



REPORT

# SP13 Under Oslo

WP1 – NATURALLY MIXED FRACTIONS OF  
BLACK SHALE AND RHOMB PORPHYRY

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## Project

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## Summary

When taking out potentially acid-producing black shale in construction projects, rock masses are often unintentionally mixed with masses of other rocks. The acid-producing potential of such mixed masses is uncertain, and as a result, large amounts of potentially clean rocks often end up in disposal sites. In WP1 of the Under Oslo project, the leaching from mixed masses with different fractions of rhomb porphyry with black shale has been investigated. Both dilution and additional buffering effects of the rhomb porphyry are assumed to contribute to the desired effects.

To investigate this hypothesis, both small scale column experiments, as well as larger scale outdoor container experiments have been conducted over the course of two years. Different amounts of black shale have been mixed with rhomb porphyry in both experiments. Additionally, for the column experiments, the effect of grain size has been investigated. While the outdoor container experiments have been exposed to ambient conditions, with varying precipitation, humidity and temperature, the column experiments are executed under more ideal laboratory conditions, with regular watering and more stable atmospheric conditions. The columns and most containers are free drainage systems, where the system is unsaturated with water and with dry periods. Some containers are closed and water-saturated; however, these are not covered in this report.

This document describes the results two years into the experiments. Additionally, one specific case related to construction of is described, where mixed fractions of syenite and black shale were analysed to evaluate an appropriate threshold for when rock masses should be deposited in appropriate landfills.

Key findings so far:

- The column containing 100 % alum shale experiences a significant decrease in pH after about one year. The pH drops below 3 after 1.5 years and has stabilized at pH 2.5 after ca. 2 years. The drop in pH results in substantial amounts of metal leaching: After two years, analyses show concentrations of 0.1-0.2 g/L for Ni and Zn, and 0.5-0.6 mg/L for Cd and Co (filtered water samples).
- Two years into the column experiments, lower fractions of alum shale (3%, 7%, 10% and 20%) do not produce eluates with a notable reduction in pH, regardless of grain size (0.22 mm, 22-32 mm and 32-50 mm). Continued monitoring of the columns is therefore desirable to see how the leaching and pH are affected in an even longer timeline.
- Due to a lower surface area available for reaction in the higher grain size columns, the buffer capacity depletes quicker in these columns compared to the corresponding 0-22 mm column. Consequently, there is an initial lowering of pH in these columns, from >pH 9 to values typically between pH 5-6 within the first year, and higher concentrations of metals in the leachates. However, due to the lower surface area, pH is manageable and not expected to decrease

significantly. Although the leaching of metals is considerably lower in the columns with 10 % alum shale and 22-32 and 32-50 mm grain size compared to the 0-22 mm 100% alum shale column, the values are still well above guideline values for fresh water and could be problematic in certain cases/areas.

- The processes seem to occur much slower in the containers compared to the columns. This is likely due to different exposure to water and temperature, as well as the different grain size distributions.

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# 1 Introduction

Different types of black shales from the Cambrian and Ordovician succession are present in the southern parts of Norway and Sweden, as well as parts of Denmark (Andersson et al. 1985; Martinsson 1974). The black shales were formed by sedimentation in anaerobic environments and contain varying amounts of sulphide minerals as pyrite and pyrrhotite, carbonates and remnants of organic matter. They are often enriched in heavy metals such as cadmium, copper, uranium, vanadium, nickel and zinc.

When a black shale is disturbed by construction work, exposure to oxygen and water leads to oxidation of the sulphide minerals to form sulphuric acid. Hence, black shale can pose a considerable challenge for construction work in these areas. In addition to acid rock drainage, which can pose a risk to both the environment and to concrete and steel constructions, black shales may be an additional environmental risk due to leaching of heavy metals which are mobilized by the acidic conditions. Consequently, the current practice for handling excavation residues containing black shale is to treat the entire quantity of masses as acid-forming masses and to deposit them in landfills, occupying valuable special landfill capacity. In cases of doubt, where it is not possible to separate the masses and the fraction of acid-forming rocks appears to be low, a special assessment of the masses can be made.

There are several types of black shale, where sulphide and heavy metal content differs. Of the black shales, alum shale is considered to have the highest risk of impact to the environment, given the formation's high content of sulphuric minerals and high concentrations of heavy metals. The knowledge of the varying environmental impact associated with different types of black shale has increased. The characteristics within a formation can have large local differences, and it is therefore important to perform testing and risk assessment of the black shale found in each project, which today is the current practice in Norway.

In the areas in connection to the Oslo Graben, permian intrusions of magmatic rocks are often present as dikes and sills in the black shale. In certain areas of Oslo, the magmatic intrusion dominates the rock mass, with only sporadic and thin horizons (0,1 – 2 m) of black shale in between. Consequently, sorting of rock masses can be difficult, and the rubble resulting from blasting or drilling may contain only a small fraction of black shales. Due to uncertainty related to such mixed masses, vast amounts of rocky masses end up in landfills, even though the masses contain only a limited amount of black shale.

The overall hypothesis in this project is that a significant share of the masses deposited in landfills could have been used for other purposes, or treated in deposits for inert waste, without posing any risk. It is hypothesized that the adverse effects of acid drainage and heavy metal leaching will be minimal when the percentage of alum shale in mixed masses is below a certain level. This level will likely depend on rock quality, buffering potential of surrounding masses as well as grain size. Dilution of acid producing masses or other contaminated masses is however not allowed, and this report is aimed at naturally mixed masses which are not possible to separate.

To test these hypothesis, two experiments are conducted: (1) Column experiments with controlled laboratory conditions, and (2) container experiments, where masses are exposed to outdoor ambient weather conditions. At the time of writing, these experiments have been running for a little more than two years. This report summarizes and discusses the results.

## 1.1 Background

Tunnel excavation using drill and blast techniques makes sorting of rock masses difficult (Figure 1 and Figure 2). A vast large of rocky masses end up in landfills, even though the masses contain only a limited amount of black shale. The rock masses may otherwise be of high quality for construction purposes. The principal reason for this is lack of suitable tools to evaluate the actual risk to the environment or to a construction. A significant share of the masses deposited in landfills could likely have been used for other purposes, without posing any risk.

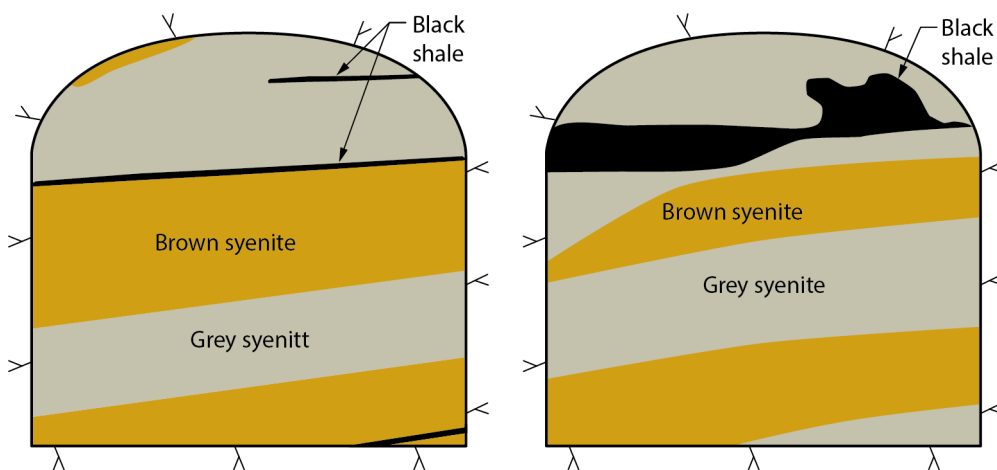


Figure 1 Example of engineering mapping of the temporary excavation face in a tunnel, black shale is illustrated with black, as thin horizons in mainly grey and brown syenite.



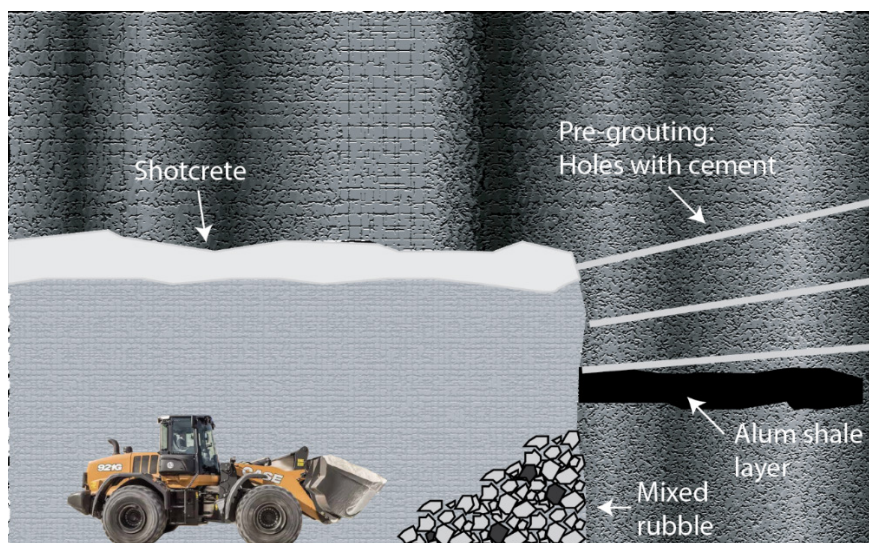


Figure 2 A longitudinal section of a tunnel excavation illustrating holes for pre grouting, layers of black shale (here alum shale) and a mixing of the excavation residues after blasting.

Furthermore, the amount of black shale may constitute only a small share of the total rock mass, and the acid drainage from the black shale could consequently be buffered by other types of rocks from the same excavation residue, depending on the properties of these rocks.

More reuse of materials containing only a small share of black shale is highly desirable, both from an economic and a sustainability perspective. When depositing rock masses in landfills, the masses must usually be transported over large distances to be deposited, which increases the CO<sub>2</sub> footprint of a project, as well as the cost. The capacity of waste fills with permission for receiving black shale is limited in Norway.

The aim of this project was initially to create a predictive model that can be used to evaluate the risks associated with a given composition of rock masses containing black shale. Exposure of water and oxygen to such masses, initiates complex processes that depends on a broad range of variables. Key variables include grain size distribution, the buffering potential of the masses, and their ability to form sulphuric acid (i.e. type and amount of black shale).

The grain size distribution is important because it determines the amount of available reactive sites (i.e. surface area), as well as the permeability of the masses. A low permeability means that water will both filter and evaporate slowly from the masses. This makes water available to the reactions over a longer period of time, giving the reaction time to proceed despite their relatively slow kinetics. However, if masses are saturated with water over a longer period the oxygen is consumed, and thereby prevents the reaction from occurring. The experiments were therefore designed so that water runs

through the rocky masses to ensure oxygen availability throughout the experimental time.

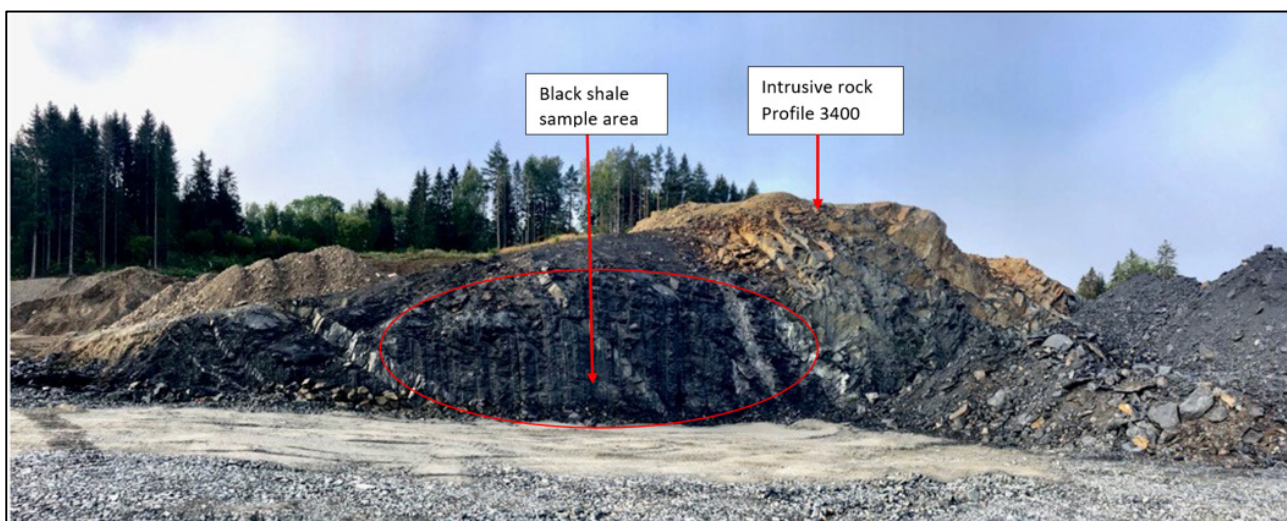
Oxidation will also occur due to air humidity, and in dry periods this may cause a certain build-up of sulphate on the surface of the alum shale. Measurements of oxidation rates in air (95% humidity) is in the order of  $10^{-7}$  -  $10^{-8}$  mol/m<sup>2</sup>s<sup>-1</sup> (Jerz and Rimstidt 2004). At lower humidity levels the process is likely significantly slower (Borek 1993). In our experiments, the black shale is dry only for limited periods of time, if at all for the finer grain size fractions. However, the possibility of pH reduction after dry periods is a consideration for long-term storage/usage of such masses. Regardless of air humidity, liquid water is required to remove sulfate from the surface, and the degradation time is dependent on liquid water. Oxidation by air humidity or residual moisture will practically stop after the exposed surface has reacted, since diffusion of oxygen through a film of ferrous sulfate occurs at a very slow rate (Jerz and Rimstidt 2004). Therefore, oxidation by air humidity exposure will also depend on the available surface area and the grain size distribution.

With this work, we aim to increase the understanding of risks and processes around acid-producing properties of naturally mixed masses with minor fractions of black shale.

## 2 Experimental

### 2.1 Materials

During construction of new E16 between Eggemoen and Olum, about 7-9 tons of alum shale (horizon 2 and 3a) was taken from an alum shale blast performed 17.09.2020 in Kleggerudkrysset (see Figure 3 and Figure 4) and transported to NGI 21-09-2020.



*Figure 3 Black shale sampling area at Kleggerud for alum shale used in the container experiments set up at NGI. Photo: Skanska AS.*

Rhomb porphyry from Bjønndalen Bruk in Nittedal (Feiring AS) was used to mix with the alum shale in some of the treatments. The rhomb porphyry is considered stable/unreactive and is used in construction projects. It was chosen in this context as it has a limited neutralizing potential and is similar to syenite (mænaitt) which is often found as intrusive rock in alum shale. Thus, there are construction projects where the separation of alum shale and syenite is not practically possible and this would constitute naturally mixed masses, as for example seen in Figure 4 where the intrusive rock (brown, syenite) may be mixed with the alum shale (green) when the masses are taken out.

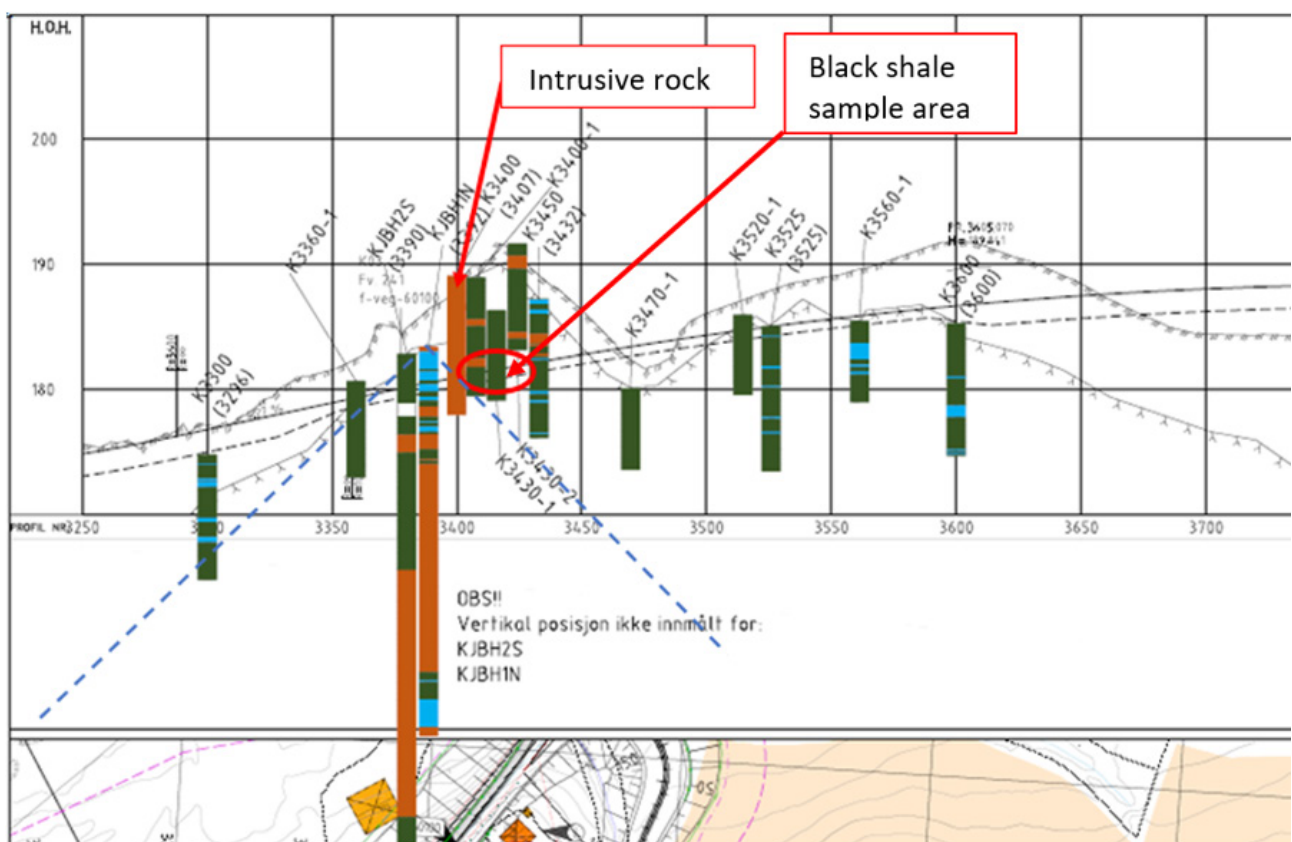
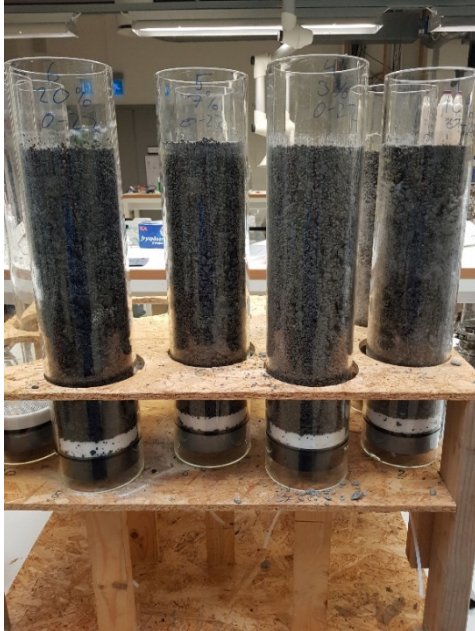


Figure 4 Cross section of tunnel at E16 Kleggerud. Placement and name of boreholes are indicated. The intrusive rock is syenite. (Statens Vegvesen, 2019).



## 2.2 Column experiments



*Figure 5. The columns have an open top, where water is added. A wide meshed filter was placed at the bottom to avoid attenuation of the water flow due to filtering. The eluates are collected in bottles and analysed. This set-up corresponds to free drainage, i.e. an unsaturated system.*

Table 1 summarizes the composition of the columns for the experiment. The alum shale and rhomb porphyry fractions given in Table 1 were homogeneously mixed before transferring to the columns. The masses were packed by dropping a weight at a similar height onto the masses three times.

*Table 1. Columns used in the experiment.*

Column #	Grain size distribution (mm)	% alum shale w/w	% rhomb porphyry w/w	height of masses in the column (cm)*
1	0-22	10.00	90	34.0
2	22-32	10.74	89.26	36.5
3	32-50	10.66	89.34	41.5
4	0-22	3.25	96.75	33.5
5	0-22	7.04	92.96	34.3
6	0-22	20.08	79.92	34.0
8	0-22	100	0	34.6
9	0-22	0.00	100	31.5

The column experiments were designed to investigate the effect of two variables; the grain size distribution and the percentage of alum shale (Table 1).

500 mL of milli-Q was added to each column for each round of watering. This is a free drainage system, where water is added at the top of the column and evacuates at the

bottom. The watering has occurred irregularly, in periods daily or weekly, occasionally with longer dry periods.

The following parameters were measured in the water after filtering through the columns, after every time the columns were watered:

- ↗ pH
- ↗ redox potential
- ↗ Conductivity

Eluates were analysed for metals, alkalinity, main cations, and main anions after 40, 365 and 730 days.

The alum shale was collected at Kleggerud. More details regarding the columns, as well as alum shale and rhomb porphyry characterization, can be found in NGI, 2020.

## 2.3 Container experiments



*Figure 6 Overview of containers for leaching experiments. Five containers "inherited" from the Norwegian Public Road Authorities are also in the photo.*

Figure 6 shows an overview photo of the containers. Some containers are closed and saturated with either fresh or salty water, while most containers are open and exposed to rain. The latter containers represent freely draining and unsaturated systems, similar to the column experiments.

The container experiment has been running since October 2020, similar to the column experiment. The containers discussed in this report contain the same alum shale and rhomb porphyry as that used for the column experiments

*Table 2. Set-up of relevant containers. AS refers to alum shale, and RP refers to rhomb porphyry.*

Name	Content	Replicates	Alum shale (% V/V)
VAS	200 L AS	2	100
B5	10 L AS 190 L RP	2	5
B10	20 L AS 180 L RP	2	10
B20	40 L AS 160 L RP	2	20
RM	200 L RP	2	0

An important difference between the container and column experiments, is that the container experiments use unsorted blasted rocks. This will include a broad grain size distribution, from 0 – 250 mm.

The eluates from the containers are collected and analysed (pH, conductivity and redox) monthly.

## 2.4 Characterization

### 2.4.1 Element analysis

The analyses show that the alum shale contains 18 900 ppm sulphur and 5.9 % organic carbon. Further, the content of CaO is 4.4 and 1.2 % for the black shale and rhomb porphyry, respectively.

*Table 3. Analysis of the solid samples by the external laboratory ALS, using mainly ICP-SFMS and XRF.*

<b>ELEMENT</b>		<b>Rhomb porphyry</b>	<b>Black shale</b>
As (Arsen)	mg/kg TS	<3	46.4
Ba (Barium)	mg/kg TS	1000	733
Be (Beryllium)	mg/kg TS	6.5	3.55
Cd (Kadmium)	mg/kg TS	0.0803	6.96
Co (Kobolt)	mg/kg TS	7.17	19.3
Cr (Krom)	mg/kg TS	9.49	89.4
Cu (Kopper)	mg/kg TS	6.05	93.5
Hg (Kvikksølv)	mg/kg TS	<0.02	0.0767
Mo (Molybden)	mg/kg TS	3.06	102
Nb (Niob)	mg/kg TS	170	18.4
Ni (Nikkel)	mg/kg TS	3.81	238
Pb (Bly)	mg/kg TS	5.97	31.1
S (Svovel)	mg/kg TS	567	18900
Sc (Scandium)	mg/kg TS	7.92	16.6
Sn (Tinn)	mg/kg TS	4.35	3.65
Sr (Strontium)	mg/kg TS	929	93.4
V (Vanadium)	mg/kg TS	63.7	1860
W (Wolfram)	mg/kg TS	4.88	2.54
Y (Yttrium)	mg/kg TS	43.9	39.3
Zn (Sink)	mg/kg TS	34.7	379
Zr (Zirkonium)	mg/kg TS	798	158
Th (Thorium)	mg/kg TS	27.6	16.7
U (Uran)	mg/kg TS	6.62	69.4
Tørrstoff (E)	%	99.9	99.7
TOC	% TS	0.24	5.92
TIC	% TS	0.625	0.269

*Table 4. Analysis of the solid samples by the external laboratory ALS, using mainly ICP-SFMS and XRF.*

<b>ELEMENT</b>		<b>Rhomb porphyry</b>	<b>Black shale</b>
SiO <sub>2</sub>	% TS	50.5	53.3
Al <sub>2</sub> O <sub>3</sub>	% TS	16.3	16
Kalsiumoksid (CaO)	% TS	4.44	1.15
Fe <sub>2</sub> O <sub>3</sub>	% TS	6.6	5.23
K <sub>2</sub> O	% TS	4.56	5.14
MgO	% TS	1.45	1.57
MnO	% TS	0.0991	0.0268
Na <sub>2</sub> O	% TS	4.87	0.476
P <sub>2</sub> O <sub>5</sub>	% TS	0.654	0.185
TiO <sub>2</sub>	% TS	1.51	0.936
Glødetap (LOI)	% TS	1.1	9.1

### 1.1.1 XRD

The XRD results show that the black shale consists of 4.5% pyrite and 1.2% calcite (Table 5). In comparison, according to the ALS analysis (ICP-SFMS) there is 18 900 ppm of sulphur, which would be equivalent of 7% pyrite assuming all sulphur in the form of FeS<sub>2</sub>. Further, the XRF analysis done by ALS showed 1.15% CaO.

The XRD lab reported that the amorphous material was difficult to estimate for the black shale and recommended using measured organic matter (from LOI) to estimate amorphous material. The results for black shale in Table 5 are therefore corrected the 9.1 % OM.

As expected, the rhomb porphyry contains no pyrite, and twice the amount of calcite. Columns with larger amounts of rhomb porphyry may therefore be expected to have a higher buffering capacity.

Hence, despite the high potential for acid drainage, there is a certain buffer capacity which will delay the reduction in pH.



*Table 5. XRD results for the black shale from Kleggerud and rhomb porphyry from Nittedal verk, used for the column experiments. Results are in % of total weight and corrected for 9.1%OM.*

<b>%</b>	<b>Rhomb porphyry</b>	<b>Black shale</b>
Smectite	0.4	ND
Illite/Smectite	ND	ND
Illite+Mica	1.8	44.8
Kaolinite	ND	0.1
Chlorite	8.4	TR
Quartz	TR	29.9
K Feldspar	17.3	6.2
Plagioclase	45.8	4.3
Calcite	2.8	1.2
Dolomite	ND	TR
Pyrite	ND	4.5
Amorphous	23.4	9.1*

TR – trace (< 0.5 %), ND – not detected.

\* Amorphous matter for black shale was estimated from LOI.

## 1.1.2 XRF

The XRF conducted at NGI deviates somewhat from that conducted by ALS (alunskiferpakka). For example, ALS reports around 50% SiO<sub>2</sub> for both materials, whereas the results presented in Table 6 gives values of 27% and 32%. The values for CaO are more similar, with 4.4 % and 1.2 % (ALS) versus 5.5 % and 1.7 % (NGI) for rhomb porphyry and black shale, respectively.

Table 6. Results of XRF measurements (average of three measurements). Only detected elements are shown.

Element	Rhomb Porphyry mg/kg	Error	Black shale mg/kg	Error
V	< LOD	84	1931	69
Cr	74	41	83	31
Ni	< LOD	30	223	20
Cu	20	15	83	10
Zn	41	7	476	12
Pb	7	4	34	4
Sr	967	7	118	2
Y	51	2	45	2
Zr	941	8	162	2
Nb	109	3	16	1
Th	30	4	19	3
U	< LOD	7	78	5
Ba	533	47	523	42
S	2449	163	13398	156
As	5	6	45	5
Mo	8	2	105	2
Si	128936	1006	148708	940
Ca	40176	906	12433	546
Al	29081	835	39388	816
Fe	45157	344	29188	227
Mn	906	69	140	46
K	27323	452	39953	485
Ti	5500	127	5171	119
P	9398	2241	3645	192
Bal	707167	1247	699919	1094
CaO	56247	0	17406	0
Fe <sub>2</sub> O <sub>3</sub>	64575	0	41738	0
MnO	1169	0	180	0
K <sub>2</sub> O	33061	0	48344	0
P <sub>2</sub> O <sub>5</sub>	21522	0	8346	0
SiO <sub>2</sub>	275923	0	318235	0
Al <sub>2</sub> O <sub>3</sub>	54964	0	74444	0
MgO	1510	0	6543	0
Cd	< LOD	12	12	8
Mg	< LOD	4587	3942	2075
W	56	37	56	31
Sb	< LOD	23	26	11
Rb	63	2	70	2
Cl	54	25	< LOD	35

### 3 Results 2020-2022

#### 3.1 Column experiments

##### 3.1.1 pH and conductivity

The data presented in this report comprises the most interesting findings. For the full analysis reports and recorded values, see Appendix A and B.

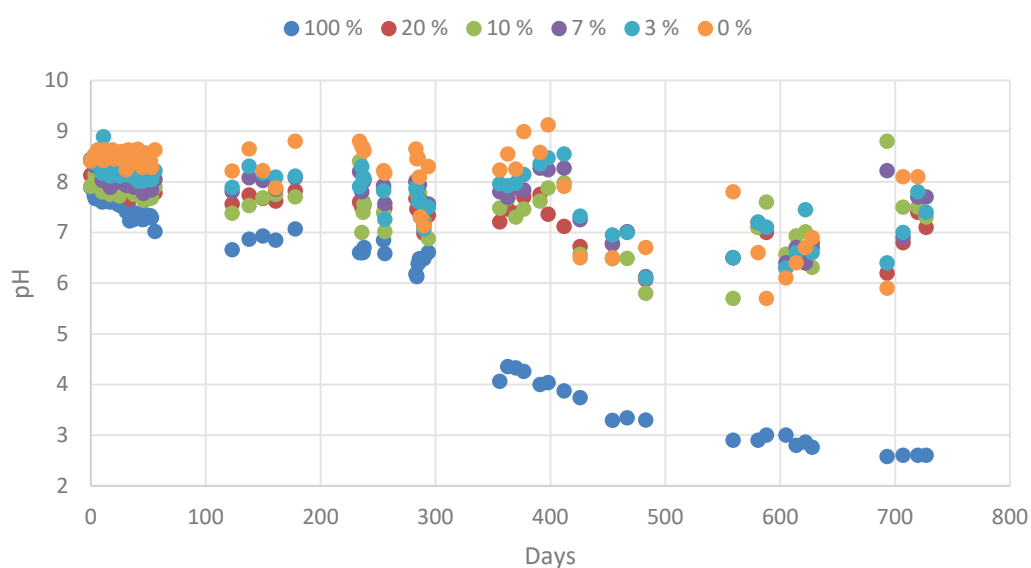


Figure 7. The effect of increasing percentage of alum shale (0-22 mm grain size) on pH.

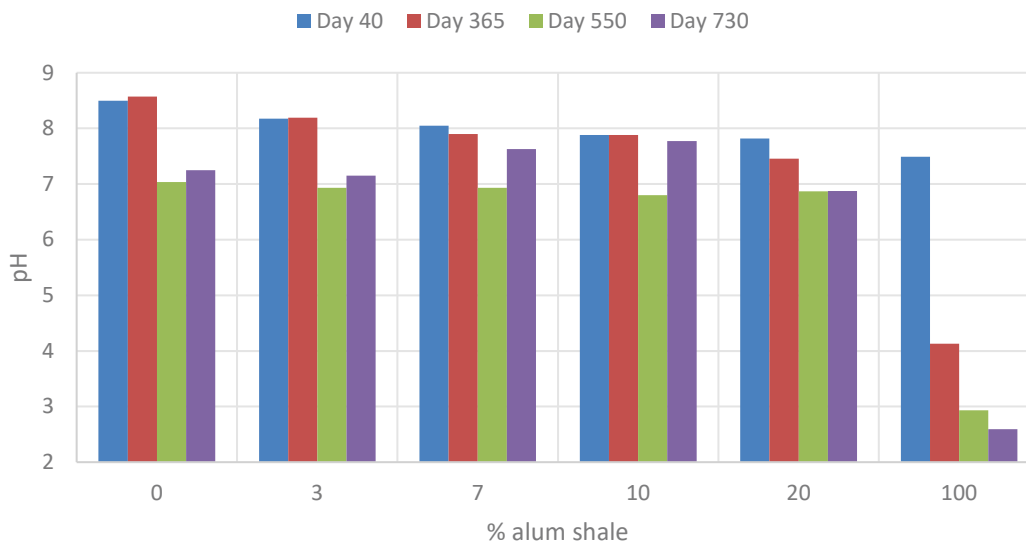


Figure 8. The effect of increasing percentage of alum shale (0-22 mm grain size) on pH, with a comparison of day 40, 365, 550 and 730 eluates. The values are averages of five measurements around day 40, 365, 550 and 730 to account for the variations.

Considering the data in Figures 7 and 8 there does not seem to be a significant difference in pH after 730 days for the 0-20% alum shale columns (0-22 mm), However, after 365 days, the pH in the 100% alum shale column has decreased markedly, to around pH 4, and it decreased further to 2.5 by 730 days.

In the other columns, the pH is not significantly different from day 40 to day 365, although a slight decrease in pH has occurred for the 20% alum shale column. The lack of a pH drop is likely due to the additional buffering effect of the rhomb porphyry. XRD analysis shows that the rhomb porphyry consists of 2.8 % calcite, compared to 1.2 % in the alum shale (section 2.4).

It is interesting to note that the reduction in pH has started earlier for the larger grain size fractions (Figure 9 and Figure 10). This can be explained by the smaller surface area available for reaction, causing the buffer potential to deplete quicker with larger grain size fractions. However, pH values are acceptable at the time of writing, and may not decrease much further. Continued monitoring is necessary. Note that pH in clean water in equilibrium with the atmosphere has a pH of 5.6. Thus, the measured pH in the 22-32 and 32-50 mm fraction does not have to reflect acid production. This is also reflected in the conductivity measurements (see Figure 13) showing that there is less chemical activity/reactions due to the smaller surface area.

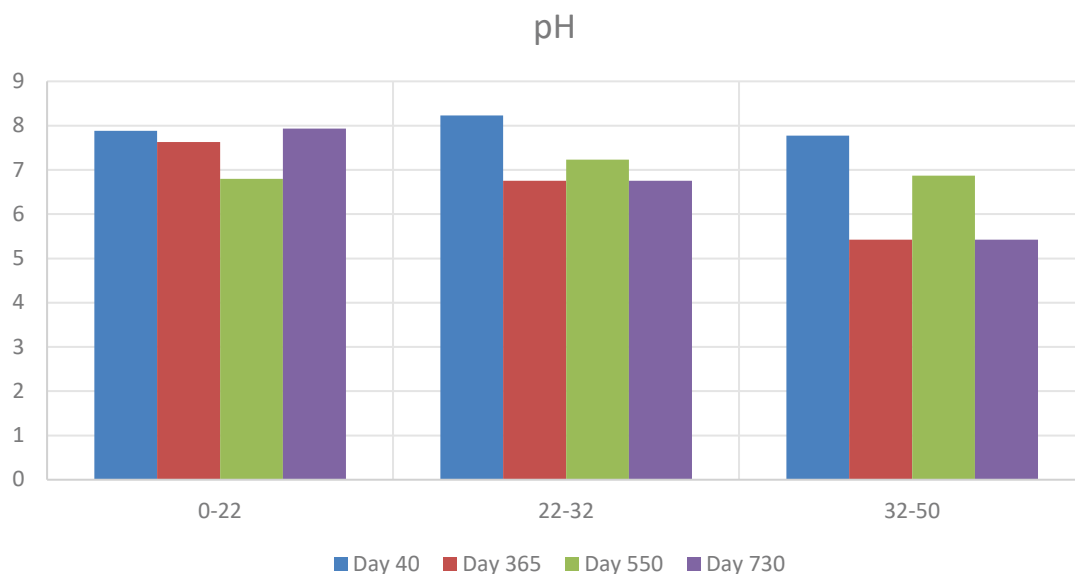


Figure 9. The effect of grain size distribution on pH (10 % alum shale), with a comparison of measurements on eluates from day 40, 365 and 730. The values are averages of five measurements around day 40 and day 365 to account for the variations.

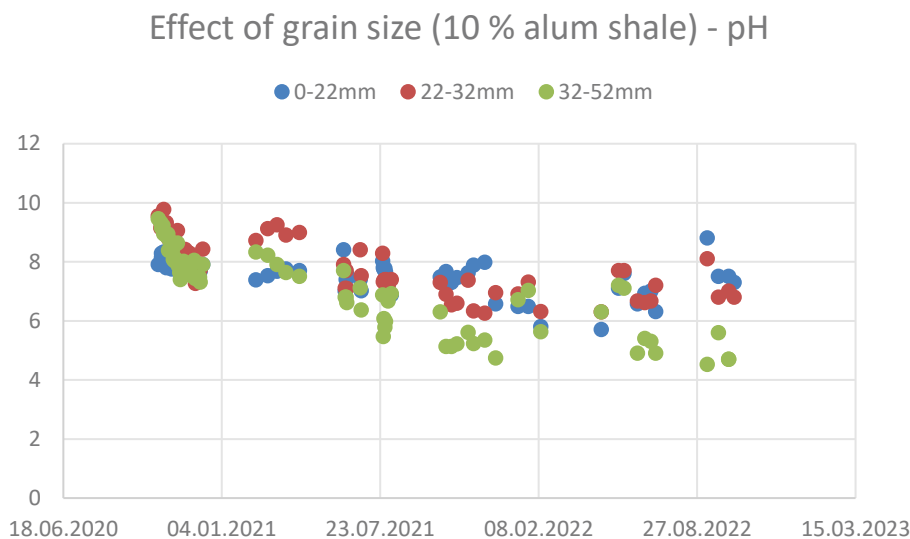


Figure 10. The effect of grain size distribution on pH (10 % alum shale).

**Conductivity:**

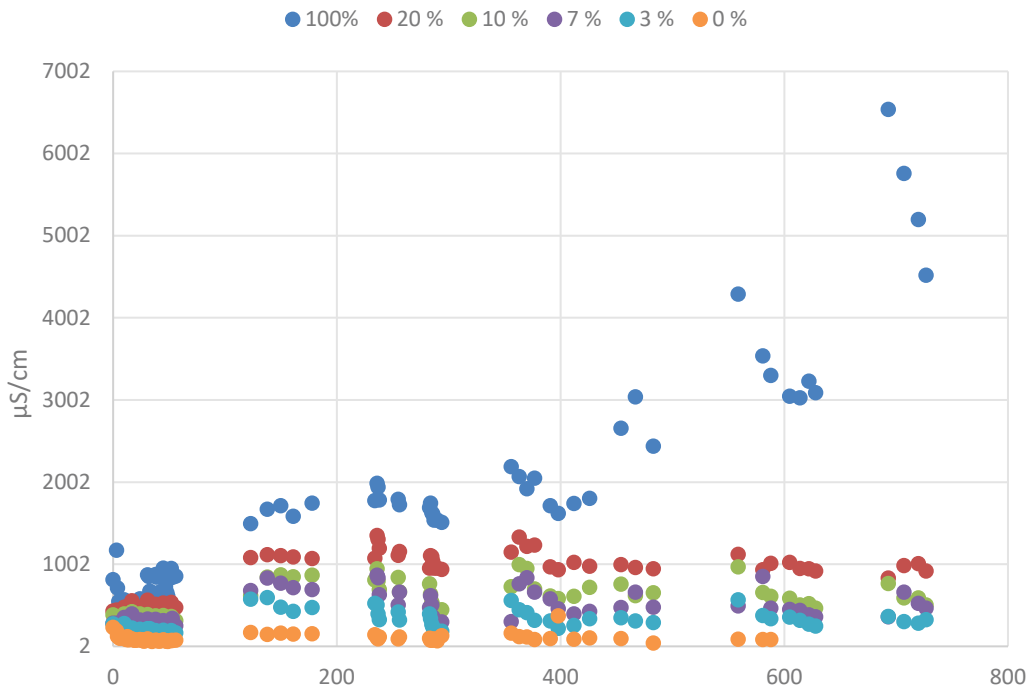


Figure 11. The effect of increasing percentage of alum shale (0-22 mm grain size) on conductivity of leachate.

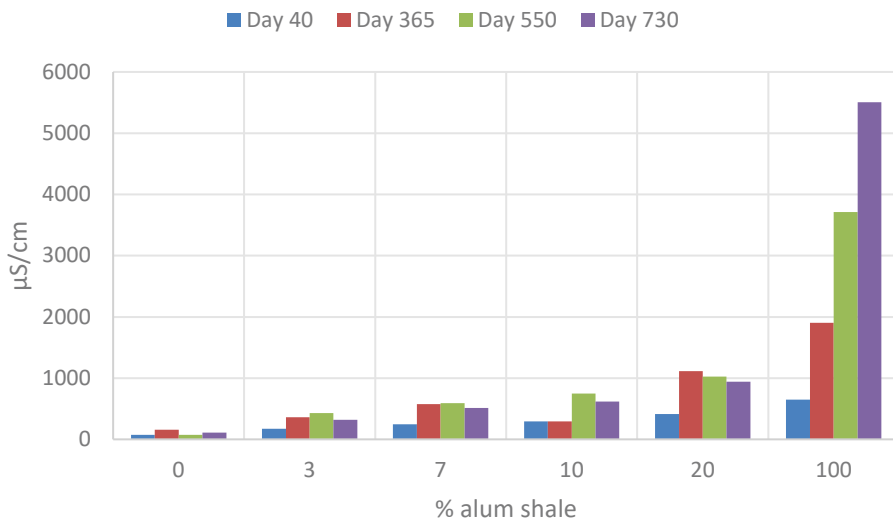
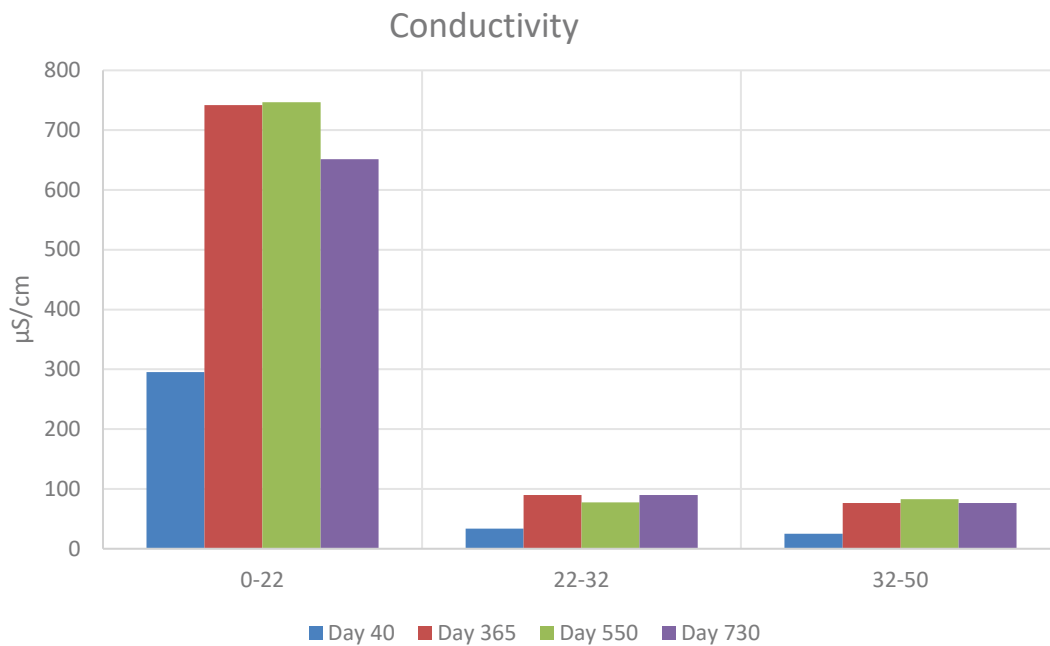


Figure 12. The effect of increasing percentage of alum shale (0-22 mm grain size) on conductivity of leachate, with a comparison of day 40, 365, 550 and 730 eluates. The values are averages of five measurements around day 40, 365 and 730 to account for the variations.



*Figure 13. The effect of grain size distribution on conductivity (10 % alum shale), with a comparison of measurements on eluates from day 40, 365 and 730. \*The values are averages of five measurements around day 40, 365 and 730 to account for the variations.*

The conductivity of eluates correlates well with the measured leaching of sulphate (see section 3.1.5), and the conductivity likely to a large extent reflects the levels of sulphide/sulphate in the eluates.

### 3.1.2 Alkalinity

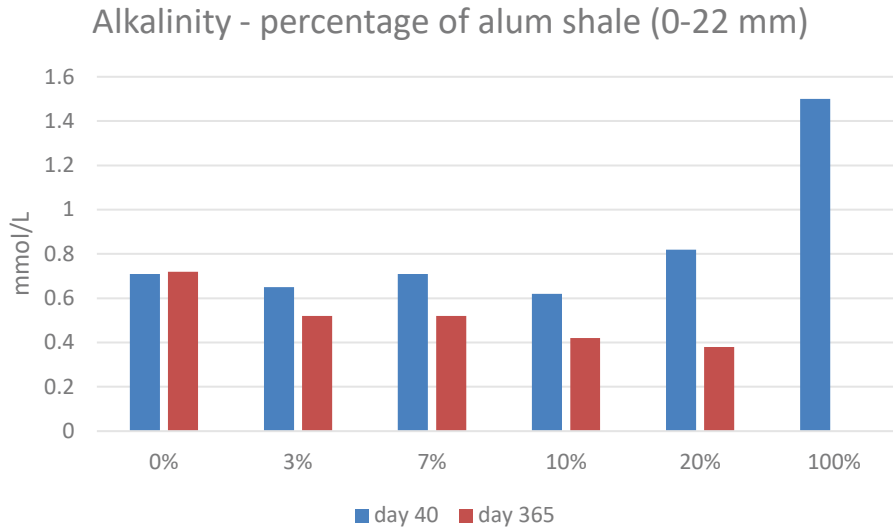


Figure 14. Comparison of alkalinity in eluates with 0-22 mm masses and varying percentage of alum shale.

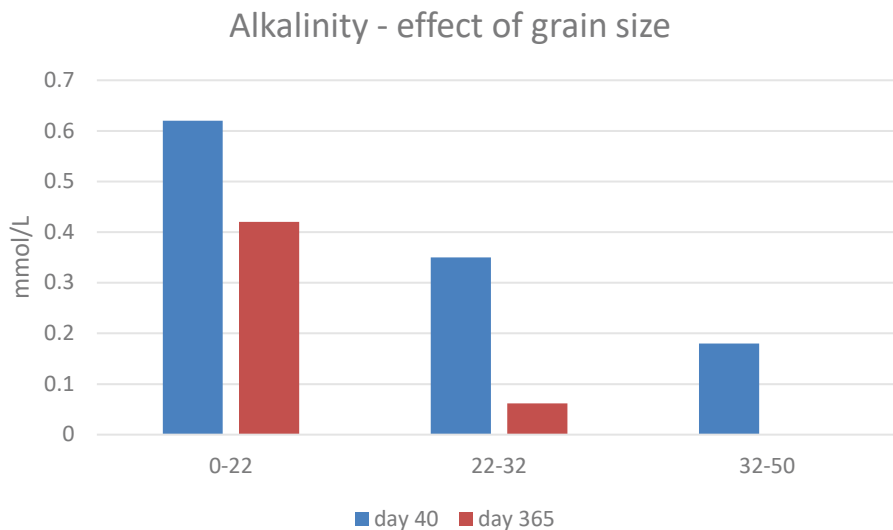


Figure 15. Comparison of alkalinity in eluates with 10 % alum shale and grain size distributions.

The alum shale and rhomb porphyry contain 1.2 and 2.8 % calcite, respectively (section 2.4). In addition, the alum shale contains of 4.5 % pyrite, which oxidises and produces acid drainage. The calcite in the alum shale might be more readily available than the



calcite in the rhomb porphyry, as the 40 week measurement is the highest in the column with 100 % alum shale. The calcite will initially neutralize the produced acid. It is interesting to note that the drop in alkalinity from day 40 to day 365 increases with increasing fraction of alum shale (Figure 14). In fact, in the eluates from the column with 100 % alum shale, the alkalinity is zero. Unfortunately, there is no alkalinity data for day 730 due to a mistake during the ordering process.

Figure 15 shows that the relative drop in alkalinity is independent of grain size. Although the eluate alkalinity in the 22-32 and 32-50 mm columns (10 % alum shale) initially is lower than in the 0-22 mm column, the drop in alkalinity is approximately the same. This is also reflected in the pH measurements, where the eluate pH is lower for the larger grain sizes. The data shown in Figure 15 shows that this is due to the buffering capacity being exhausted more rapidly with larger grain sizes. The important question is which grain size would have a larger chance of developing a pH drop and ARD, as that is when leaching of metals really increases and the low pH in itself is a problem for environment and constructions. As shown below, the dilution caused by the smaller surface area of the 22-32 and 32-50 mm columns makes the leaching manageable. It is possible that if the buffering capacity of the 0-22 mm column (10 % alum shale) is exhausted, the leaching would exceed that of the 22-32 and 32-50 mm columns. This is reflected by the leaching data of the 100 % alum shale, 0-22 mm column shown in the subsequent subsections.

### 3.1.3 Heavy metal leaching

This chapter focuses on the metals with greatest abundance in the leachates; Zn, Ni, Co and Cd. The environmental guideline values for freshwater are given in Table 7.

*Table 7. Guideline values for fresh water (M-608), in µg/L. There are no values for Co in the guidelines. The values for Cd depend on CaCO<sub>3</sub> levels in the water, which is conservatively assumed to be low.*

	Klasse I	Klasse II	Klasse III	Klasse IV	Klasse V
Ni	0-0.5	0.5-4	4-34	34-67	>67
Zn	0-1.5	1-5-11		11-60	>60
Cd	0-0.003	0.003-0.08	0.08-0.45	0.45-4.5	>4.5

Figure 16 shows the values of these heavy metals measured in eluates after 40 and 365 days. As above, two plots are given for each metal, illustrating the effect of alum shale percentage and grain size.

For the 0-22 mm masses, the levels of Ni and Zn in the eluates increase to values above Klasse IV-V only at alum shale percentages of 20% or higher. If this runoff pollutes a receptor, considerable dilution occurs (depending on the volume/water flow of the

receptor), and the 20% alum shale sample will likely not be problematic in terms of Zn and Ni, even after 730 days.

However, in the 100% alum shale column, the levels of Zn and Ni are 'off-the-charts' after 730 days, with 203 000 and 108 000 µg/l, respectively. At the time of sampling, the pH in this column had dropped markedly to around 2.5, and an increase in leaching is expected relative to the other columns with less alum shale. This rate of leaching could potentially be highly problematic for a small receptor.

Considering the guideline values, the levels of Cd are equally problematic for the 100% alum shale eluates, with the level of Cd in the eluates being 1150 times higher than the limit for 'Klasse V' after 730 days. Cd and Co seem to be mobilized to a much greater extent compared to Zn and Ni. Despite a lower presence in the alum shale (Table 8), the leaching is in the same order of magnitude.

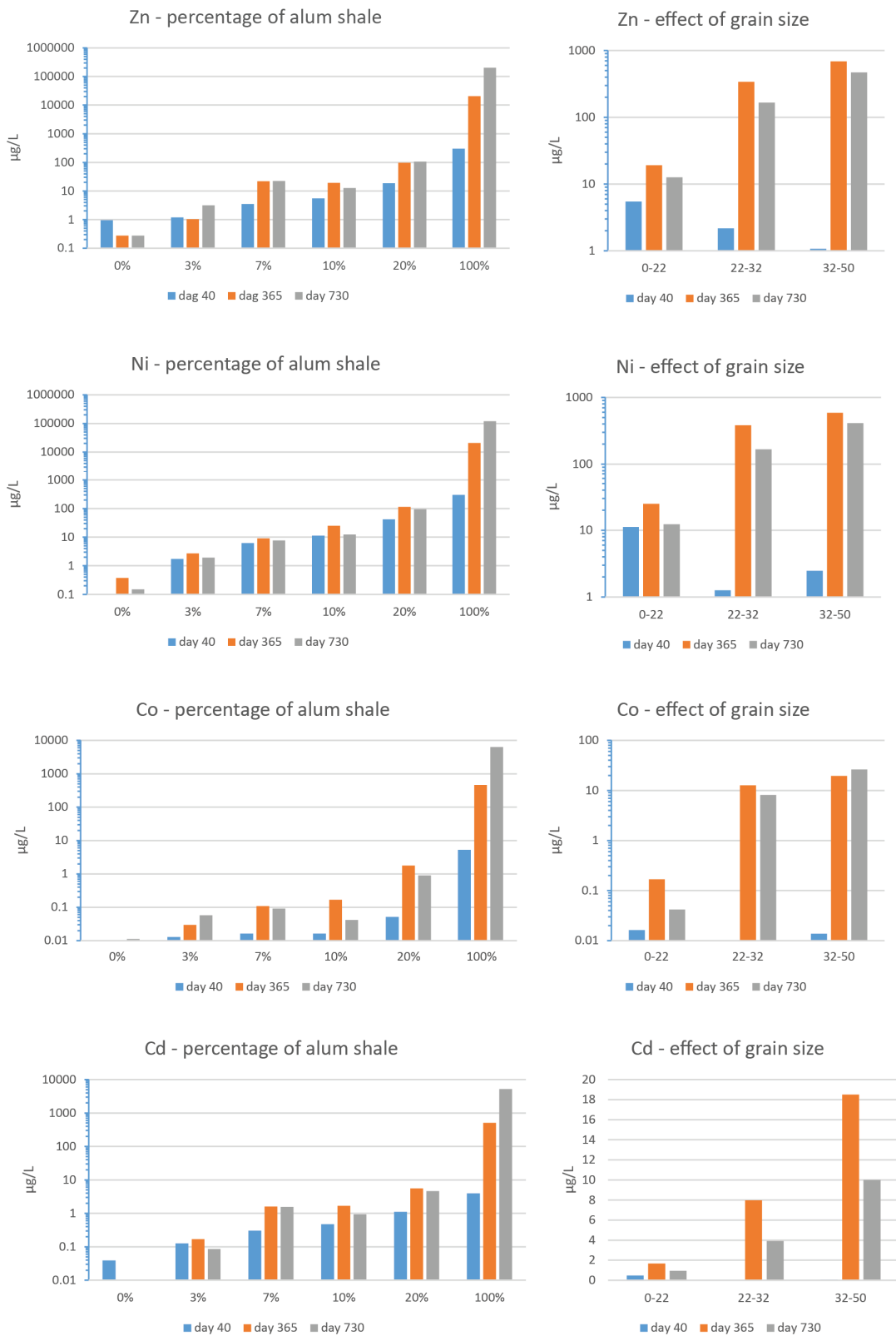


Figure 16. Heavy metal measured in eluates from columns with varying amounts of alum shale and rhomb porphyry. Note that most of the y-axes are logarithmic.

The effect of lowered pH is further apparent in the columns with varying grain size. In these samples, the pH is lower for increasing grains size, due to the buffering capacity being depleted earlier when the available surface area for reaction is smaller. The pH is assumed to reflect the pH of water in equilibrium with CO<sub>2</sub> in the air, which is around 5.6. The low surface area also results in a lower potential for leaching, with consequent greater dilution. Despite this, the levels of Zn and Ni in the eluates from the 22-32 and 32-50 mm grain size columns are in the order of 10 times the limits for fresh water after 365 days, and 1-2 orders of magnitude higher than in the 0-22 mm column. As higher reactivity is expected with lower grain size this is unexpected, but likely an effect of pH. A comparison of leaching from the 22-32 and 32-50 columns after one and two years, however, indicates that the leaching has levelled off. In fact, for Zn and Ni there is a 51 % and 32 % decrease in concentration, respectively, from year one to year two. Similarly, there is a 46% and 51% reduction for Cd for the 22-32 and 32-50 columns, respectively, from year one to year two. While there number of data points is a bit low , there is a trend of decreasing concentrations in the eluates of the 22-32 and 32-50 with time. The only exception is a 35% increase in Co in the 32-50 mm sample.

The decreased leaching of heavy metals is assumed unrelated to changes in pH (Figure 9) and may indicate that more easily accessible metals are starting to deplete. As there are few data points for the metal analyses, there are also some uncertainties. The uncertainty in the very long term is whether the large pieces of alum shale will start to break down. If so, the surface area will increase and leaching rates may increase exponentially. After two years, there are no visual indications of such processes taking place.

Table 8. Levels of discussed elements in the solid phases.

ELEMENT		Rhomb porphyry	Black shale
Cd (Kadmium)	mg/kg TS	0.0803	6.96
Co (Kobolt)	mg/kg TS	7.17	19.3
Ni (Nikkel)	mg/kg TS	3.81	238
S (Svovel)	mg/kg TS	567	18900
Zn (Sink)	mg/kg TS	34.7	379
U (Uran)	mg/kg TS	6.62	69.4

### 3.1.4 Uranium

The trend of U leaching is comparable to that of the other parameters, with a considerably higher leaching in the 100 % alum shale column (0-22 mm) compared to all other columns, and also higher leaching from the 22-32 and 32-50 mm columns with 10 % alum shale compared to the 0-22 mm 10 % alum shale column. As with the other columns, the latter is explained by the earlier depletion of the buffer potential in the cruder columns, and consequently higher leaching/lower pH at this stage of the experiment.

There are no guideline levels for uranium in the M-608 guidelines. However, in the guidelines for drinking water an upper value of 140 µg/l U is given, which is to account for the radioactive properties of U. WHO has given a guideline value of 30 µg/l based on the toxicological properties of U. Environmental PNEC (Predicted no-effect concentration) for uranium is however 5 µg/L (Sheppard et al., 2005). Perhaps except for eluates in the 100 % alum shale column, the uranium levels do not seem as problematic as concentrations for the metals at this stage.

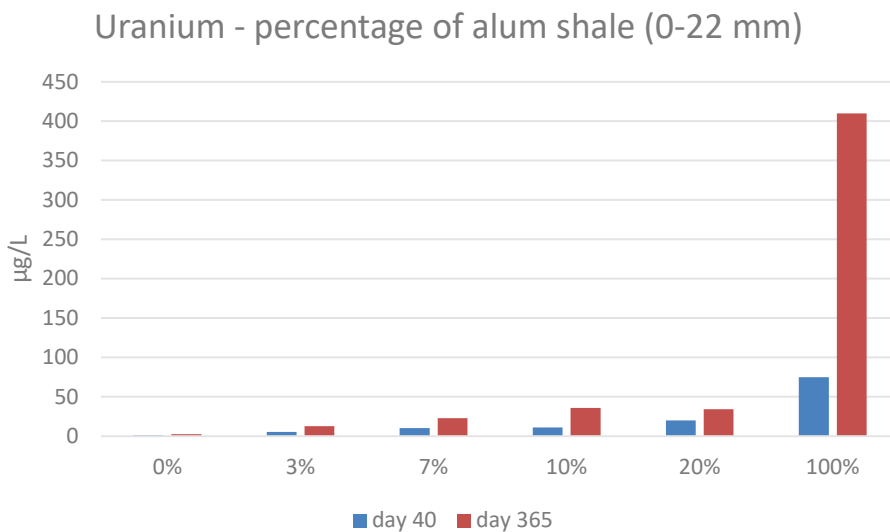


Figure 17. Comparison of uranium in eluates with 0-22 mm masses and varying percentage of alum shale.

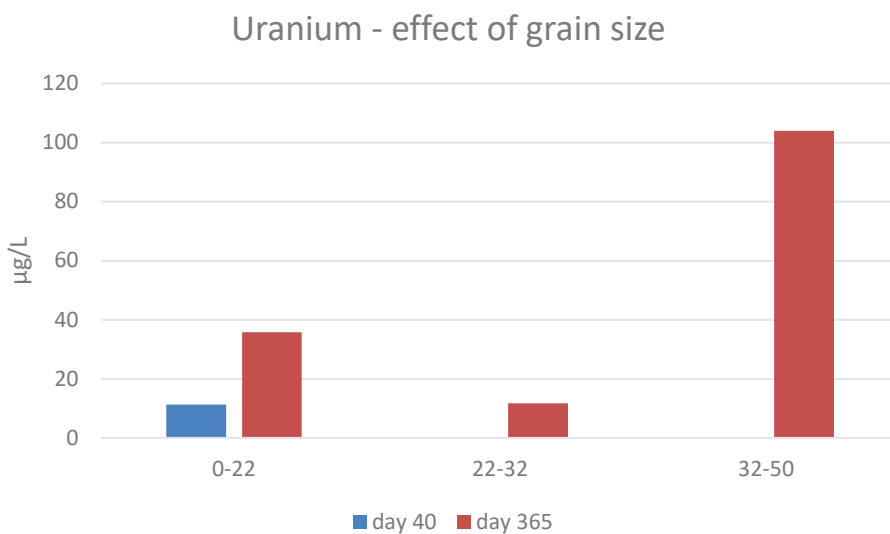


Figure 18. Comparison of sulphate in eluates with 10 % alum shale and varying grain size distributions.

### 3.1.5 Sulphate leaching

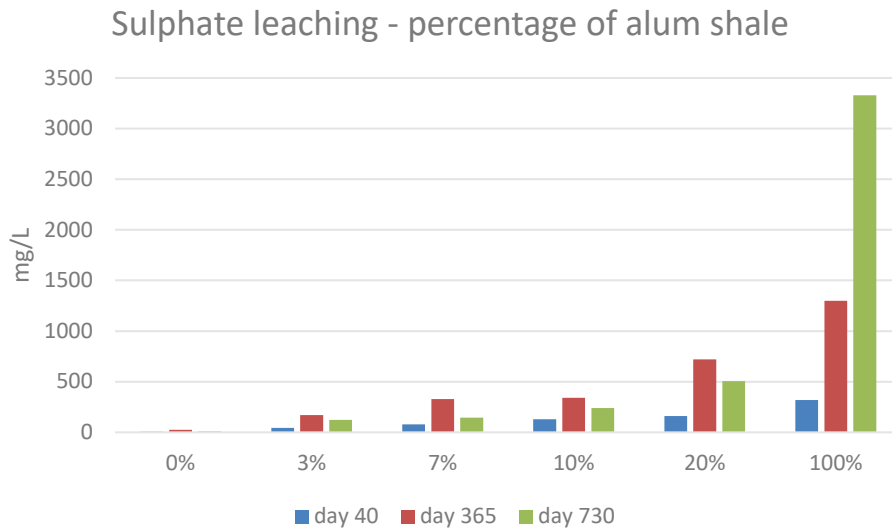


Figure 19. Comparison of sulphate in eluates with 0-22 mm masses and varying percentage of alum shale.

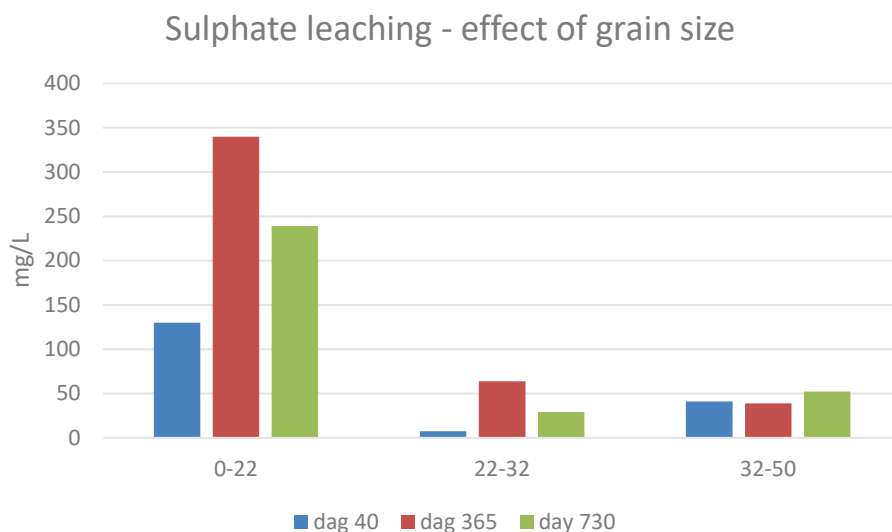


Figure 20. Comparison of sulphate in eluates with 10 % alum shale and varying grain size distributions.

The leaching of sulphur is related to the abundance of sulphide minerals in the black shale. Analysis of the alum shale showed that it contains 18 900 ppm of sulphur (section 2.4). Hence, as expected, the presence of sulphate in the eluates reflects the percentage

of alum shale in the column (Figure 19). Figure 20 further show that leaching is higher in the 0-22 mm column (10 % alum shale) compared to the larger grain size columns, and supports the initial theory that sulphide oxidation reactions would be faster in the column with larger surface area. The presence of sulphate in the eluates is likely a principal cause of variation in conductivity in the eluates (section 3.1.1). In all columns the leaching is considerable higher after one year compared to after 40 days, but lower after 730 days. The exception is the 100 % alum shale column, where leaching accelerates considerably from day 365 to day 730, as was also seen in pH, conductivity and metal measurements.

### 3.1.6 Discussion

It is unquestionable that a higher surface area gives a higher potential for leaching, and hence it was expected that the 0-22 mm (10 % alum shale) column would leach at a higher rate compared to the corresponding 22-32 and 32-50 mm columns. However, two years into the experiments the opposite is still the case for metals.

The explanation for this is in the reduced pH values measured in eluates of the higher grain size columns (Figure 9), and the explanation for the reduced pH values in these particular columns are found in the alkalinity, shown in Figure 15. Due to the lower available surface area, the 22-32 and 32-50 mm columns initially produced less alkalinity than the corresponding 0-22 mm column. However, the drop in alkalinity with time is equal or higher in the 22-32 and 32-50 mm columns ( $>0.2$  mmol/L per year). In the 22-50 mm column no alkalinity is detected after one year. This could indicate that the pH will not drop further, which indeed is the case after two years. The low conductivity and sulphate concentrations supports that the sulphide oxidation reactions in the columns with larger grain size is slower, as expected.

It is expected that the 0-22 mm column over time will produce a significantly higher leaching rate than what is currently observed for the higher grain size columns, if there is a pH drop in this column. It is therefore important that the experiments continue in 2023, as it is important to verify this assumption. This is crucial information in order to give correct advice for the disposition of similar masses.

The high surface area of the 0-22 mm column may also induce more precipitation, which can be an additional reason for the delayed onset of leaching.

## 3.2 Container experiments – a comparison

As shown in Table 2, the containers relevant for comparison with the column experiment are B5, B10 and B20, containing 5%, 10% and 20 % alum shale, respectively, as well as the VAS (100 % alum shale) and RM containers (0 % alum shale, only rhomb porphyry).

The principal difference between the columns and the containers, apart from the conditions, is that the containers consist of unsorted blasted rocks. This will include a broad grain size distribution, from 0 – 250 mm.

### 3.2.1 pH and conductivity

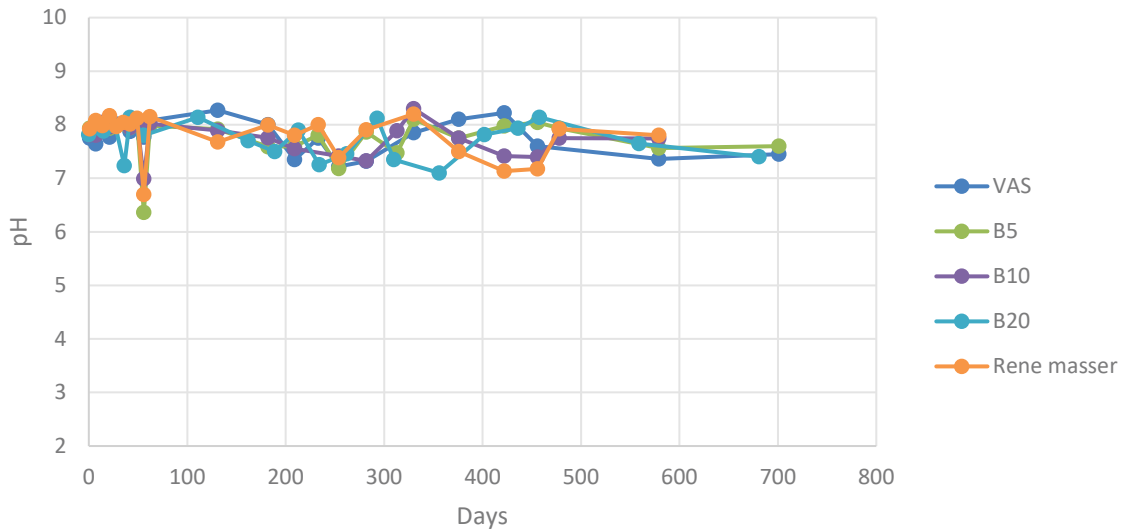


Figure 21. pH measured in the runoffs from the containers at different times. All values are averages of two parallel containers.

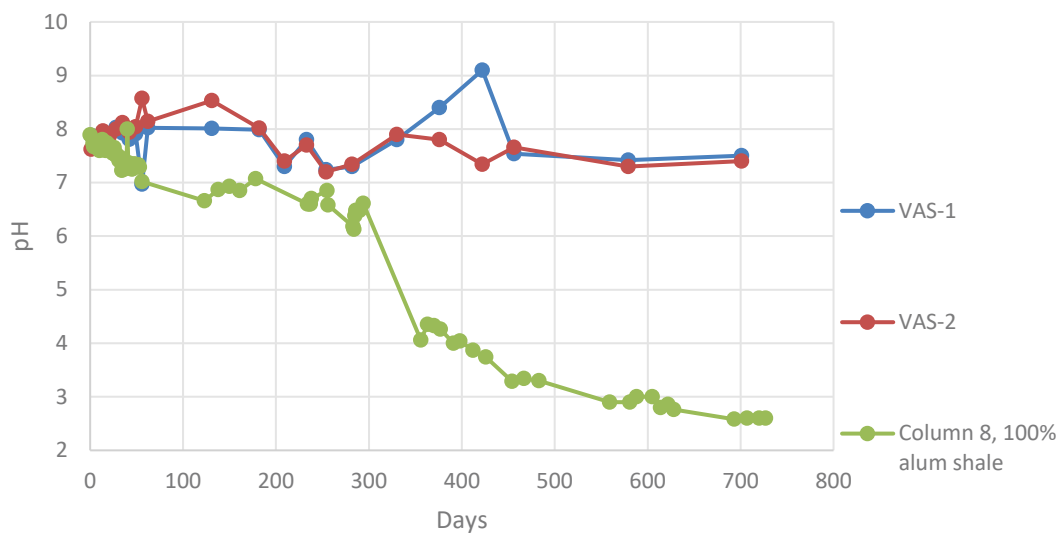


Figure 22. Comparison of pH measured in the 0-22 mm 100% alum shale column and the 'VAS' containers, with 100% blasted alum shale.



In contrast to the 0-22 mm column with 100% alum shale, no drop in pH is observed for the eluates from the 'VAS' containers (Figure 21 Figure 22). The reason for this is unclear but can be related to temperature and different quantities of moisture as well as grain size. Spring and summer of 2022 (the second year of the experiment) was unusually dry, and this may have delayed the onset of ARD. The measured conductivities show an increase early on for the column, from around 500 to 1500-2000  $\mu\text{S}/\text{cm}$  during the first 3-4 months, whereas the conductivity for the containers remained more or less unchanged during the same period. For the 'VAS' containers, the conductivity increase occurred about six months into the project Figure 24. Hence, a process that occurred quickly in the column, needed more time in the containers. The containers were started in the autumn, and the lower winter temperatures may have contributed in delaying the reactions.

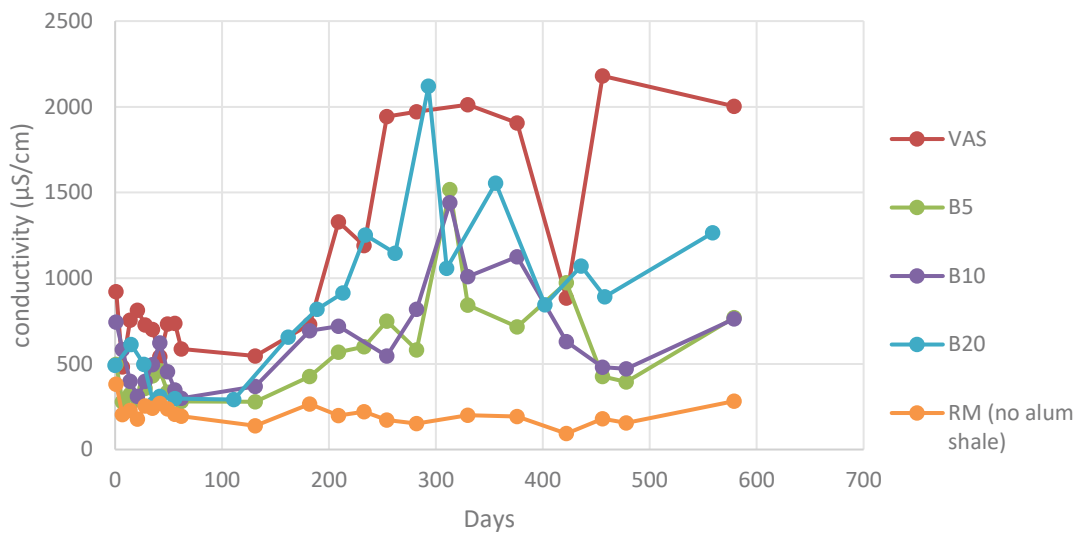


Figure 23. Development of conductivity in the containers. Values are averages of two parallel containers.

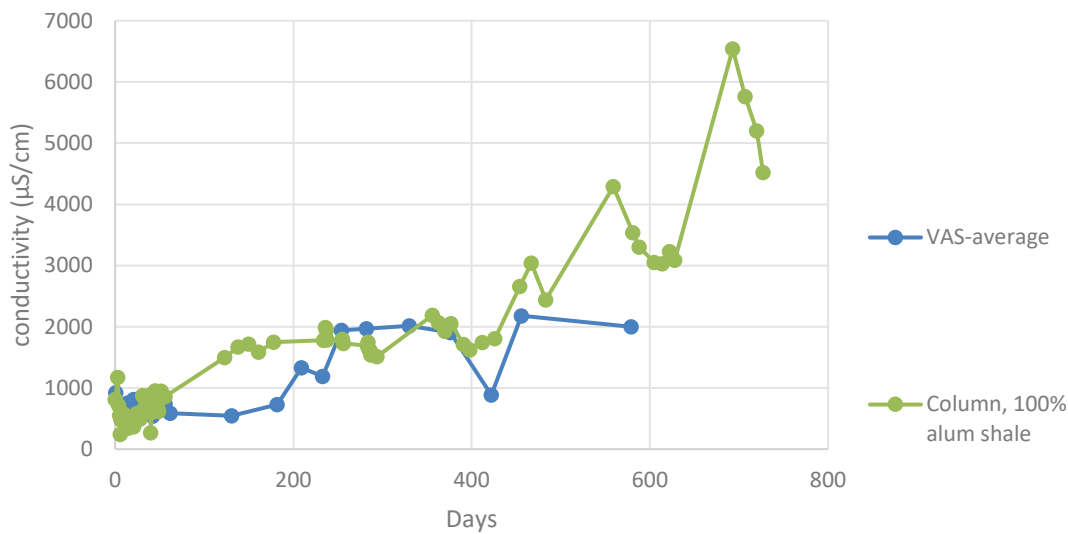


Figure 24. Comparison of conductivity measured in the 0-22 mm 100% alum shale column and the 'VAS' containers, with 100% blasted alum shale. From the curve of the column it is obvious that longer dry periods increase the conductivity significantly. For the columns the dry periods are indicated by no sampling points as pH and conductivity were always measured when columns were watered. This is not the case for the containers.

### 3.2.2 Alkalinity

Alkalinity results are compared for containers and columns in Figure 25 and Figure 26. Higher values for the containers compared to columns could e.g. be due to different liquid to solid ratio (fixed for columns, varying with precipitation for containers), and likely slower percolation through the containers giving longer contact time.

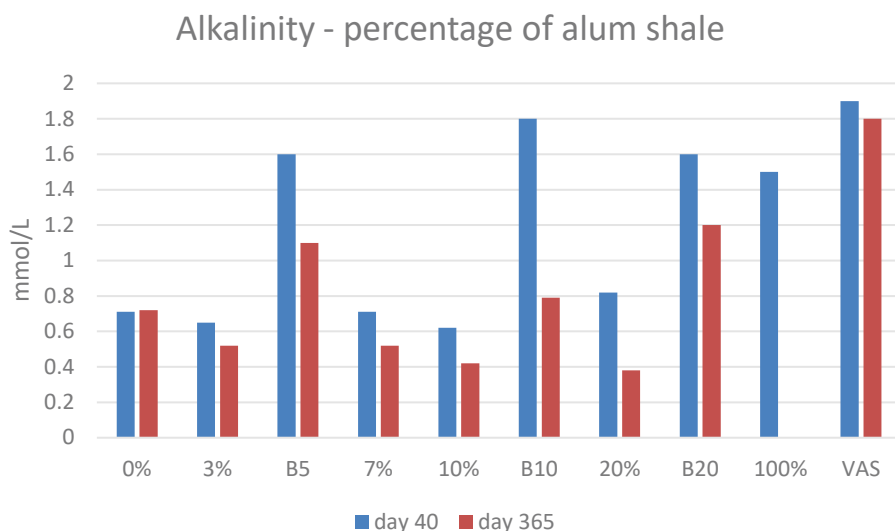


Figure 25. Comparison of alkalinity in eluates with 0-22 mm masses and varying percentage of alum shale. B5, B10, B20 and VAS are corresponding results from containers of blasted rock (0-250 mm) containing 5%, 10%, 20% and 100% alum shale, respectively.

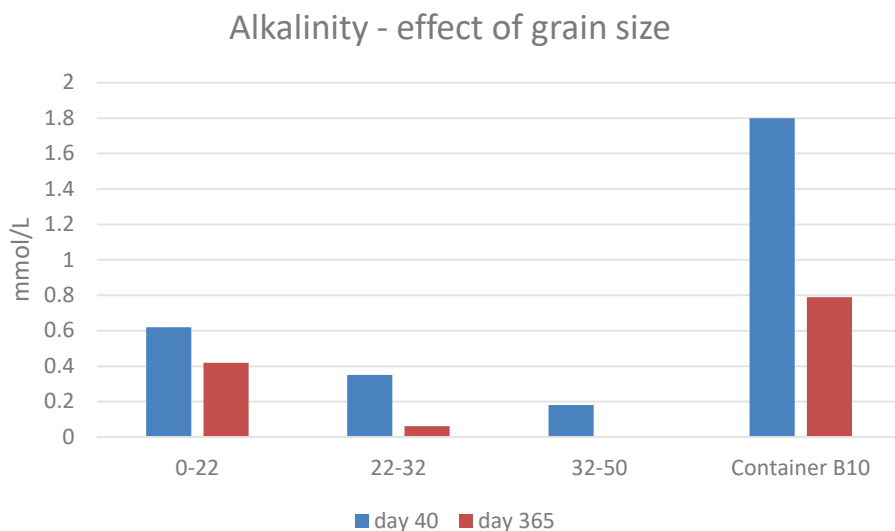


Figure 26. Comparison of alkalinity in eluates with 10 % alum shale and grain size distributions. Container B10 is the container with blasted rock (0-250 mm) containing 10 % alum shale.

### 3.2.3 Heavy metal leaching

Comparison of leachate concentrations of sink (Figure 27 and Figure 28) and cadmium (Figure 29 and Figure 30) for containers and columns are presented below.

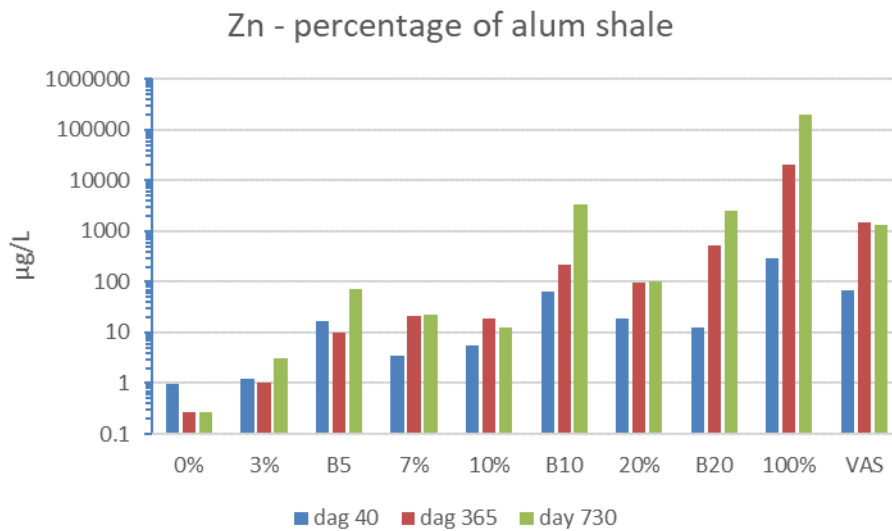


Figure 27. Comparison of Zn in eluates with 0-22 mm masses and varying percentage of alum shale. B5, B10, B20 and VAS are corresponding results from containers of blasted rock (0-250 mm) containing 5%, 10%, 20% and 100% alum shale, respectively. Note that y-axis is logarithmic.

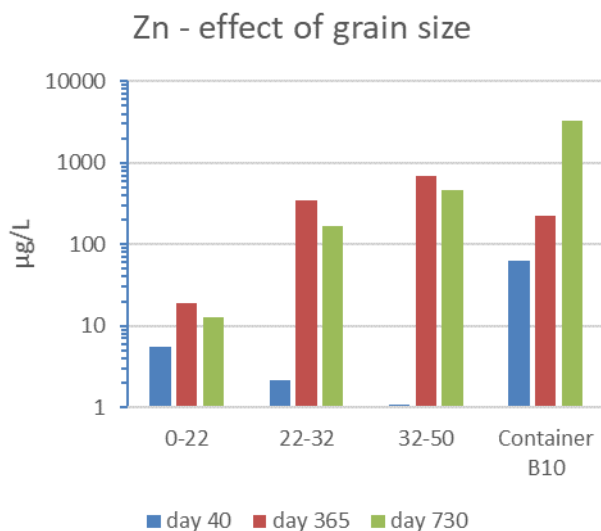


Figure 28. Comparison of Zn in eluates with 10 % alum shale and grain size distributions. Container B10 is the container with blasted rock (0-250 mm) containing 10 % alum shale. Note that y-axis is logarithmic.

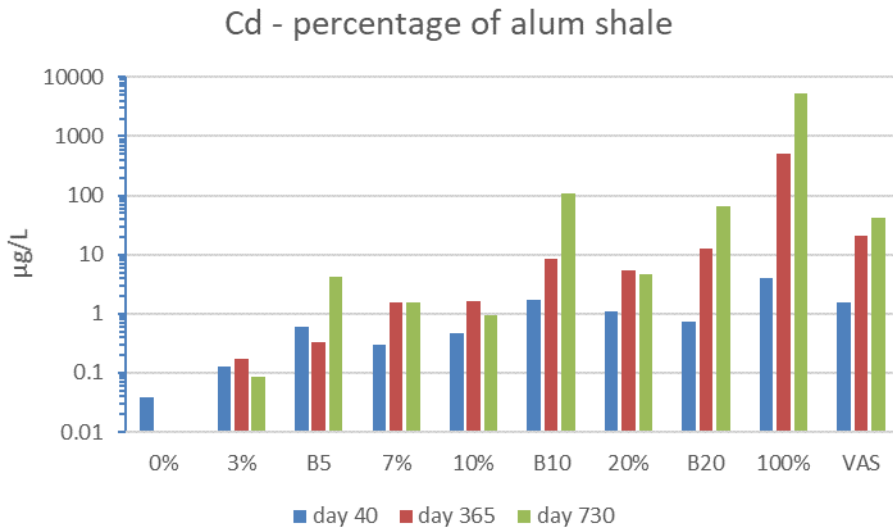


Figure 29. Comparison of Cd in eluates with 0-22 mm masses and varying percentage of alum shale. B5, B10, B20 and VAS are corresponding results from containers of blasted rock (0-250 mm) containing 5%, 10%, 20% and 100% alum shale, respectively. . Note that y-axis is logarithmic.

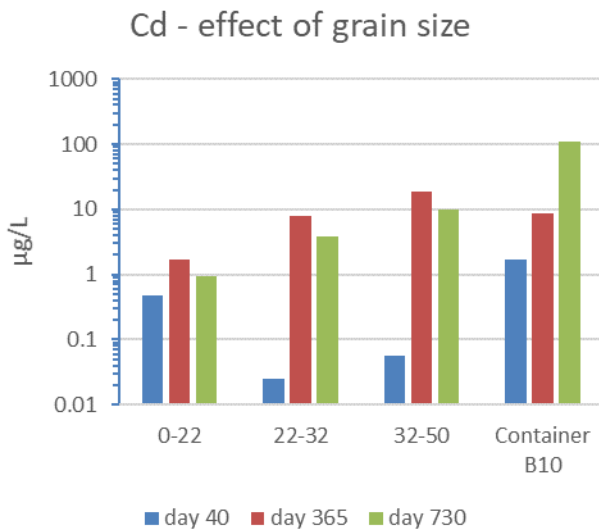


Figure 30. Comparison of Cd in eluates with 10 % alum shale and grain size distributions. Container B10 is the container with blasted rock (0-250 mm) containing 10 % alum shale.

### 3.2.4 Uranium

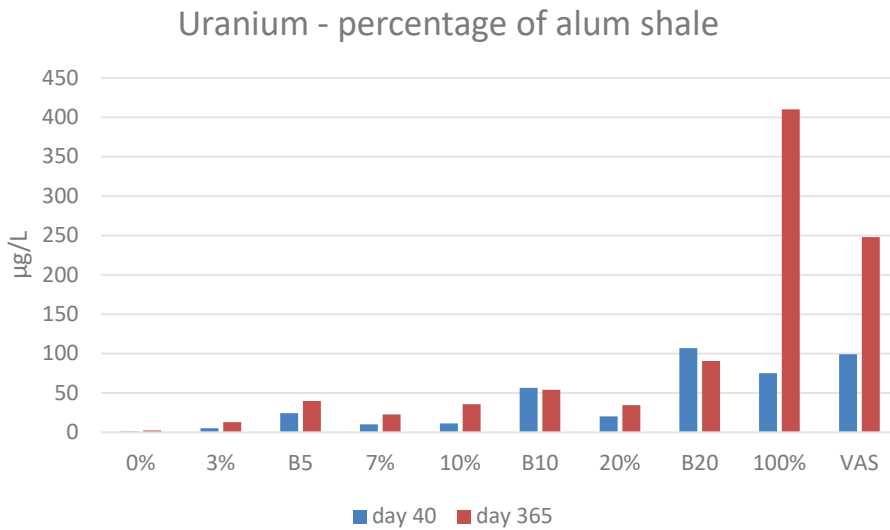


Figure 31. Comparison of U in eluates with 0-22 mm masses and varying percentage of alum shale. B5, B10, B20 and VAS are corresponding results from containers of blasted rock (0-250 mm) containing 5%, 10%, 20% and 100% alum shale, respectively.

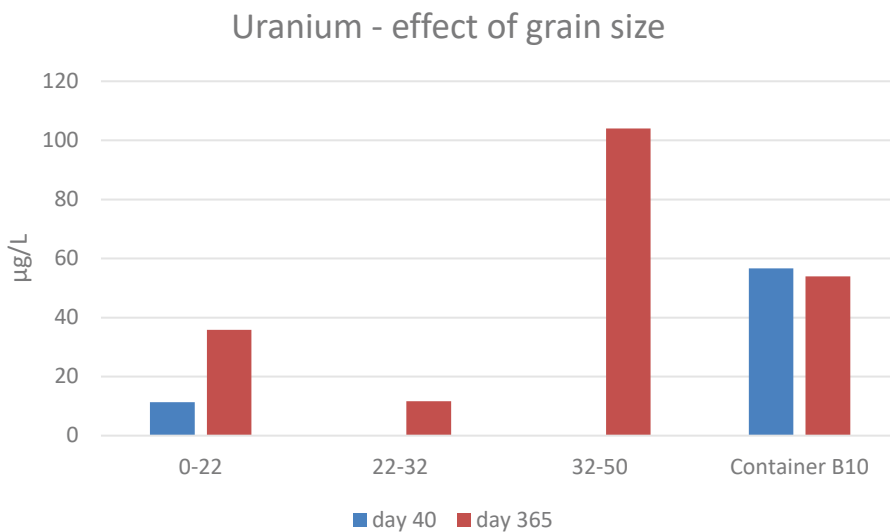


Figure 32. Comparison of U in eluates with 10% alum shale and grain size distributions. Container B10 is the container with blasted rock (0-250 mm) containing 10% alum shale.

### 3.2.4.1 Sulphate leaching

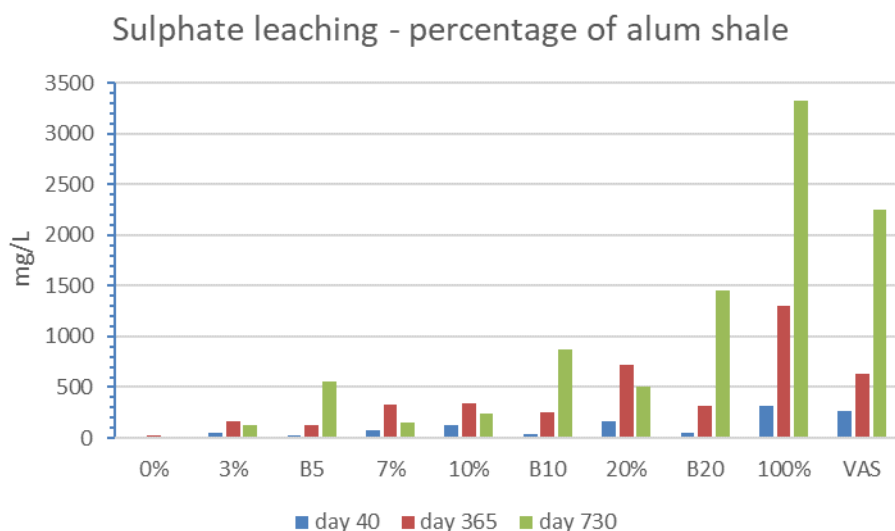


Figure 33. Comparison of S in eluates with 0-22 mm masses and varying percentage of alum shale. B5, B10, B20 and VAS are corresponding results from containers of blasted rock (0-250 mm) containing 5%, 10%, 20% and 100% alum shale, respectively.

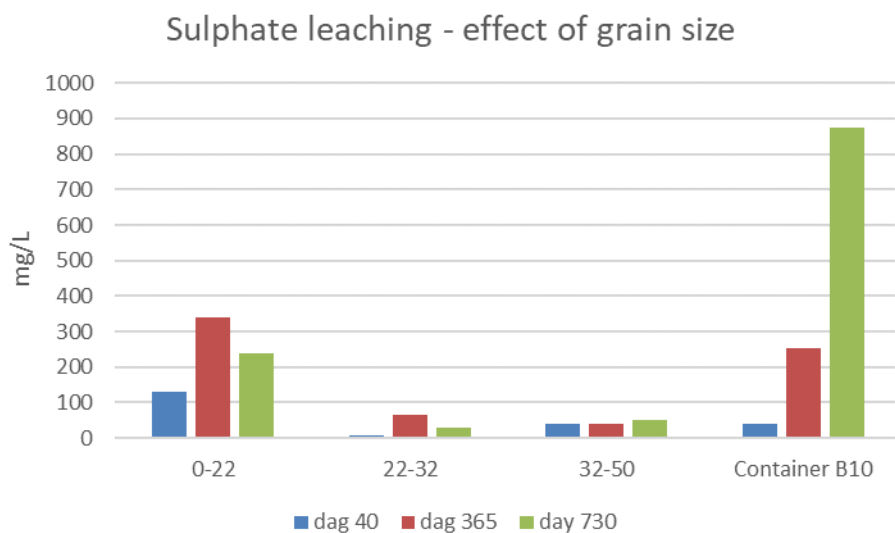


Figure 34. Comparison of S in eluates with 10 % alum shale and grain size distributions. Container B10 is the container with blasted rock (0-250 mm) containing 10 % alum shale.

### 3.2.5 Discussion

Regarding the measured leaching from the columns and the containers, the general trend is that there is a larger difference between the day 40 and day 365 analyses. For the columns there is generally low variation from 365 to 730 days, except for the 100% alum shale column. This is not the case for the containers, which show significantly increased leaching for the 701 day samples (represented as 730 days in the plots for simple comparison with the column data), compared to the 365 day samples. This is surprising considering the pH in all containers are near neutral at the point of sampling.

It is interesting to note that analysis of container eluates at day 579 showed considerably lower levels of leaching compared to the 701 day samples (data not shown). At day 579 the containers needed to be added water for sampling due to a longer dry period, so these data are excluded in the report due to lower comparability. However, the results may indicate that the concentrations of leachates in containers are highly dependent on season and weather conditions leading up to the sampling.

Dilution effects may play a major role. Whereas column eluates are the product of the same amount of water added at regular intervals, this varies considerably for the containers. Hence, it is difficult to directly compare the container and column experiments.

## 4 Summary

Despite the experiments should run longer in order to draw certain conclusions, some trends are apparent, in particular for the columns. Further, some of these trends are unexpected, such as the amount of leaching from the larger grain size columns.

These are the main points so far:

- Lower content of alum shale delay the onset of pH reduction and reduces the leaching, likely due to the additional buffering of the rhomb porphyry. Continued monitoring of the columns is therefore required. Stable conductivity in the leachates of the columns with mixed masses indicates that weathering processes are continuing at a constant rate, while for the container experiments the conductivity, sulphate and calcium leaching seems to be increasing, indicating increasing weathering rates
- Due to a lower surface area available for reaction in the higher grain size columns, the buffer capacity depletes quicker in these columns compared to the 0-22 mm column, leading to lower pH values and higher leaching of metals in the 22-32 and 32-50 mm columns one year into the experiment, compared to the corresponding 0-22 mm column. However, due to the lower surface area, pH is manageable and not expected to decrease significantly. Although the leaching of metals is considerably lower in the 22-32 and 32-50 mm columns compared to the 0-22 mm 100% alum shale column, the values are well above



guideline values for fresh water, and could be problematic in certain cases. New eluate analyses should be conducted in six months to verify that leaching rates does not increase further.

- The processes occur much slower in the containers compared to the columns. This is likely due to different temperature and exposure to water, as well as the different grain size distributions.
- It was observed during the column experiments that the finer mass fractions (0-22 mm) may take weeks to completely dry; in fact, the masses in the lower part of the 40 cm column presumably remained moist for most of the two-year period.
- Continuous access to moisture and oxygen means that the first flush of water has a lower pH and higher conductivity.

## 5 Case report

### 5.1 Mixed masses from TBM-excavation of pipeline

In a case with drilling with TBM for a pipeline through alum shale and syenite, the material was tested, and risk assessment were performed to see if alternative disposition of the mixed masses was possible. Part of the route contained 0.1-2 m layers of black alum shale (Uranium concentrations below 80 mg/kg) mixed with syenite and lime-rich shales. The latter types of masses do not produce acid, and therefore do not require any special treatment. To some extent they are buffering, which was confirmed. Since this case is of direct relevance for the research discussed in this report, the work done for this specific case is elaborated here.

The TBM processes mixes the masses and produces a higher fraction of fines compared to blasting.

The key questions treated were:

- What is the potential for acid drainage and leaching from the mixed masses containing different amounts of alum shale?
- Are there any measures that can provide safe reuse of the spoil or safe storage on deposit as inert waste?

An evaluation was done for alum shale fractions between 5-30% in combination with the local and specific mix of syenite and limestone. In addition to calculating the buffering capacity of the combined masses, the grain size distribution of the masses was also considered an important factor.

It was assumed that the softer alum shales would crush into finer fractions compared to the syenite. This means that the acid producing potential of the alum shale must be weighted more than the buffering capacity of the syenite due to higher available surface

area. In addition, it means that separating the masses by size could give lower content of alum shale in the fraction of larger grain size.

A petrographic analysis of the spoil, sieved into different fractions, showed that the content of alum shale was lower in the larger size fractions (Figure 35). The fraction of alum shale in the 16-19 mm fractions deviates from the trend, and this might have been caused by little material in this fraction. The effect of the different grain size distribution on surface area was estimated by calculating surface area based on the grain size distribution curves for both the alum shale and syenite. The acid average producing capacity of the alum shale was weighted proportionally more than the average buffering capacity of the other masses when calculating the potential for acid drainage. To also account for future weathering of the alum shale, which is expected to increase the surface area, the factor chosen to account for higher alum shale surface area was conservative and estimated to 0.4 (40% reactive surface area from syenite and limestone compared to 100% reactive surface from alum shale). Hence, in the calculations of NP:AP (neutralization potential: acidification potential), the concentrations of total inorganic carbon (TIC) in the syenite was multiplied by 0.4. Based on this, the NP:AP relationships for different percentages of alum shale in the mixed masses was calculated (Table 9).

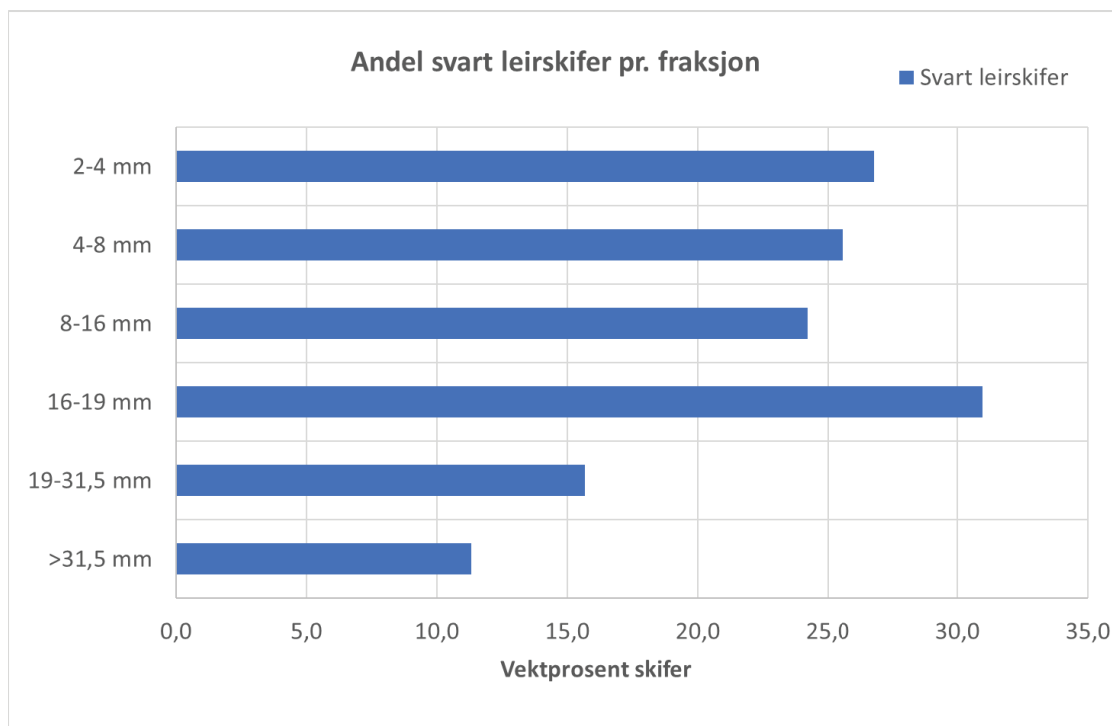


Figure 35. Data from petrographic analysis, where the masses were sieved into different fractions and the black/non-black grains were counted and weighed.

*Table 9. Results of NP:AP calculations for the mixed masses. PN:NP greater than 1 means that the neutralization potential is higher than the acidification potential.*

Fraction black shale – fraction syenite	Sulphur mg/kg	TIC mg/kg	PA kgCaCO <sub>3</sub> eq/t	PN kgCaCO <sub>3</sub> eq/t	PN:AP
5% - 95%	6783	3437	21,2	28,6	1,4
10% - 90%	8820	3344	27,6	27,9	1,0
15% - 85%	10858	3252	33,9	27,1	0,8
20% - 80%	12895	3159	40,3	26,3	0,7
30% -70%	16969	2973	53,0	24,8	0,5

Table 9 shows that a 10 % fraction of alum shale gives a NP:AP of 1, meaning the neutralization potential equals the acidification potential.

In the project, the sorting of masses was monitored and controlled manually, to document that the spoil with low content of potentially acid-producing rocks was handled according to procedures made for the calculated fractions. In this way, the masses with low content alum shale could be delivered to disposal sites for inert masses in stead of being treated as acid-producing.

However, it is of crucial importance to get experimental data on such mixed fractions in order to give more secure advice in the future. In particular, we need the theoretical understanding to predict how these masses will behave under different conditions in both the near and far future. In the worst case, unforeseen processes can cause these masses to become problematic in the future, and this must be avoided.

In comparison, the containers with 10 % alum shale does not have a particularly high leaching of metals, and do not produce acid after two years of exposure. Although it should be noted that these masses have a different geographic origin, the knowledge we obtain from these experiments will be highly valuable for future advising.

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