellen nedenfor

Element	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	MgO	CaO	K ₂ O	Na ₂ O	MnO	P ₂ O ₅	BaO*	Cr ₂ O ₃ *	CuO*	HfO ₂ *	NiO*	PbO*	SrO*	V ₂ O ₅ *	ZnO*	ZrO ₂ *
$K_{ELEMENT} / \%^{\frac{1}{2}}$	0,09	0,04	0,06	0,02	0,08	0,06	0,04	0,07	0,01	0,02										

De oppgitte usikkerhetene er for 1σ nivå (68% konfidensnivå), ved å multiplisere usikkerheten med 2 oppnås et 95% konfidensnivå.

EKSEMPEL

Det er rapportert et analyseresultat på 20.0% (dvs. $C_i = 20.0\%$) for Al₂O₃. Denne konsentrasjonen samt K -verdien fra tabellen over innsatt i likn. [1] gir:

$$\text{USIKKERHET} = \pm 0.04 \cdot \sqrt{0.1 + 20} = 0.2 [\%]$$

Et konfidensintervall på 68%-nivå vil da bli: $20.0 \pm 0.2\%$, og konfidensintervallet på 95%-nivå: $20.0 \pm 0.4\%$

PRESISJON : Det kjøres rutinemessig kontrollprøver, som føres i kontrolldiagram (X-diagram). Disse kan forevises om ønskelig.

ANTALL PRØVER: 12

ANMERKNINGER: Ingen

Rapporten må ikke gjengis i utdrag uten skriftlig godkjenning fra NGU-Lab.

Ferdig analysert		13.feb.08	Bjørn Nilsen
		Dato	Operator

XRF ANALYSER
GEOLOGISK MATERIALE
ANALYSEKONTRAKTNR. 2007.0458



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Prøve id. *	SiO ₂ [%]	Al ₂ O ₃ [%]	Fe ₂ O ₃ [%]	TiO ₂ [%]	MgO [%]	CaO [%]	Na ₂ O [%]	K ₂ O [%]	MnO [%]	P ₂ O ₅ [%]	Gl.tap [%]	Sum Hoved [%]
1	54,3	15,1	5,02	0,878	1,50	2,06	0,32	4,82	0,043	0,079	12,8	96,8
2	54,8	15,2	5,47	0,882	1,59	1,90	0,32	4,71	0,040	0,091	12,7	97,7
3	55,1	14,6	4,69	0,844	1,83	2,22	0,28	4,70	0,034	0,072	13,2	97,6
4	55,4	14,3	4,90	0,827	1,63	1,97	0,28	4,66	0,027	0,077	13,5	97,5
5	53,5	16,4	5,86	0,860	1,05	0,246	0,68	5,76	<0.01	0,183	14,9	99,5
6	51,6	15,8	5,68	0,836	1,03	0,248	0,68	5,68	<0.01	0,189	14,9	96,7
7	53,2	16,2	5,77	0,853	1,05	0,251	0,68	5,71	<0.01	0,185	15,2	99,1
8	51,7	15,8	5,73	0,844	1,03	0,264	0,69	5,68	<0.01	0,196	15,2	97,2
9	57,3	17,2	4,02	0,975	1,41	0,180	0,81	5,60	<0.01	0,033	9,61	97,1
10	57,4	17,3	3,53	0,971	1,30	0,253	0,82	5,64	<0.01	0,036	9,71	96,9
11	55,2	16,7	1,28	0,888	1,14	0,055	0,67	5,33	<0.01	0,025	17,5	98,8
12	54,4	16,4	1,29	0,882	1,12	0,056	0,65	5,28	<0.01	0,026	18,0	98,1

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 Tlf.: 73 90 40 00
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	BaO* [%]	Cr ₂ O ₃ * [%]	CuO* [%]	HfO ₂ * [%]	NiO* [%]	PbO* [%]	SrO* [%]	V ₂ O ₅ * [%]	ZnO* [%]	ZrO ₂ * [%]
1	0,144	<0.02	<0.01	<0.02	<0.01	0,021	<0.04	0,080	<0.01	0,020
2	0,157	<0.02	<0.01	<0.02	<0.01	0,021	<0.04	0,076	<0.01	0,021
3	0,151	<0.02	<0.01	<0.02	<0.01	0,020	<0.04	0,066	<0.01	0,021
4	0,146	<0.02	<0.01	<0.02	<0.01	0,021	<0.04	0,067	<0.01	0,023
5	1,95	<0.02	0,020	<0.02	0,024	0,023	<0.04	0,148	<0.01	0,022
6	1,93	<0.02	0,018	<0.02	0,026	0,024	<0.04	0,144	<0.01	0,022
7	1,94	<0.02	0,021	<0.02	0,025	0,020	<0.04	0,148	<0.01	0,022
8	1,92	<0.02	0,020	<0.02	0,023	0,023	<0.04	0,145	<0.01	0,021
9	0,273	<0.02	<0.01	<0.02	0,013	0,023	<0.04	0,383	<0.01	0,023
10	0,277	<0.02	<0.01	<0.02	0,014	0,021	<0.04	0,375	<0.01	0,025
11	0,231	<0.02	<0.01	<0.02	<0.01	0,020	<0.04	0,149	<0.01	0,023
12	0,231	<0.02	<0.01	<0.02	<0.01	0,022	<0.04	0,142	<0.01	0,022

Metoden anvendes på analyseløsninger fremstilt ved ekstraksjon med 7 N HNO₃ i autoklav i samsvar med Norsk Standard - NS 4770

Analysen er således basert på partiell syreekstraksjon i 7N HNO₃ og de rapporterte analyseverdier representerer derfor ikke totalverdier i prøven

INSTRUMENT TYPE : Perkin Elmer Optima 4300 Dual View

NEDRE BESTEMMELSESGRENSER FOR PLASMA ANALYSER BASERT PÅ AUTOKLAVEKSTRAKSJON (1 g prøve i 100 ml analysevolum)

(For analyser med tynningsfaktor som avviker fra 100, blir deteksjonsgrensene automatisk omregnet).

Si* ppm	Al ppm	Fe ppm	Ti ppm	Mg pp	Ca ppm	Na ppm	K ppm	Mn ppm	P ppm	Cu ppm	Zn ppm	Pb ppm	Ni ppm	Co ppm
100	20	2	1	100	200	200	100	0,2	10	0,5	1	1	1	0,1

V ppm	Mo ppm	Cd ppm	Cr ppm	Ba ppm	Sr ppm	Zr ppm	Ag ppm	B ppm	Be ppm	Li ppm	Sc ppm	Ce ppm	La ppm	Y ppm	As ppm
1	0,5	0,1	0,2	1	1	1	2	5	0,2	1	0,1	2	1	0,1	2

*)NGU-lab er ikke akkreditert for Si (geologisk materiale).

(1 mg/kg = 1 ppm)

ANALYSEUSIKKERHET For samtlige elementer regnes med en total usikkerhet i ekstraksjon og analyse på $\pm 10\%$ rel.

PRESISJON : Det kjøres rutinemessig kontrollprøver, som føres i kontrolldiagram (X-diagram).
Disse kan forevises om ønskelig.

ANTALL PRØVER: 12

ANMERKNINGER: Ingen

Rapporten må ikke gjengis i utdrag uten skriftlig godkjenning fra NGU-Lab.

Ferdig analysert	13-des-07	Baard Søberg
Dato		OPERATØR

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	7491 TRONDHEIM Tlf.: 73 90 40 00 Telefaks: 73 92 16 20															
	Si	Al	Fe	Ti	Mg	Ca	Na	K	Mn	P	Cu	Zn	Pb	Ni	Co	
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	
Konowsgt. 20070320S1	488	7920	30500	38,3	3230	13500	544	3960	298	310	98,5	76,8	20,9	40,0	17,5	
Konowsgt. 20070321S1	308	8910	30500	42,7	3980	13000	429	4400	309	320	91,2	58,2	21,0	40,1	16,7	
Konowsgt. 20070322S2	413	7920	32900	44,2	5840	18300	499	4510	279	370	76,9	55,1	22,4	29,6	12,4	
Konowsgt. 20070323S2	306	8040	30600	41,4	4950	15800	532	4450	229	347	84,5	57,2	22,9	29,8	11,9	
SlemmestadFE20070320S1	271	11400	36000	92,3	2140	1930	579	5960	60,9	743	197	36,5	30,3	141	23,4	
SlemmestadFE20070320S2	498	10500	35700	82,9	2100	2040	613	5490	57,7	779	194	31,9	30,0	142	23,7	
SlemmestadFE20070320S3	275	11700	34900	87,4	2120	1870	615	6020	57,4	718	191	31,4	29,0	141	23,2	
SlemmestadFE20070320S4	427	11700	35400	81,6	2150	1930	568	6040	58,4	746	201	33,0	28,3	145	24,1	
SlemmestadC20070320S1	461	8480	22600	47,4	1440	1420	471	4730	39,7	98	75,8	47,4	38,9	81,3	9,61	
SlemmestadC20070321S1	403	8590	19500	48,7	1450	1770	495	4890	35,4	109	76,5	45,0	40,6	75,2	9,05	
Taraldrud20070320S1	418	8280	3900	56,4	1400	465	516	3920	11,2	69	12,9	6,2	26,1	6,5	0,42	
Taraldrud20070321S1	505	8470	4050	59,4	1420	516	502	3980	11,3	72	12,7	6,8	27,0	6,7	0,44	
	7491 TRONDHEIM Tlf.: 73 90 40 00 Telefaks: 73 92 16 20															
	V	Mo	Cd	Cr	Ba	Sr	Zr	Ag	B	Be	Li	Sc	Ce	La	Y	As
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	
Konowsgt. 20070320S1	46,6	39,1	0,96	8,05	133	108	36,1	<4	<10	1,13	5,3	2,91	75,3	39,0	14,0	38,8
Konowsgt. 20070321S1	54,3	43,3	0,70	9,22	180	87,8	37,6	<4	<10	1,12	5,7	3,40	75,7	39,7	14,1	40,5
Konowsgt. 20070322S2	53,8	45,1	0,68	9,15	163	87,3	34,8	<4	<10	0,95	4,8	4,30	71,4	38,1	12,9	50,1
Konowsgt. 20070323S2	51,2	40,7	0,67	8,87	155	77,6	34,1	<4	<10	0,93	5,1	3,59	74,4	38,6	12,2	48,0
SlemmestadFE20070320S1	236	128	1,11	19,8	3650	60,1	54,8	<4	14,2	1,83	12,7	3,86	59,3	33,1	20,0	69,1
SlemmestadFE20070320S2	220	126	1,19	18,5	3040	58,9	54,1	<4	<10	1,76	12,1	3,70	57,1	32,0	20,9	69,1
SlemmestadFE20070320S3	234	126	1,18	19,6	3280	59,2	53,7	<4	12,8	1,82	12,3	3,79	57,5	31,7	19,8	67,0
SlemmestadFE20070320S4	233	129	1,10	19,1	2990	61,2	54,8	<4	12,7	1,83	12,3	3,88	57,5	31,6	20,6	69,1
SlemmestadC20070320S1	319	87,4	1,13	14,0	232	25,6	41,3	<4	11,6	1,03	7,5	2,62	76,9	43,3	14,2	39,9
SlemmestadC20070321S1	320	89,5	1,05	13,6	243	30,3	41,3	<4	13,0	1,04	7,6	2,70	78,4	43,5	14,5	36,6
Taraldrud20070320S1	134	168	0,22	9,67	142	13,9	42,5	<4	17,7	1,50	8,2	1,73	72,7	38,0	5,83	18,1
Taraldrud20070321S1	140	169	0,24	9,97	149	14,9	41,8	<4	18,9	1,49	8,1	1,76	75,3	39,2	5,76	18,5

TOTAL KARBON/TOTAL SVOVEL/TOTAL ORGANISK KARBON
Geologisk materiale
ANALYSEKONTRAKTNR. 2007.0458



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BESTEMMELSE AV TOTAL KARBON(TC) / TOTAL SVOVEL(TS) / TOTAL ORGANISK KARBON (TOC) (LECO OVN)

INSTRUMENT TYPE : Leco SC-444

I) TOTAL KARBON (TC)

Nedre bestemmelses grense [% C]: **0,07**

Analyse usikkerhet

Måleområdet / %	Usikkerhet
0.07-0.5	± 0.07 %
>0.5	±15% rel.

II) TOTAL SVOVEL (TS)

Nedre bestemmelses grense [% S]: **0,01**

Analyse usikkerhet

Måleområdet / %	Usikkerhet
0.01-3.0	±30% rel.
>3.0	±20% rel.

III) TOTAL ORGANISK KARBON (TOC)

Nedre bestemmelses grense [% TOC]: **0,1**

Analyse usikkerhet

Måleområdet / %	Usikkerhet
0.1-3.0	±25% rel.
>3.0	±20% rel.

34

Det er her oppgitt usikkerhet med dekningsfaktor 2 (jfr. 2 standard avvik), noe som da korsponderer med et konfidensintervall på 95 %

PRESISJON : Det kjøres rutinemessig kontrollprøver, som føres i kontrolldiagram (X-diagram). Disse kan forevises om ønskelig.

ANTALL PRØVER: 12

ANMERKNINGER: Ingen

Rapporten må ikke gjengis i utdrag uten skriftlig godkjenning fra NGU-Lab.

Ferdig analysert | 14. jan. 2008 | Anne Nordtømme
Dato OPERATØR

Prøve id.	Svovel [%]	Karbon [%]	TOC [%]
1	2,72	6,52	5,81
2	2,80	6,51	5,76
3	2,68	5,96	5,32
4	2,77	5,93	5,28
5	3,88	9,67	9,11
6	3,79	9,62	9,04
7	3,74	9,61	8,98
8	3,82	9,52	8,93
9	2,53	4,65	4,31
10	2,27	4,58	4,33
11	1,47	10,81	10,06
12	1,48	10,72	9,96

OPPDRAAGSGIVER: Ola M Sæther**NGU PROSJEKT :****319000****ANTALL PRØVER:** 12**PRØVETYPE :** Humus**GLØDETAP 1 (Temperatur: 480°C i 20 timer)****Beregninger**

$$\Delta X_{G1} = B - C \Rightarrow Y_{G1} = \frac{B - C}{A} \cdot 100 = \frac{\Delta X_{G1}}{A} \cdot 100$$

A: Innvekt prøve B: Vekt av prøve+digel C: Vekt av digel+prøve etter varming ΔX_{G1} : Vekttap (Glødetap) Y_{G1} : %Vekttap (%Glødetap)

96

Analyseusikkerhet : 8 % rel. (1 σ -nivå)**ANMERKNINGER:** 480°C i 20 timer**Rapporten må ikke gjengis i utdrag uten skriftlig godkjenning fra NGU-Lab.**

Ferdig analysert	28. feb. 2008	Frank Berge
Dato		OPERATØR

Bestemmelse av Glødetap ved 480°C
Geologisk materiale
ANALYSEKONTRAKTNR. 2007.0458



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Prøve navn	Vekt Skål	Vekt prøve(A)	Vekt digel+prøve(B)	Vekt etter 480°C(C)	Gl.tap
	[g]	[g]	[g]	[g]	[%]
1	2,1754	5,043	7,2184	6,6922	10,4
2	2,1835	5,040	7,2231	6,6916	10,5
3	2,1883	5,070	7,2583	6,7683	9,7
4	2,1785	5,040	7,2185	6,7058	10,2
5	2,1652	5,101	7,2658	6,5834	13,4
6	2,1674	5,065	7,2327	6,5522	13,4
7	2,1762	5,084	7,2599	6,5813	13,3
8	2,1817	5,094	7,2758	6,5946	13,4
9	2,1781	5,052	7,2302	6,8092	8,3
10	2,1697	5,105	7,2745	6,8537	8,2
11	2,1700	5,116	7,2862	6,5029	15,3
12	2,1709	5,055	7,2255	6,4514	15,3