

# Classification of Olivin (0-3 mm) with respect to MARPOL Annex V criteria



# Norwegian Institute for Water Research

- an institute in the Environmental Research Alliance of Norway

# REPORT

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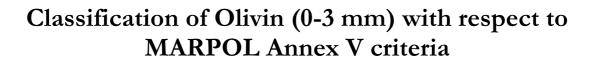
Classification of Olivin (0-3 mm) with respect to MARPOL Annex V criteria. Olivin (0-3mm) did not meet any of the Annex V criteria for garbage.

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# Preface

This report is an assessment of Olivin (0-3 mm) with respect to the MARPOL Annex V criteria.

Oslo, 10 March 2016

August Tobiesen

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# 1. Summary

An assessment has been made of the product Olivin (0-3 mm) with respect to MARPOL Annex V criteria. Olivin (0-3 mm) is fine gravel intended to be shipped by bulk carriers. Olivin (0-3) does not fulfil Criteria 1 and 2 with respect to ecotoxicity and is therefore not assessed to be "Harmful to the environment". With respect to criteria 3-6 regarding human exposure, these are not fulfilled. Criteria 7 is not met as Olivin (0-3 mm) does not contain plastics.

# 2. Introduction

Sibelco Nordic AS Norway produces gravel of different sizes. The Olivin rock is blasted out by explosives and pre-ground at site. The raw material (0-120 mm) is transported on conveyer belts to the loading port. At the loading port the process plant separates the Olivin gravel into different fractions prior to shipment. This assessment concerns the shipment of the Olivin 0-3 mm size fraction. Prior to shipment the product is stored outside and has a water content of 4-6 % when loaded onto bulk carriers cargo boats (max. size 70 000 tons). The revised International Convention for the Prevention of Pollution from Ships (MARPOL) Annex V lays out specific compliance requirements affecting the carriage of cargoes by sea and came into effect on the 1st of January 2013. Sibelco Nordic AS Norway commissioned NIVA to provide an assessment of how their Olivin material stands with regard to the MARPOL Annex V criteria. A key requirement of Annex V is that residues or wash water containing residues of cargoes identified as "harmful to the marine environment" cannot be discharges into the sea, but will have to be discharges at adequate port reception facilities (IMO 2012).

# 3. Assessment criteria Annex V

### Applicable criteria

MARPOL Annex V (IMO 2012) lists the specific criteria that results in a substance being considered as "harmful to the marine environment". These classifications are:

- 1) Acute Aquatic toxicity Category 1; or
- 2) Chronic Aquatic toxicity Category 1 or 2; or
- 3) Carcinogenicity 1A/1B and high bioaccumulation AND not rapidly degradable; or
- 4) Mutagenicity 1A/1B and high bioaccumulation AND not rapidly degradable; or
- 5) Reproductive toxicity 1A/1B and high bioaccumulation AND not rapidly degradable; or
- 6) Specific Target Organ Toxicity repeated exposure (STOT-RE)Category 1 and high bioaccumulation and not rapidly degradable; or
- 7) If a solid bulk cargo contains or consists of synthetic polymers, rubber, or plastics feedstock pellets it is automatically classified as "harmful to the marine environments.

Assessment of the first six Criteria is made using the UN Globally harmonized system for classification and Labelling of chemicals (GHS), 6th revised edition (UN 2013). Both the International Maritime Dangerous Goods Code (IMDG) (IMO 2010) and the UN Recommendations on the transport of Dangerous Goods- Model Regulations (UN, 2013) call up the GHS. Thus dangerous goods classification conducted in accordance with the IMDG code and the UN model regulations informs the classification of the substance as per the MARPOL Annex V criteria.

The tests performed on the Olivin material, to enable the classification to be performed is summarized in **Table 1** and the test report Molab (2008) and NIVA (2016) are provided in Appendix 1.

**Table 1.** Tests provided on Olivin (0-3 mm) material. The Olivin tested originates from Sibelco Nordic stone quarry at Åsheim, Norway

| Test type                 | Test material           | Aim of study           | Reference    |
|---------------------------|-------------------------|------------------------|--------------|
| Leaching tests – initital | Oliflux (0-3 mm) Olivin | Determine leakage of   | Molab (2008) |
| and stabilized leaching   |                         | Cr, Cu and Ni          |              |
| Algal growth inhibition   | Olivin (0-3 mm)         | Determine Growth       | NIVA (2016)  |
| test OECD 201             |                         | inhibition in WAF from |              |
|                           |                         | Olivin (0-3 mm)        |              |
| Mesocosm test with        | Olicap (0-0.075 mm)     | Determine effect on    | NIVA (2009)  |
| Olicap.                   |                         | sediment dwellers      |              |

# 4. Characterization of Olivine (0-3 mm)

## 4.1 Colour

Fine grained material light greenish gray color

## 4.2 Composition

Olivin is a stone mineral with high content of  $(Mg,Fe)_2SiO_4$  (**Table 2**). The Olivin (0-3 mm) has a surface area of 1.5 m<sup>2</sup>/g. Density is 3.2-3.3 kg/l. Grain size distribution is shown in **Table 3**, and indicates that close to 95 % of grain is <3 mm.

Trace elements of Cr, Cu and Ni is present, see also **Table 4** 

**Table 2.** Chemical composition of Olivin (0-3 mm)

| Chemical | %   |
|----------|-----|
| MgO      | 49  |
| SiO2     | 41  |
| Fe2O3    | 7   |
| Al2O3    | 0.5 |
| Cr2O3    | 0.3 |
| NiO      | 0.3 |
| MnO      | 0.1 |

Table 3. Grain size distribution for Olivin (0-3 mm) (Oliflux tehnical data sheet)

| Sieve size | 8 | 5.6 | 4   | 2   | 1   | 0.5  | 0.25 | 0.063 | < 0.063 |
|------------|---|-----|-----|-----|-----|------|------|-------|---------|
| (mm)       |   |     |     |     |     |      |      |       |         |
| %          | 0 | 1.0 | 2.5 | 7.0 | 9.5 | 20.0 | 26.0 | 29.0  | 5.0     |

## 4.3 Water solubility, Leaching

Stone minerals are not water soluble; however some leaching of minerals may occur following grinding to a fine powder. The degree of leaching from Olivin (0-3 mm) has been investigated by Molab (2008). Leaching was investigated according to shaking test NS EN 12457-2 using 100 g of test compound in 1 liter of water, and the Column test CEN EN 14405 using 110g of test compound in 1 liter of water. The dissolution water had a pH of 9.9 following 24h shaking. The results for the leaching test are shown in **Table 5**. The analysis showed values well below the criteria for class III. The results for the column test are shown in **Table 5** and **Table 6**. **Table 4** shows that when correcting for levels in control water all analytical values are below stabilized class II acceptance criteria. **Table 6** shows a steady decrease in levels for copper and nickel, while chromium remains steady within the scope of this test.

**Table 4.**Results from the analysis of shaking eluates. Results are compared to accept values set forth by the Norwegian Environment Agency for leaching in marine water (Molab, 2008).

| Parameter | Shaking test | Uncertainty | Control water | Stabilized leaching |
|-----------|--------------|-------------|---------------|---------------------|
| TT        | 0.0          | +/-         |               | Class II            |
| pH        | 9.9          | 0.1         |               | -                   |
| Cr (µg/l) | 0.549        | 0.189       | 0.451         | 1.5                 |
| Cu (µg/l) | 0.385*       | -           | 0.642         | 1.5                 |
| Ni (µg/l) | 0.827        | 0.35        | < 0.5         | 5                   |

<sup>\*</sup>non accredited analysis

**Table 5.** Results from the analysis of the column eluates. The results are compared to the accept values set forth by the Norwegian Environment Agency for leaching in marine water. Analysis results for eluates has not been corrected for values in control water. (Molah, 2008)

| Parameter | Column test<br>L/S 10 | Uncertainty<br>+/- | Control water | Initial leaching<br>Class III |
|-----------|-----------------------|--------------------|---------------|-------------------------------|
| рН        | 10.0                  | 0.1                | -             | -                             |
| Cr (µg/l) | 0.427                 | 0.190              | 0.451         | 1.5                           |
| Cu (µg/l) | 0.913                 | 0.331              | 0.642         | 1.5                           |
| Ni (μg/l) | < 0.5                 | -                  | < 0.5         | 5                             |

**Table 6.**Results from the analysis of the column eluates for all 7 fractions. Values are not corrected for control water content. (Molah, 2008)

| (1110)    | ,, <b>-</b> 000) |         |         |         |         |         |        |         |
|-----------|------------------|---------|---------|---------|---------|---------|--------|---------|
| Parameter | L/S 0.1          | L/S 0.2 | L/S 0.5 | L/S 1.0 | L/S 2.0 | L/S 5.0 | L/S 10 | Control |
|           |                  |         |         |         |         |         |        | water   |
| рН        | 9.5              | 9.5     | 9.7     | 9.5     | 9.3     | 8.1     | 10.0   | -       |
| Cr (µg/l) | 0.414            | 0.530   | 0.496   | 0.645   | 1.02    | 0.626   | 0.427  | 0.451   |
| Cu (µg/l) | 36.6             | 9.89    | 4.37    | 3.17    | 2.46    | 1.58    | 0.913  | 0.642   |
| Ni (μg/l) | 5.05             | 1.95    | 0.773   | 0.555   | 1.01    | < 0.5   | < 0.5  | < 0.5   |

# 5. Aquatic toxicity classification

Criteria 1 and 2 of the MARPOL Annex V criteria directly relates to the environmental Hazard classification of substance (i.e. Class 9, Miscellaneous Dangerous Goods). Environmental hazard is determined by the aquatic toxicity of a substance. Because the aquatic toxicity of metals depends on the bioaccessible fraction this roughly translates to the dissolved free ion concentration, the classification rules are limited to the hazard posed when they exits as "dissolved" metal ions (UN 2013). The key criterion for the classification of metals and poorly soluble inorganic metal compounds is whether the substance is sufficiently soluble that the concentrations dissolved following normal attempts at solublisation exceed the aquatic toxicity benchmarks (LC50 or EC50 values). This means that if the dissolved metal concentration ≥ L(E)C50 for fish, invertebrate or algae, then the product is classifiable as a Class 9 Miscellaneous Dangerous Goods (Environmentally hazardous substance). If classified, the substance is assigned to an aquatic category (1 or 2) based on the severity of its toxicity. The Olivin (0-3 mm), contains trace amounts of chromium, copper and nickel.

Relevant studies applicable for dangerous goods classification purposes are:

- 72 h or 96 h algal test with EC50 or LC50
- 48 h crustacean test with EC50 or LC50
- 96 h fish toxicity test with EC50 or LC50

The 3 metals measured in the dissolution test are evaluated below using endpoints arrived at in the EU risk assessment of these metals. EU Risk assessments are performed using only high quality data and a conservative approach.

#### 5.1 Chromium

Chromium exists dissolved as either chromium (III) or (VI), at a high pH typical of seawater chromium will mainly be in the chromium (VI). This is also the more toxic oxidation state. The EU risk assessment report for chromium (EU 2005), concludes that it is the crustaceans and fish that are the most sensitive group of organisms, and they conclude based on a NOEC (No Effect Concentration) of  $10 \mu g/l$  that the chromium (VI) PNEC (Predicted No Effect Concentration)=  $3.4 \mu g/l$ .. Highest measured chromium value in is  $1.02 \mu g/l$  (**Table 6**). As "dissolved" concentration of chromium in eluate is <LC50 and EC50 for chromium, Olivin (0-3mm) is not classified as hazardous with respect to chromium leaching.

## 5.2 Copper

An EU Risk assessment (EU, 2008a) has been performed on Copper where a PNEC<sub>marine</sub> of 2.6 μg/l has been established for the marine environment. This value is based on NOEC studies. They determinded that algae were the most sensitive organism group. A search in the EPA ECOTOX database (Ecotox, 2015) for acute test data gave a 72 h growth inhibition test EC50 of 18 μg/l for the alga *Nitzchia closterium* as the lowest acute endpoint. Lowest chronic endpoint a NOEC for algae is 4.4 μg/l for the algae *Pheadactylum tricornutum*. While the initial column eluate gave a maximum value of 36 μg/l, this is rapidly reduced to <1.0 μg/l, in the final eluate (see **Table 5** and **Table 6**). Also the Shake Test (**Table 4**) gave a concentration of less than 1.0 μg/l. As both shaking test and final column eluate test give concentrations < the marine PNEC of 2.6 μg/l, Olivin is not classified as hazardous with respect to copper leaching.

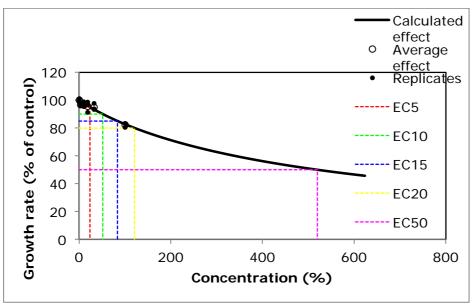
## 5.3 Nickel

An EU Risk assessment (EU, 2008b) has been performed on Nickel where a PNEC of  $2.3\mu g/l$  has been established for the marine environment. This value is based on NOEC studies, with the lowest being an EC10 of 22  $\mu g/l$ , and can be compared with measured concentrations in (see **Table 4** and **Table 6**), where a maximum of  $5.05 \mu g/l$  was measured. As this is less than the EC10 of  $22 \mu g/l$ . Olivin is not classified as hazardous with respect to nickel leaching.

## 5.4 Algal growth inhibition test

To further provide assurance that the combined leachate from Olivin (1-3 mm) is not acute toxic to marine organism a algal growth inhibition test was performed on the marine diatom *Skeletonema costatum* (NIVA, 2016). This test will test the effect of all possible compounds leaching from Olivin together and therefore include possible additive or multiplicative effects. The algal growth test covers many generation and is therefore considered to be a very sensitive test.

In the test 200 g of Olivin (1-3 mm) was mixed with 1 liter of alga growth medium (ISO 10253) and shaken on an orbital shaker for 24h. After being left to settle for 30 min the medium was decanted and filtered through 0.22 µm filter. The pH was adjusted from 8.6 to 8.0 by addition of HCl. The result of the test is shown in **Figure 1**, where it is evident that EC50 is not reached even at 100 % concentration at a loading rate of Olivin of 200g/l. The required test dissolution loading is 100 mg/l. As indicated in **Figure 1**, an EC50 growth inhibition was not reached in this study. This study supports the results from the dissolution tests which indicate that release of toxic metals is less than the concentration that gives acute effects on algal growth.



**Figure 1.** Dose-response curve of Olivin showing growth rate d-1 (% of control) after 72 hours of exposure. Concentration is given in % Olivin solution where the loading rate is 200g to 1L medium.

## 5.5 Effect of Olivin on sediment dwellers, a Mesocosm study

A mesocosm sediment study was performed with the object of providing experience of Olivine (Olicap is a finer grain size compared to Olivin 1-3 mm) as a capping material for remediation purposes. Apart from ascertaining Olvins effectiveness to contain contaminats the study also included effects on ragworm (*Neris virens*) burrowing activity. The sediments containing ragworm were capped with an approximately 2.5 cm layer of Olivine (Olicap has a smaller grain size than Olivine(0-3 mm) and left for 6 months with feeding twice a week with algal pellets. Control sediments were capped with equal amounts of sand. The 10 cm water layer above the sediment was aerated constantly during the test period. There was no ragworm mortality observed in either sediment treatments and ragworm activity as observed as bioturbation of capping material into sediments were similar for both sand and Olivin. The high activity observed for ragworms in sediments capped with Olicap indicate that Olivin do not effect behavior of ragworms.

## 5.6 Environmental hazard classification of Olivin (0-3 mm)

The Dangerous Goods classification for environmentally hazardous material is achieved by comparing the metal concentration under specified solubility test criteria with concentrations shown experimentally to affect aquatic organisms. The solubility test criteria stipulated for dangerous goods classification is the dissolution test described in Appendix 1. The results are presented in section 3. The results are presented in section 4

The results for the 24 h dissolution test are assessed in the following manner:

- If the soluble concentration in a 100 mg(l loading ≤ L(C)50 then it is not an environmentally hazardous substance for the purpose of transport (UN model regulations and IMDG section 2.9.3.3.1)
- If the soluble concentration in a 100 mg/l loading > L(C)50 then there is a need to progress to a full 7 day transformation/dissolution test for further evaluation (UN model regulations and IMDG, section 2.9.3.3.1)

The dissolved concentration of chromium, copper and nickel was < acute aquatic L(C)50 and chronic NOEC for marine organisms, when comparing results for shaking test and also when comparing the latter column eluate fractions.

# 6. Criteria 3-6: Humane / animal toxicity

From the composition for the product and particle size distribution results, together with information from Safety Data Sheet (Oliflux technical data sheet, SDS(2016)), an assessment is made for Olivin (0-3 mm) based on the criteria for the GHS (UN 2015). Neither of Criteria 3-6 is noted in the SDS (2016). From the information provided, the Olivin (0-3 mm) is not classified as hazardous to human health

# 7. Criteria 7: Contain plastics

The gravel product Olivin (0-3mm) does not contain any plastic compounds (see **Table 2**) and therefore this criterion is not met.

# 8. Conclusions of Annex V criteria with respect to Olivin (1-3 mm)

## 8.1 Criteria 1-2: Aquatic toxicity

Olivine (0-3 mm) product is not classifiable as a Class 9 Miscellanous Dangerous Good (i.e it is not an environmental hazardous substance) (see section 3.5).

Criteria 7; synthetic polymers, rubber, or plastics feedstock;

Olivin (0-3 mm) does not come into any of these categories.

Therefore, with respect to Olivine (0-3 mm) this product does not meet the criteria to be considered "harmful to the marine environment" under the revised MARPOL Annex V (IMO, 2012)

## 8.2 Criteria 3-6: Human/animal toxicity

From the composition for the product and particle size distribution results, together with information from Safety Data Sheet (Oliflux technical data sheet, SDS(2016)), an assessment is made for Olivin (0-3 mm) based on the criteria for the GHS (UN 2015). Neither of Criteria 3-6 is noted in the SDS (2016). From the information provided, the Olivin (0-3 mm) is not classified as hazardous to human health

## 8.3 Criteria 7: Contain plastics

The gravel product Olivin (0-3mm) does not contain any plastic compounds (see **Table 2**) and therefore this criterion is not met.

# 9. References

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# 10. Appendix

Appendix 1



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Fax: 22 18 52 00

#### TESTREPORT

# Algae, Growth inhibition Skeletonema costatum

NIVA method K6



Test substance: Olivin 0-3 mm Lab. code: **B860** 

Recieved at NIVA: 30.11.2015

Test method: ISO 10253: Water quality – Marine growth inhibition test with

Skeletonema costatum and Phaeodactylum tricornutum

Organism: Skeletonema pseudocostatum (formerly S.costatum) NIVA BAC1

Growth rate 72 timer Test parameter:

Stem culture: Grown in 10% Z8 growth medium in seawater, continuous orbital

shaking and low intensity light at 20±2°C.

Test start date: 15.12.2015

Preparation of sample: 200 g Olivin was mixed with 1L ISO 10253 growth media and left to

shake for 24 hours. Solution was filtered through a 0.22µm filter and

adjusted from pH 8.60 to 8.04 by adding 258 µl 1M HCL.

Concentrations: 100; 32; 18; 10; 3.2 % Olivin solution with a loading rate of 200g/L

Preparation of test: 14.12.15-15.12.2015

Dilution medium: ISO 10253 (seawater collected 28.09.15, salinity: 34‰)

Replicates: 3 for each concentration. 6 control replicates.

Incubation: A temperature controlled incubator with orbital shaking (90rpm)

Test vessels: 25 ml glassvials with ca. 12 ml sample

Lighting: Measured: 2500 LUX (fluoroscent) equal to 34  $\mu$  mol m<sup>-2</sup> s<sup>-1</sup>

Temperature: 20±1°C

Inoculum: 5·106 cells/l from an exponentially growing culture

Registration of growth: Particle counting with Coulter Multisizer after 24±2, 48±2 and 72±2

hours.

Calculation of growth rate: Logarithimic increase in cell density from start to 72 hours.

Non-linear regression using Excel Macro Regtox 7.0.6, based on growth Calculation of EC<sub>X</sub> <sup>1</sup>

rate as % of control. ECx is given as % Olivin solution with a loading

rate of 200g to 1 L ISO10253 media.

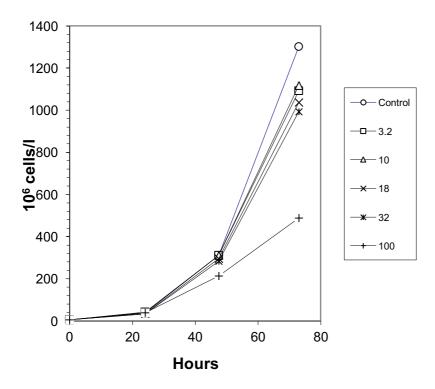
Calculation of NOEC 2 Kruskal-Wallis test, Dunn's multiple comparisons test. Calculated using

Graphpad Prism 6. NOEC is given as % Olivin solution with a loading

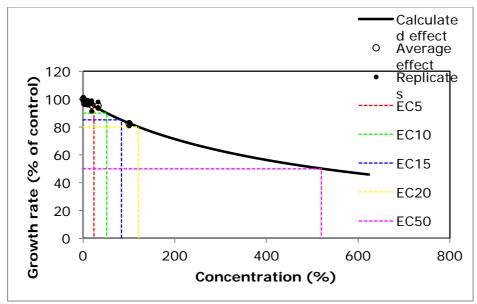
rate of 200g to 1 L ISO10253 media.

<sup>1</sup> EC<sub>x</sub>: The concentration which gives x % reduction of growth rate compared to the control

16



**Figure 1.** Change in cell density (mill cells/L) over time (hours) in all concentrations of Olivin (% olivine solution with a loading rate of 200g/L medium).



**Figure 2.** Dose-response curve of Olivin showing growth rate d<sup>-1</sup> (% of control) after 72 hours of exposure. Concentration is given in % Olivin solution where the loading rate 200g to 1L medium.

**Table 1** Effect concentrations (%) of Olivin at a loading rate of 200g to 1L medium.

<sup>&</sup>lt;sup>2</sup> NOEC: The highest tested concentration which has no significant reduction in growth compared to the control

|                                | EC <sub>50</sub> | EC <sub>10</sub>  | NOEC |
|--------------------------------|------------------|-------------------|------|
| Concentration (%)              | >100%            | 51.4% (41.3-58.2) | 32%  |
| (with 95% confidence interval) |                  |                   |      |
|                                |                  |                   |      |

Table 2 Validity criteria in ISO 10253

| Criteria                                     | Observed |
|--|----------|
| Coefficient of variation < 6% in control     | 0.8      |
| Increase from start concentration > 16 times | 260      |
| pH increase in control < 1 unit              | 0.62     |

#### **Results:**

Growth was exponential in the controls during the experiment (Figure 1), and the test met the validity criteria in ISO 10253 (Table 2). Cell numbers, growth rates and pH measurements are presented in Appendix 1. Significant reduction in growth rate was was measured at a 100% Olivin solution (loading rate of 200g to 1 L ISO 10253 growth media) (Dunn's multiple comparisons test, p<0.05). Effect concentrations (ECx) was calculated with Non-linear regression and are summerised in Table 1. Calculated EC10 at 72 hours was at 51.4% (41.3-58.2%) (Table 1). Estimated EC50 was above highest tested concentration and therefore given as >100% with a loading rate of 200g to 1 L media (Table 1). The lowest concentration with no significant effect (NOEC) was at 32% Olivin solution.

#### **Conclusion:**

At a loading rate of 200g Olivin in 1 L algal growth medium there was a significant reduction on the growth rate of *Skeletonema pseudocostatum* at 100% Olivin solution (Dunn's multiple comparisons test, p<0.05). EC10 was calculated to 51.4% (41.3-58.2%), whereas EC50 was not reached in this study. The NOEC (No Effect Concentration) was at 32% Olivin solution at the loading rate used.

| Oslo 12.01.2016 | Lene Fredriksen    |
|-----------------|--------------------|
|                 | Research assistent |

Appendix 1. Cell density, growth rate and pH measurement

| Concentration |       | 10  | <sup>6</sup> cells/L |      | Growth                 | Average                |       |     |
|---------------|-------|-----|----------------------|------|------------------------|------------------------|-------|-----|
|               |       | Da  | Da                   | Da   | rate                   | growthrate             | start | end |
|               |       | y 1 | y 2                  | y 3  | day 3, d <sup>-1</sup> | Day 3, d <sup>-1</sup> | Start | enu |
|               |       | 35  | 318                  | 1262 | 1.82                   |                        |       |     |
|               |       | 38  | 310                  | 1362 | 1.84                   |                        |       |     |
| Contro        | 0 %   | 37  | 321                  | 1244 | 1.81                   | 1.8                    | 8.22  | 8.8 |
| 1             | 0 %   | 38  | 309                  | 1286 | 1.82                   |                        | 0.22  | 4   |
|               |       | 38  | 307                  | 1263 | 1.82                   |                        |       |     |
|               |       | 39  | 297                  | 1391 | 1.85                   |                        |       |     |
|               |       | 40  | 307                  | 1112 | 1.78                   | 1.8                    |       | 8.9 |
|               | 3,2 % | 41  | 316                  | 1113 | 1.78                   | 1.0                    | 8.22  | 4   |
|               |       | 44  | 308                  | 1049 | 1.76                   |                        |       | 4   |
|               |       | 38  | 307                  | 1111 | 1.78                   | 1.8                    |       |     |
|               | 10 %  | 41  | 310                  | 1016 | 1.75                   | 1.0                    | -     | -   |
|               |       | 36  | 314                  | 1226 | 1.81                   |                        |       |     |
|               |       | 37  | 321                  | 1210 | 1.80                   | 1.7                    |       |     |
| Olivin        | 18 %  | 28  | 219                  | 801  | 1.67                   | 1.7                    | -     | -   |
|               |       | 37  | 346                  | 1098 | 1.77                   |                        |       |     |
|               |       | 32  | 275                  | 897  | 1.71                   | 1.7                    |       |     |
|               | 32 %  | 30  | 260                  | 930  | 1.72                   | 1./                    | -     | -   |
|               |       | 37  | 316                  | 1150 | 1.79                   |                        |       |     |
|               |       | 37  | 210                  | 511  | 1.52                   | 1.5                    |       | 8.4 |
|               | 100 % | 40  | 221                  | 512  | 1.52                   | 1.3                    | 8.11  | 6.4 |
|               |       | 40  | 208                  | 440  | 1.47                   |                        |       | U   |

Appendix 2. Graphpad Prism 6 Kruskal Wallis test

| Table Analyzed                           | Data 1      |
|--|-------------|
|  |             |
| Kruskal-Wallis test                      |             |
| P value                                  | 0.0065      |
| Exact or approximate P value?            | Approximate |
| P value summary                          | **          |
| Do the medians vary signif. $(P < 0.05)$ | Yes         |
| Number of groups                         | 6           |
| Kruskal-Wallis statistic                 | 16.13       |
|  |             |
| Data summary                             |             |
| Number of treatments (columns)           | 6           |
| Number of values (total)                 | 21          |

| Number of    |           |              |           |    |     |     |
|--------------|-----------|--------------|-----------|----|-----|-----|
| families     | 1         |              |           |    |     |     |
| Number of    |           |              |           |    |     |     |
| comparisons  |           |              |           |    |     |     |
| per family   | 5         |              |           |    |     |     |
| Alpha        | 0.05      |              |           |    |     |     |
| 1            |           |              |           |    |     |     |
| Dunn's       |           |              |           |    |     |     |
| multiple     |           |              |           |    |     |     |
| comparisons  | Mean rank |              |           |    |     |     |
| test         | diff.     | Significant? | Summary   |    | A-? |     |
|              | <u></u>   |              | <u>j</u>  |    |     |     |
| Control vs.  |           |              |           |    |     |     |
| 3.2          | 8.167     | No           | ns        |    | В   | 3.2 |
| Control vs.  |           |              |           |    |     |     |
| 10           | 7.833     | No           | ns        |    | C   | 10  |
| Control vs.  |           |              |           |    |     |     |
| 18           | 9.500     | No           | ns        |    | D   | 18  |
| Control vs.  |           |              |           |    |     |     |
| 32           | 10.50     | No           | ns        |    | Е   | 32  |
| Control vs.  |           |              |           |    |     |     |
| 100          | 16.50     | Yes          | ***       |    | F   | 100 |
|              |           |              |           |    |     |     |
|              |           |              |           |    |     |     |
|              | Mean rank | Mean rank    | Mean rank |    |     |     |
| Test details | 1         | 2            | diff.     | n1 | n2  |     |
|              |           |              |           |    |     |     |
| Control vs.  |           |              |           |    |     |     |
| 3.2          | 18.50     | 10.33        | 8.167     | 6  | 3   |     |
| Control vs.  |           |              |           |    |     |     |
| 10           | 18.50     | 10.67        | 7.833     | 6  | 3   |     |
| Control vs.  |           |              |           |    |     |     |
| 18           | 18.50     | 9.000        | 9.500     | 6  | 3   |     |
| Control vs.  |           |              |           |    |     |     |
| 32           | 18.50     | 8.000        | 10.50     | 6  | 3   |     |
| Control vs.  |           |              |           |    |     |     |
| 100          | 18.50     | 2.000        | 16.50     | 6  | 3   |     |



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|--|---|--|--|--|--|--|
| Customer:<br>NORTH CAPE MINERALS<br>Att: Øystein Wærnes<br>ÄHEIM | Leaching t  | REPORT  Leaching tests – initial and stabilised leaching |  |  |  |  |
| 6146 ÅHEIM   | Order no.: 29398  | Number of pages + endosures:<br>2                        |  |  |  |  |
|  | Project / Report reference<br>North Cape Minerals   |  |  |  |  |  |
| Rev. Nr.: Customer order no. / ref.:                             | Performed by:<br>T.Pedersen / W.Brennbakk   | Weiche Frankalik   |  |  |  |  |

Test material: OLIFLUX 0-3 - Olivine. Material received for testing on the 23rd of November 2007.

#### Description of task

The material was to undergo a leaching test related to the test program set forth by the Norwegian Pollutant Control Authorities. Chemical stability was thus to be established through tests determining the potential leakage from the test material when placed as a covering material at sea bed. Test method is described in the Authorities reference program TA-2143/2005, which really is a guiding test program for minerals intended to be used as covering materials for polluted sediments at sea. Shaking test according to NS EN 12457-2 and column test according to CEN EN 14405 has thus been performed. The clustes have been analysed for pH, Chromium (Cr), Copper (Cu) and Nickel (Ni).

#### Results

#### Shaking test

The results from the shaking test is shown in table 1.

NB! The results have not taken the blind sample into account, thus not corrigated.

Amount of material for the shaking test: 100 grams

Table 1: Results from analysis of shaking eluates. Results are compared to the accept values set forth by the Norwegian Pollutant Control Authorities for leaching in salt water.

| Parameter | Shaking test<br>L/S 10 | Uncertainty<br>+/- | Blind salt<br>solution | Initial leaching,<br>Class III |  |
|-----------|------------------------|--------------------|------------------------|--------------------------------|--|
| pH        | 9,9                    | 0,1                |                        |                                |  |
| Cr (ug/l) | 0,549                  | 0,189              | 0,451                  | 1,5                            |  |
| Cu (ug/I) | 0,385*                 | -                  | 0,642                  | 1,5                            |  |
| Ni (ug/l) | 0,827                  | 0,350              | <0,5                   | 5                              |  |

<sup>\*</sup> Not accredited

The analysis reveals values well below the criteria of acceptance for class III.



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#### Column test

The results from the column test are shown in tables 2 and 3.

Amount of material for column testing:

I 110 grams

Table 2: Results from analysis of the column cluates. The results are compared to the accept values set forth by the Norwegian Pollutant Control Authorities for leaching in salt water.

(Not corrected for value of blind)

| Parameter | Column test<br>L/S 10,0 | Uncertainty<br>+/- | Blind salt<br>solution | Stabilised leaching,<br>Class II |
|-----------|-------------------------|--------------------|------------------------|----------------------------------|
| pH        | 10,0                    | 0,1                |                        |                                  |
| Cr (ug/l) | 0,427                   | 0,190              | 0,451                  | 0,5                              |
| Cu (ug/l) | 0,913                   | 0,331              | 0,642                  | 0,7                              |
| Ni (ug/l) | <0,5                    | -                  | <0,5                   | 2                                |

Corrected for the values of the blinds, the analytical values are far below the acceptance criteria of Class II.

Table 3: Results from analysis of the column eluates, all 7 fractions (Not corrected for value of blind)

| Parameter | L/S 0,1 | L/S 0,2 | L/S 0,5 | L/S 1,0 | L/S 2,0 | L/S 5,0 | L/S 10,0 | Blind salt<br>solution |  |
|-----------|---------|---------|---------|---------|---------|---------|----------|------------------------|--|
| pH        | 9,5     | 9,5     | 9,7     | 9,5     | 9,3     | 8,1     | 10,0     |                        |  |
| Cr (ug/i) | 0,414   | 0,530   | 0,496   | 0,645   | 1,02    | 0,626   | 0,427    | 0,451                  |  |
| Cu (ug/l) | 36,6    | 9,89    | 4,37    | 3,17    | 2,46    | 1,58    | 0,913    | 0,642                  |  |
| Ni (ug/l) | 5,05    | 1,95    | 0,773   | 0,555   | 1,01    | <0,5    | <0,5     | <0,5                   |  |

The results show a steady decrease in the amount of copper during the column test. The same is valid for the amount of Nickel, except from the single fraction of L/S 2,0, which shows a slight increase. Chromium values is quite stable all through the column test.

It is important to note that the analysis have slight uncertainties and that the analytical values are not corrected for the value of the blind solution.

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