Norwegian University of Life Sciences
Faculty of Environmental Science and Technology
Department of Environmental Sciences (IMV)

Master Thesis 2015
60 credits

Immobilization of Pb and Sb in Shooting Range Soil. Column Experiment with Fe-based Sorbent

Kristin Frøsland

Contents

Preface

This master thesis has been the end of my journey and education at The Norwegian University of Life Sciences (NMBU). The master thesis constitutes 60 ects. and is a part of the master degree of Environmental Science conducted the fall of 2014 and spring 2015. The experimental work was done at Bioforsk in Ås and at the Institute of Environmental Science at NMBU.

The work in this thesis was a cooperation between the Norwegian Geotechnical Institute (NGI) and Lindum AS. The work was a part of a bigger pilot project by Lindum AS.

The in situ sampling was done by Lindum AS. The ICP-MS analyses were done by lab engineer Solfrid Lohne, other analysis were executed by lab engineer Oddny Gimmingsrud at the Institute of Environmental Science and the author. I would like to express my gratitude for the laboratory staff who helped me.

I would like to thank my main supervisor Dr. Åsgeir Rossebø Almås at NMBU for guiding me through this year, discussing problems along the way and for being very patient throughout the whole process. I would also like to thank my co-supervisor Gudny Okkenhaug at NGI for help during the year.

I have the most gratitude towards my family, my friends and especially my partner Emil for much support during this year.

12.05.15, Ås
Kristin Frøsland

Dictionary

Adsorption	The binding of a compound to the surface of a particle.
DOC	Dissolved organic carbon with a size fraction of under 0.45µm.
TOC	Total organic carbon.
ICP-MS	Inductive Coupled Plasma Mass Spectronomy. Apparatus used for detecting metals and non-metals at low concentrations.
Desorption	The release of a previously bound substance to a particle.
Pb_w	Concentration of lead (Pb) in water solution
Sb_w	Concentration of antimony (Sb) in water solution

Abstract

Tons of trace metals such as lead, antimony, copper and zink, get deposited every year in Norwegian shooting ranges (Strømseng et al. 2011). Due to the accumulation and use of bullets and ammunition, these shooting ranges represent a potensially big environmental hazard. Shooting range soil is most often contaminated with trace metals such as lead (Pb) and antimony (Sb). A way of immobilizing the trace metals in this kind of contaminated soil is to add an iron-based sorbent material.

The main aim of this thesis was to see if an industrial waste iron-based sorbent could immobilize Pb and Sb from contaminated shooting-range soil. Two types of soil were collected from two different shooting-tracks in Tittelsnes shooting range in Norway; mineral and organic soil. Column extractions were used, and the iron-sorbent was mixed into the soil at 5 and 10 % volume, as well as added in layers of 5 %, 10 % and two times 5 %. The water extracts from the columns showed that the sorbent had a good remediation effect on Pb and Sb in both soils. Treatments with the sorbent mixed together with the soil reduced the leaching of the trace metals to some extent, but the concentration of Pb and Sb was most effectively kept back in treatments with sorbent added in a bottom layer. The treatment with sorbent was the most important parameter that controlled the leaching of the trace metals, but the sorbent also affected the geochemistry of other parameters in the lachates. As a result of treatment, the leaching of Pb and Sb were well correlated with the variation in pH, DOC and SO_4^{2-}.

Another aim was to see if the sorbent would immobilize the trace metals over time. The total time of the study was 5 weeks, and water extractions were taken during the study. Statistics done showed that immobilization was effective at 5 weeks. Further studies are needed to know the long-term effect of stabilization of shooting range soil by an iron sorbent.

.

Sammendrag

Flere tonn bly, antimon, kopper og zink blir årlig avsatt i norske skytefelt (Strømseng et al. 2011). Bruken og akkumuleringen av skytevåpenammunisjon har ført til at disse skytefeltene representerer en potensielt stor miljøfare. Skytefeltsjord er ofte kontaminert med spormetaller som bly (Pb) og antimony (Sb). En etablert metode for å immobilisere spormetallene i denne typen forurenset jord, er å tilsette et jernholdig sorbentmateriale.

Hovedmålet med dette studiet var å se om et jernbasert industrielt avfallsprodukt kunne immobilisere bly (Pb) og antimony (Sb) fra kontaminert skytefeltsjord. To typer jord ble samlet inn fra to ulike skytebaner i Tittelsnes skytefelt i Norge; mineral- og organisk jord. Et kolonneforsøk ble utført, og den jernbaserte sorbenten ble blandet inn i jord ved 5 og 10 % volum, samt i lag ved 5 %, 10 % og to lag med 5 %. Vannprøvene fra kolonnene viste at sorbenten hadde en god remedieringseffekt på både Pb og Sb i begge jordtyper. Behandlinger hvor sorbenten var blandet inn med jorda reduserte utlekkingen, men konsentrasjonene av Pb og Sb ble mest effektivt holdt tilbake av behandlingene med sorbent tilsatt lagvis. Behandling var den viktigste faktoren som kontrollerte utlekking av spormetallene, men sorbenten påvirket også geokjemien til andre parametre i vannprøvene. Som et resultat av behandling, korrelerte variasjoner i pH, DOC og SO_4^{2-} bra med utlekkingen av Pb og Sb.

Et annet mål ved studien var å se om sorbenten immobiliserte spormetallene over tid. Den totale tiden for forsøket var fem uker, og vannprøver ble tatt underveis i forsøket. Statistikken kjørt på dataen fra forsøket viste at immobiliseringen var effektiv etter 5 uker i begge jordtyper. Videre studier trengs for å vite langtidseffekten av stabilisering av skytefeltsjord med jernsorbent.

1. Introduction
Background for the study

1.1 Contamination at shooting ranges
Because of the use of handgun ammunition, several tons of heavy metals and metalloids are deposited at shooting ranges every year. Projectiles used in past years contained mainly Pb, Cu, Zn and Sb. The core consisted of a Pb-Sb alloy, surrounded by a jacket of zinc and copper (Strømseng et al. 2011). The use of these has halved from 2007 to 2012 because of the transition to lead free ammunition with an iron core. Despite of this, Pb-containing ammunition is still being used, and it was reported that approximately 26 tons Pb, 86 tons copper, 3 tons Sb and 8 tons zinc coming from spent bullets by the Norwegian armed forces were deposited in Norwegian shooting ranges in 2012 (Reistad et al. 2013).

Over the years, spent bullets accumulate on the shooting-ranges and in areas close by. Because these bullets corrode and release metals, they can become a large point source pollution and hazard to the environment; plants, microorganisms and water. The bullets mainly corrode on the surface. This surface is released into the soil either by dissolution or physical wearing of the surface. Also, when the bullets hit the ground, they might be fragmented. That leaves a bigger corrosion surface. This can also happen when a new bullet hits a spent bullet on the ground (Heier et al. 2010).

1.2 Characterization of relevant trace elements
The release of metals into soil solution depends on conditions like the pH, content of organic matter, soil texture and particle fraction, water balance and reducing conditions (Herzel 2012). The impact of these soil parameters on Pb and Sb solubility will be discussed individually as Pb is a cation and Sb behave as an anion in soil. Metals also vary in size, charge and density, which influence the biological uptake and the transport of these elements. Metals can be in the form of free ions, bound to inorganic complexes or large organic molecules, or be in a different oxidation level, and this created various possibilities for a metal to be of a certain 'chemical species'. A change in any of the parameters above can lead to a change in the metal speciation.(Nordberg et al. 2004; Paquin et al. 2002) states that: "(…) metal toxicity was poorly correlated with total metal concentrations", meaning that uptake and transport of

metals depend on the speciation of the metal. The LMM – low molecular mass species – are now assumed more bioavailable than metals bound to particles (Paquin et al. 2002).

The clean-up of Norwegian shooting-ranges has been getting an increased amount of attention during the last decade. To gain knowledge of how to manage and assess the impacts heavy metals and metalloids can have on the environment, it is important to have knowledge of the relevant metals and their speciation. The focus of this master thesis has been on two of the most important pollutants from shooting ranges - Pb and Sb.

1.3 Adsorption

In adding iron-based sorbents the main goal is to create a bonding between the sorbate (which is the substance bound to the sorbent) and the sorbent added (Blume et al. 2010; Fränzle et al. 2012). The bond between the sorbate and the surface of the sorbent can be of several types. Specific adsorption is a process where the sorbate creates a bonding with reactive surface functional groups on the sorbent. Such a bond can be an outer-sphere complex or an inner-sphere complex. An outer-spere complex consists of an electrostatic bond to the sorbent, and a water molecule exists between the sorbate and the functional group on the surface of the sorbent. An inner-sphere complex is either an ionic or a covalent bond between the sorbent and sorbate, and this creates a stronger bond than outer-sphere complexes (Blume et al. 2010; Bradl 2004).

1.4 Reducing conditions (in shooting-range soils)

Soils undergo changes in redox (reducing and oxidation) conditions. Microorganisms effectively catalyzes redox reactions, which are usually slow reactions without any microbial mediation. Reduction can change the effect of iron-based remediation processes. Changes in redox conditions alter the stability of iron hydroxides in soil. Fe^{3+} ions are reduced to Fe^{2+}, releasing metals into the soil solution (Alloway 1995).

It is under reducing conditions that Sb can be reduced from its pentavalent form to its trivalent form. Even though this chemical form of Sb more easily adsorbs to adsorption surfaces, it is considered more toxic than its pentavalent form. (Krachler et al. 2001; Nordberg et al. 2014) Pb is prone to have an increase in mobility under reducing conditions (Charlatchka & Cambier 2000).

1.5 Influence on sorption by DOC, inorganic anions and pH

Trace metals are not readily available to organisms in soil because they are bound to solid soil fractions, either inorganic or organic. This is not a locked state. Changes in some parameters in soil can make the bound metals react. The geochemistry of the trace metals depend on several factors. The most important factor in soil controlling the porewater concentration of a trace metal, is the total concentration of the metal. After that, the amount of organic matter in the soil, the pH and possible binding agents control the concentration of the trace metals.

The redox conditions play a part directly on oxyanion speciation, but for both oxyanions and metal cations, the redox conditions can alter the binding properties of for instance Fe-oxides by shifts in redox state of Fe from Fe(III) to Fe(II). When Fe(III) is reduced to Fe(II), the binding capacity for Sb will be reduced.

DOC is often defined as the concentration of dissolved organic carbon with a size under 0.45 µm. Adsorption by DOC has been shown to increase with increasing soil profile depth (Jardine et al. 1989). The importance of anions in the pore water on the concentration of trace metals is variable, but organic matter in soil may be the most important factor. This is because dissolved organic matter binds trace metals in soil very well. pH can also affect the adsorption by DOC, with the adsorption being the most effective at around pH 4,5. (Jardine et al. 1989)

1.6 Lead (Pb)

Lead is an element often classified as a heavy metal (specific density higher than 5 g cm^3.) It is not an essential element for living organisms and plants, and has no known beneficial effects or purposes. Compared to other soil pollutants, Pb has a long residence time in soil. This is due to its low solubility in soils and sediments, and its strong affinity for particularly organic material and oxides.

Lead is mostly represented in two different oxidation states; Pb^{4+} and Pb^{2+}, the most common species being Pb^{2+} (Davies 1995). Since Pb usually acts as a cation in the soil (Strømseng et al. 2011) the cations can be adsorbed by negatively charged clay minerals in the soil. The degree of adsorption of Pb depends on the soil pH and the adsorption to the surfaces of different mineral oxides (such as iron- and manganese oxides) (Darling & Thomas 2003). Soil organic matter is also a factor that plays an important part in the retention of Pb in the soil (Vega et al. 2006). The solubility of Pb is shown to decrease when the pH value in the soil increases, and increase when the pH value decreases (Santillan-Medrano & Jurinak 1975). This allows soils to act as both a sink and a source of Pb (Mielke et al. 2007). In soils with a low pH value, Pb can form large complex molecules with organic matter. This can increase the mobility of the metal.

1.7 Antimony (Sb)

Antimony is a non-essential substance for both organisms and plants, and is toxic in low concentrations for humans. It has also been assumed to be a carcinogenic substance, while it is not as toxic to plants. (Okkenhaug et al. 2011) Because of its toxicity, The US Environment Protection Agency has Sb and compounds containing Sb on their priority list of pollutants (USEPA 2013).

Antimony is a metalloid that exists in different oxidation states. In nature it is usually found as antimonate $[Sb(OH)_6]^-$ under oxidizing conditions or antimonite $Sb(OH)_3$ under reducing conditions (Okkenhaug et al. 2011; Strømseng et al. 2011). Antimonate is the form that dominates the soil solution at shooting ranges (Johnson et al. 2005). The trivalent species is known to be ten times more toxic than the pentavalent species of antimony (Gebel 1997; Krachler et al. 2001). Under a high pH Sb will destabilize, therefore it is better to keep the pH value low for it to remain immobile (Filella et al. 2002). The mobility in soil is low, due to sorption by humic acid in organic material and mineral hydroxides such as manganese, iron and aluminum oxides. (Mitsunobu et al. 2008) Due to the low natural background concentrations, as well as the low solubility in water, Sb has not been one of the most studied environmental pollutants. Thus, the data collected and the information available has previously been scarce (Filella et al. 2002).

1.8 Classification of conditions for contaminated soil

The Norwegian Pollution Control Authority (MD in Norwegian) has developed a classification system for the different conditions of contaminated soil (as seen in Table 1). The system is based on considering the health risk of being in the area where the soil is, and the danger of the contaminant being transported. The classifications were made to make the work on evaluating cases related to contaminated soil more manageable for the pollution authorities. The classes express the health risk of a specific soils content of environmental toxins and substances, and range from class 1 through 5. Amounts of substances exceeding class 5 is labelled hazardous waste. Normal values are considered to be within the classes 1 and 2 (Hansen & Danielsberg 2009).

The classification of condition for an area is determined by measuring the level of contamination in several soil samples. The different concentrations for Pb, copper (Cu) and zinc (Zn) are given in Table 1. The concentrations for Sb are taken from a report by the Norwegian Defence Research Establishment (Voie et al. 2010).

Table 1. Overview of classification of conditions for contaminated soil. Modified from a report by the Norwegian Pollution Control Authority (Hansen & Danielsberg 2009). Concentrations for Sb (grey color) are taken from a report by the FFI (Voie et al. 2010). Concentrations are given in mg kg^{-1} dry soil material.

Condition/class	**1**	**2**	**3**	**4**	**5**
Description of condition	**Very good**	**Good**	**Moderate**	**Poor**	**Very poor**
Pb	<60	60-100	100-300	300-700	700-2500
Cu	<100	100-200	200-1000	1000-8500	8500-25000
Zn	<200	200-500	500-1000	1000-5000	5000-25000
Sb	<40	40-100	100-300	300-700	700-10000

1.9 Waste Regulations

The Waste Regulation chapter 9 regulates the deposit of polluted soil and sediment in Norway. In the Waste Regulation it says its purpose is to:" […] *ensure that waste is landfilled in a sound and controlled manner such that adverse effects on the environment and human health are prevented or reduced as far as possible*" (Lovdata 1983). Each landfill has to belong to one of the three different categories for landfills:

- Class 1: Hazardous waste
- Class 2: Non-hazardous waste
- Class 3: Inert waste

Landfills in class 2 of non-hazardous waste can be used for both non-hazardous waste and hazardous waste that has been stabilized. Leaching from the waste then have to be tested and has to be equivalent to the leaching from non-hazardous waste. The leaching from all landfills must also be under the limits shown in Table 2.

Table 2. Limits for leaching of selected parameters from landfills of hazardous waste, non-hazardous waste and inert waste. (Lovdata 1983)

Parameter	**Hazardous waste**		**Non-hazardous waste**		**Inert waste**	
	Batch test* [mg kg^{-1}]	Column test ** [mg L^{-1}]	Batch test* [mg kg^{-1}]	Column test ** [mg L^{-1}]	Batch test* [mg kg^{-1}]	Column test ** [mg L^{-1}]
Lead (Pb)	50	15	10	3	0.5	0.15
Antimony (Sb)	5	1	0.7	0.15	0.06	0.1
Copper (Cu)	100	60	50	30	2	0.6
Zinc (Zn)	50	60	50	15	4	1.2
Chloride	25000	15000	15000	8500	800	460
Sulphate	50000	17000	20000	7000	1000	1500
DOC	1000	320	800	250	500	160

*For column tests, the values are based on concentrations at L/S 0.1 L kg^{-1}. **For batch tests, values are based on concentrations at L/S kg^{-1}.

1.10 Remediation and treatment of contaminated soil

Applying different remediation practices can change the speciation of contaminants such that they become less prone to transportation. Thus posing a smaller threat to human health and vegetation. In the establishment of new shooting ranges, or maintaining the ones already in use, it is most desirable to take measures that can limit the risk of trace metals transported by water runoff. One way is to mix the soil with adsorbing materials inhibits the leaching and bioavailability of heavy metals and metalloids (Strømseng et al. 2011). Remediation of soil can be conducted *in situ*; remediating contaminated soil where it is located, or *ex situ*; removing soil to treat it and then bring it back to its original spot or to a landfill. Trace metals like Pb and Sb can be a hazard even at low concentrations in soil (approximately 1-500 mg kg^{-1} in solid soil) (O'Day & Vlassopoulos 2010). At shooting ranges the trace metals will be spread at low concentrations as a diffuse contamination, making it hard to remediate the soil or remove it. Any attempt to stabilize the contaminated soil has to be cost-efficient and have a noticeable effect (O'Day & Vlassopoulos 2010).

1.11 Retention of lead and antimony by minerals

Antimony and Pb can be retained in soil (Flynn et al. 2003). Retention reduces the mobility and transport of trace metals from soils into the water. Adsorption to oxides and hydroxides has shown to be important to Sb as well as Pb, and the addition of these substances is a widely studied method for immobilization. Antimony is most often present as oxyanions and Pb as cations in soil. They therefore adsorb differently to minerals. Adding zero-valent iron, iron hydroxides and oxides has proven to reduce leaching of both Sb and Pb (Manaka 2006; Okkenhaug et al. 2013; Sipos et al. 2008; Strømseng et al. 2011). Iron-based sorbent materials usually have a low cost, are easy to synthesize and represent a low risk of additional pollution to the system it is used in (Deliyanni et al. 2009).

1.12 Remediation effect in the study

The purpose of column experiments, in this context with added sorbent, is to create a model system to conduct a detailed study on the immobilization of trace metals by adsorption. It is important to not only evaluate the immobilization of the sorbent, but also to study how the system acts over time. This is to investigate the persistence of the initial stabilization. The overall goal is for the soil to retain the trace elements in their immobile form, and hence pose insignificant threat to the surrounding environment. Different approaches can be taken to evaluate the stabilization, such as using extraction tests and leaching tests over a long period of time (Gleyzes et al. 2002; Lambrechts et al. 2011).

1.13 Motivation for study

The purpose of this study is to add an iron-based sorbent to mineral and organic soil from a shooting range to try and immobilize lead and antimony. The hypothesis were:

I. The sorbent will immobilize lead and antimony in both mineral and organic shooting-range soil
II. Time (here: 45 days) will not influence the stabilization effect of the sorbent under field capacity conditions

III. Repeated extractions and washing will not influence the stabilization effect of the sorbent.

To investigate these hypotheses the sorbent was added in different amounts and mixes to both soils in a column experiment. Time of the whole experiment was set to five weeks, in order to see if the amounts of lead and antimony would change in the leachates. To reduce the adsorption capacity of the iron, the soil in the columns were kept at field capacity to simulate moisture conditions in field (without it being over saturated). This will provide test results to conclude whether hypothesis II can be verified or not.

2. Materials and methods

2.1 Tittelsnes shooting range (Norway)

Tittelsnes shooting range is located by an old German fort at the Tittelsnes peninsula in the Bømlo area, in the county of Hordaland on the southwestern coast of Norway. An overview of the two shooting tracks in the shooting range is shown in figure 1. It is 62 decars large and the area consists of both forest and wetland (Garmo 2015). The shooting range was created in the early 1950s, and used by the FORSVARET until 2005, when it was closed due to lack of safety/security (Forsvarsbygg 2013).

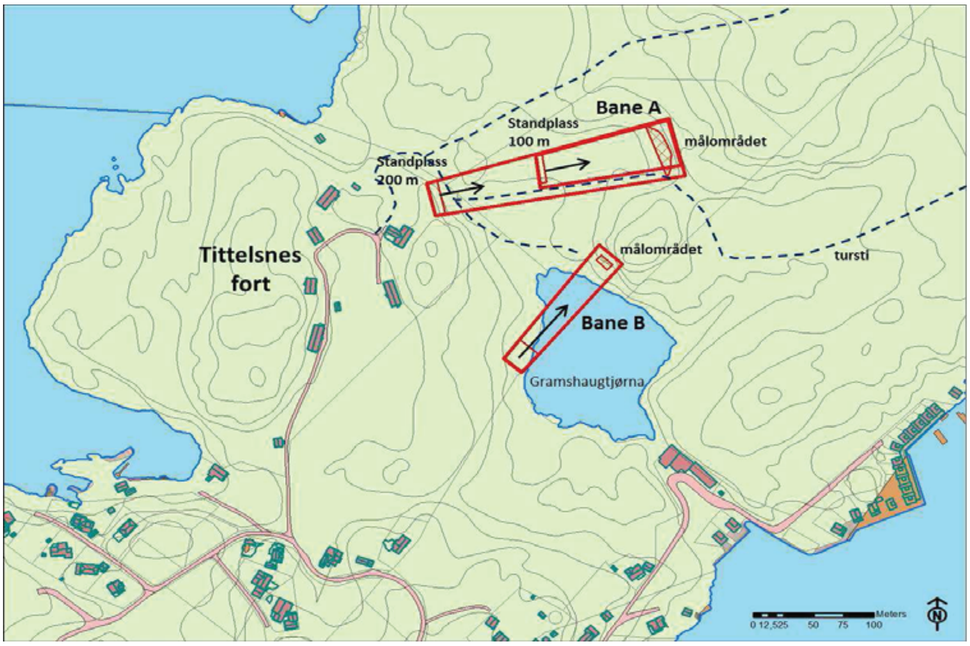


Figure 1. Map over the two SKYTEBANER in Tittelsnes SKYTEFELT. (Forsvarsbygg 2013) Arrows indicate the target area of the ammunition.

2.2 Soil sampling and characterization

The soil used in this study came from Tittelsnes shooting range (see chapter 4.1.2). Several tons of shooting range soil were collected by Lindum Gjenvinning AS and stored under a roof. The soil had then been exposed to air, but not water. The goal of the soil sampling was to get contaminated soil from two different soil types in the shooting range with different levels of TOC:

I. 'Organic' soil – Soil from a wetland area at the shooting range with a high content of organic material
II. 'Mineral' soil – Collected at a part of the shooting range in a cleared forest landscape with a lower content of organic material and a higher content of clay particles

Table 3 shows different qualities of the soils used in the study.

Table 3. Different qualities (pH, TOC, dry matter, LOI) of the two soil types used in the study.

Soil type	**pH**	**TOC [%]**	**Dry matter [%]**	**LOI [%]**
Organic	4,3	18	20	16
Mineral	7,7	5	83	77

Shooting-range soil can contain «hot-spots» with elevated concentrations of trace metals. To make sure the soil samples were homogenized, they were taken from different places in the soil masses. The soil was mixed both manually as well as with a concrete mixer within each soil type (as depicted in figure 2). Approximately 80 L of soil were collected from each soil type and put into buckets with a solid plastic bag separating it from the bucket walls. The buckets were as airtight as possible with a lid, and the samples were transported from the landfill at Lindum AS to the lysimeter lab at Bioforsk in Ås. The buckets were then stored at 4 ºC for approximately two weeks between time of sampling and the start of the experiment.

Figure 2. Sampling and mixing of soil at Lindum Gjenvinning AS. (Photo source: Kristin Frøsland.)

2.3 The sorbent

Based on its availability and low cost, an iron-oxide containing industrial waste was chosen as the sorbent material for the column experiment. The sorbent chosen was from the company Tinfor Titan & Iron AS based in Tyssedal, Norway. It is a partly crushed and sintered waste product created under the melting process when creating titanium and iron, and consists mostly of oxides of iron, calcium, titanium, zink, aluminium and sulfur. It has a high pH of around pH 11. It is considered insoluble in water, and has a relative density of 1.50 – 1.86 g cm^{-3}. To ensure the particles used had a sufficient size for adsorption, the sorbent was manually sifted through a 6.7 mm sift. Table 4 shows the content of various elements in the sorbent material. The content of the metals were assumed the same after sifting as before.

Table 4. Content of various elements in the sorbent material used in the experiment.

Element	**Content in the sorbent, [mg kg^{-1}] dry material**
Arsenic (As)	17
Lead (Pb)	40
Cadmium (Cd)	<0.3
Copper (Cu)	30
Chromium (Cr)	60
Mercury (Hg)	0.06
Nickel (Ni)	60
Zinc (Zn)	930
Cobolt (Co)	40
Molybdene (Mo)	<1
Antimony (Sb)	<50
Selenium (Se)	<10
Tin (Sn)	20
Barium (Ba)	40
Vanadium (V)	160
Iron (Fe)[a]	96[b]

[a] Values for iron is taken from (Okkenhaug & Breedveld 2014). [b] The unit for the value is [g kg^{-1}].

2.4 Column experiment

38 cylinders were used; 18 of which were 100cm in depth and with a diameter of 10cm, and 20 of which were 50cm in depth and with a diameter of 10cm. There were triplicates for each treatment (except for the two test columns that contained a mix of the two soil types), but only the first column of the triplicates were in a 100cm column. Figure 3 shows the lysimeter laboratory.

Figure 3. Column experiment setup in the lysimeter lab. (Photo souce: Kristin Frøsland.)

Soil and sorbent were put in the columns both in layers and by homogenously mixing by hand in a large container. Soil was added to approximately 47 cm height, leaving 3 cm and 53 cm space at the top on the 50cm and 100cm columns. Table 6 shows the setup of the columns in the lysimeter lab.

Table 6. Setup of different treatments for each soil type. The different treatments also had triplicates.

1. Organic soil	2. Mineral soil
A. Reference (without sorbent)	A. Reference (without sorbent)
B. 5% added sorbent (mixed in)	B. 5% added sorbent (mixed in)
C. 10% added sorbent (mixed in)	C. 10% added sorbent (mixed in)
D. 5% bottom layer of sorbent	D. 5% bottom layer of sorbent
E. 10% bottom layer of sorbent	E. 10% bottom layer of sorbent
F. 5% bottom layer and 5% mid layer of sorbent	F. 5% bottom layer and 5% mid layer of sorbent

The temperature was kept at normal room temperature for the reason that temperature would not influence the experiment at a noticeable level. Whatman 40 ashless 9,0cm filter papers were put on top of the columns to spread the water evenly. Schleicher & Schuell 589^2 Weissband Rundfilter aschefrei 125mm filter papers were put under the columns to prevent smaller particles (especially finer particles from the sorbent) going through. The sorbent was sifted through a 6.7 mm sift before it was used in the experiment.

Deionized water was added to each column by hand by slowly adding 100mL at a time on each column until a calculated amount of water (from 470 to 1350 mL) had been added, to obtain field capacity. The columns were left overnight for equilibration to occur. This is to test the effect of moisture conditions on the sorbent capacity. The next day, the same amount of water was added to replace the water already in the pores of the soil. After a pause of approximately two hours, water was extracted from the containers below the columns. During the experiment, this procedure was repeated three times for 24 hours equilibration time, and once after one week equilibration time, and finally after five weeks.

The water went into sterile bottles under the columns. The leachates sampled were filtered at 0.45 μm using 60mL Lurlock syringes with a polyether sulfone membrane filter (VWR). Samples were collected in 15mL plastic tubes with a red screw cork. The water samples were conserved with 2.5% concentrated saltpeter acid and stored at 4 ºC before Inductive Coupled Plasma Mass Spectroscopy (ICP-MS) analysis.

2.5 Water sample analysis

2.5.1 pH

pH was measured manually on an Orion SA 720 pH meter. Calibration of the pH meter was done 6 times using standard solutions with pH 4 and pH 7.

2.5.2 DOC

Since all samples were filtered through a 0.45 μm filter (vwr.com) before analysis, the TOC results were interpreted as DOC. DOC was measured on a Shimadzu TOC-V_{CPN} carbon analyzer.

2.5.3 Inorganic anions

The inorganic anions analyzed consisted of chloride (Cl^-), nitrate (NO_3^-) and sulfate (SO_4^{2-}). The samples were measured by a Lachat IC 5000 ion chromatograph.

2.5.4 Trace metals

After being conserved by 10 % (final concentration) concentrated Ultra Pure HNO_3 acid an internal standard was added to the samples going to the trace metal analysis. The apparatus used was ICP-MS (Inductive Coupled Plasma Mass Spectroscopy). ICP-MS separates the atoms by mass and charge. This method does not differ between the speciation of the compounds measured, thus it only determines the total amount of the analyte in the samples. (Skoog et al. 2007) The trace metals measured were lead (Pb), iron (Fe) and antimony (Sb) amongst others. The focus in this study was on Pb and Sb.

2.6 Treatment of soil samples before testing

The columns in the experiment were filled with mineral, organic and a mixture of both solid (see Table 6), and therefore both of the soil types and the mixture had to be dried to see the amount of dry bulk material in the soil. This was to calculate the pore volume and the amount of water that needed to be added to fill the pore void and achieve field capacity the soils. From the soil added for each type, it was calculated that approximately 70g organic soil, 110g mineral soil and 100g mixed soil was needed. The amounts were put in three small containers, and dried at 105 degrees C for 48 hours. The samples were then measured and the pore water was calculated.

2.7 Statistical analysis

The raw data was processed in a statistical computer software JMP (SAS Institute, Inc., 2013). This was done to process data, find possible correlations and parameters that were linked to each other. The tests are reported being significant when $\alpha < 0,05$.

3. Results

3.1 Lead and antimony

Figure 4 shows the effect of the different treatments on mineral soil with respect to the concentrations of Pb and Sb in leachates over the time of the study. Comparing the results of the different treatments to the results of the untreated columns, the sorbent has had a significant effect (t-test) for retaining both Pb and Sb. Over time the concentration of Pb seems to be stable or decline a little. The concentration of Sb remain immobilized until the extraction taken in day 45 (after 5 weeks), where it has a tendency of increasing. The three treatments with a bottom layer of sorbent was the most effective treatment for immobilizing both trace elements.

Figure 5 shows the effect of the different treatments on organic soil with respect to the concentrations of Pb and Sb in the water leachates over the time of the study. There is a significant difference between the untreated soil and the treated soil (t-test). The concentration of Pb in the leachate from the treated soil is between 64 and 176 µg/L for the untreated soil, while the concentration stays well under 15 µg/L for all the different treatments with added sorbent.

For Sb in organic soil, the concentrations in the leachate varies between a maximum of around 35 µg/L in untreated soil to a maximum of approximately 13 µg/L in the leachate from the treated soil.

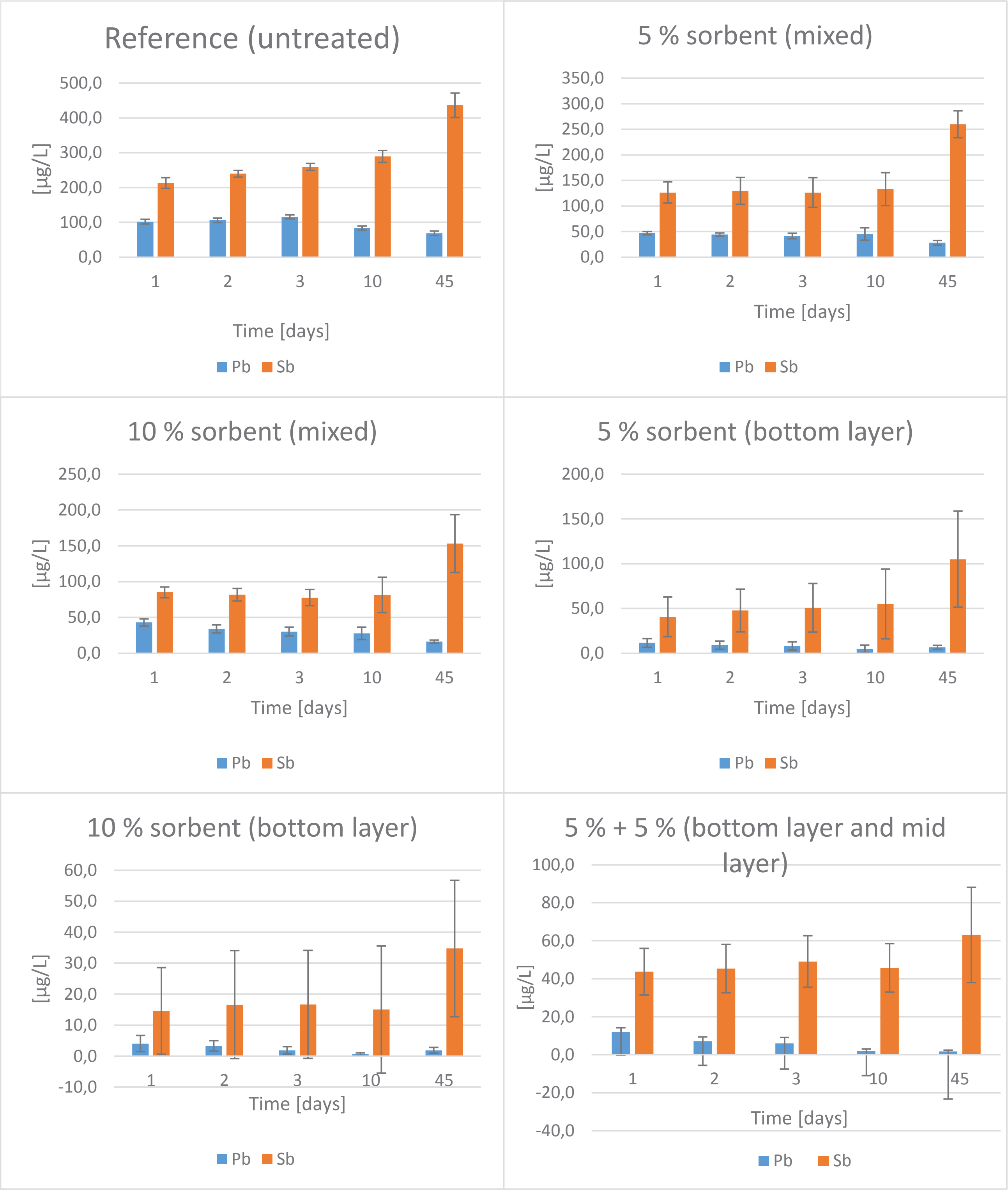


Figure 4. The effect of different sorbent additions (treatments) on the concentrations of Pb_w and Sb_w in mineral shooting-range soil leachates. Note the differencse in scales on the y-axis.

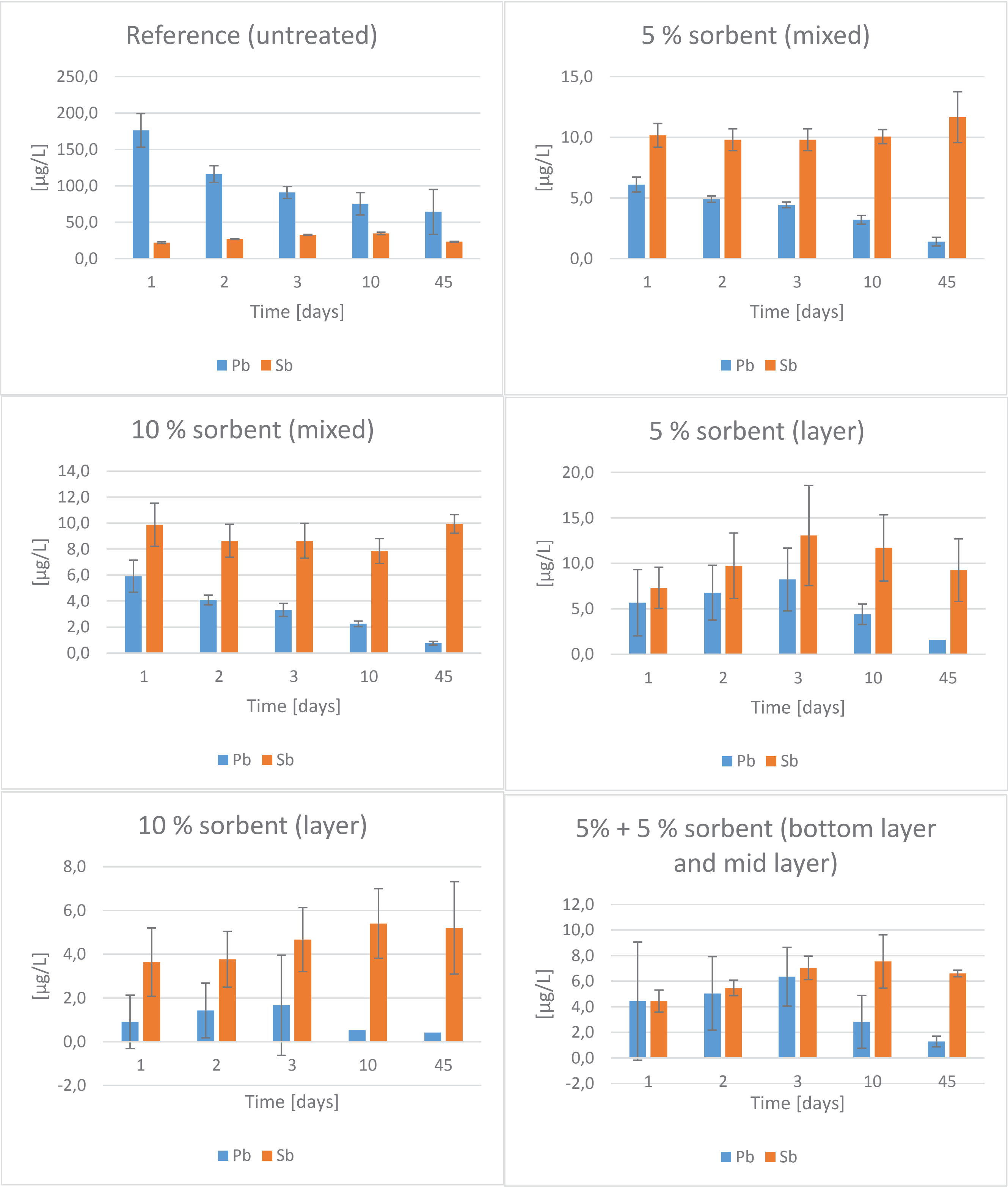


Figure 5. The effect of different sorbent additions (treatments) on the concentrations of Pb_w and Sb_w in organic shooting-range soil leachates. Note the difference in scales on the y-axis. There is no standard deviation for the Pb_w values in the treatments with 10 % sorbent (layer) and 5 % + 5 % (bottom and mid layer), due to the reason that the author chose to not use one replicate. The replicate had a value below the reference blanc measured (<0,29 µg/L).

3.2 Iron (Fe)

The concentrations of Fe were measured in the leachates, and the results for the mineral soil can be seen in figure 6. The trend seems to be that the elevated concentrations are found in the leachates from treatments with the highest perentage of the Fe-based sorbent. The leachates from the reference columns contained the least amount of iron. The standard deviations are high in some of the treatments because there were big gaps between the triplicates analysed.

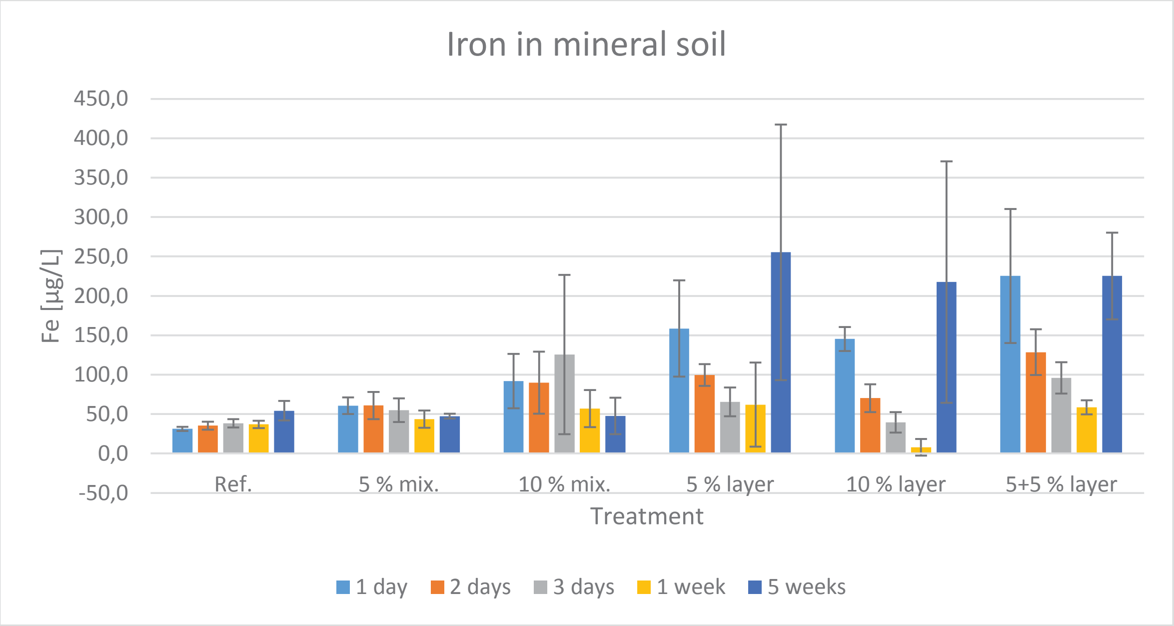


Figure 6. The levels of Fe_w in leachates from mineral soil exposed to different treatments with the sorbent.

Figure 7 shows the Fe measured in the water leachates from the organic soil. The leachates from the organic soil generally contained a higher concentration of Fe.

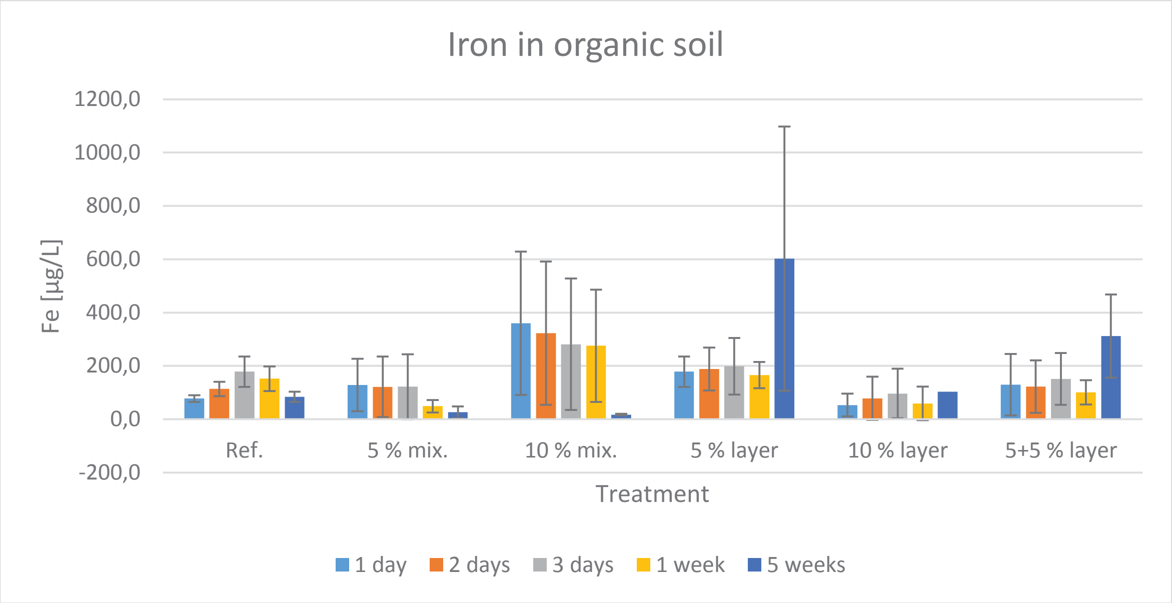


Figure 7. The concentrations of Fe_W in organic soil exposed to different treatments with the sorbent. There is no standard deviation on the treatment with 10 % layer after 5 weeks, due to the use of duplicates and not triplicates. One of the replicates were very high for an unknown reason, so the author chose to remove it and not have a standard deviation.

3.3 pH, DOC and inorganic anions

3.3.1 pH

Figure 8 and Figure 9 shows pH measured in the water leachates from the different treatments over the 45 days of study.

In leachates from the mineral soil the change in pH over time between the treatments with added sorbent and the reference columns are similar. The pH value were stable in the interval of a minimum of 6,8 (in the treatment with a 10 % added bottom layer of sorbent) and to a maximum of 7,6 (in the first four treatments). The introduction of the sorbent effectively raised the pH (t-test), but the difference between the treatments (control soil excluded in test) were not significant.

In leachates from the organic soil the pH values are the lowest in the reference columns throughout the time of the study (Figure 9). As known, the sorbent had a pH of approximately 11, while the pH of the organic soil is around 4,3. The columns with added stayed at around a pH of 7.

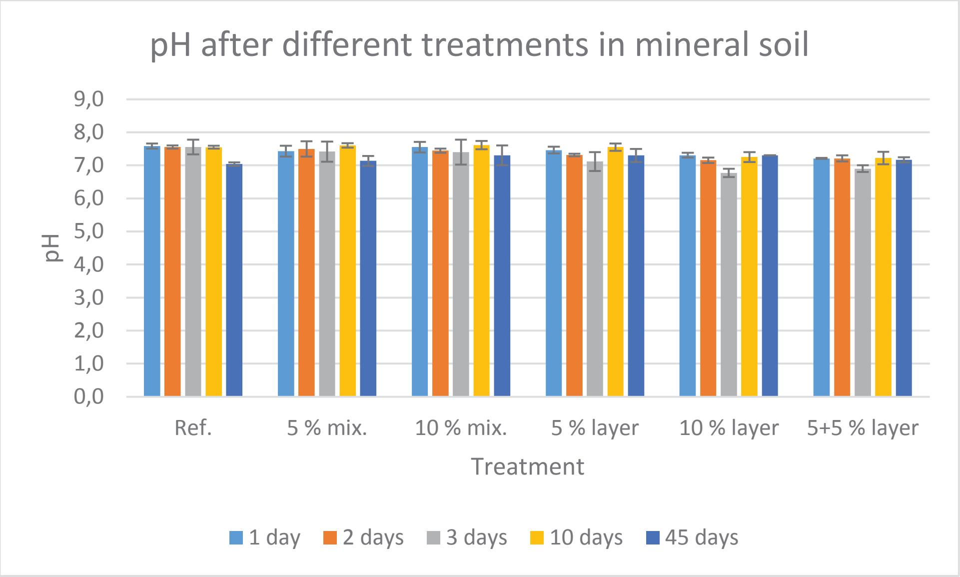


Figure 8. pH measured in water extracts from the columns with differently added sorbent in mineral soil. The time between extractions going from 1 day to 45 days.

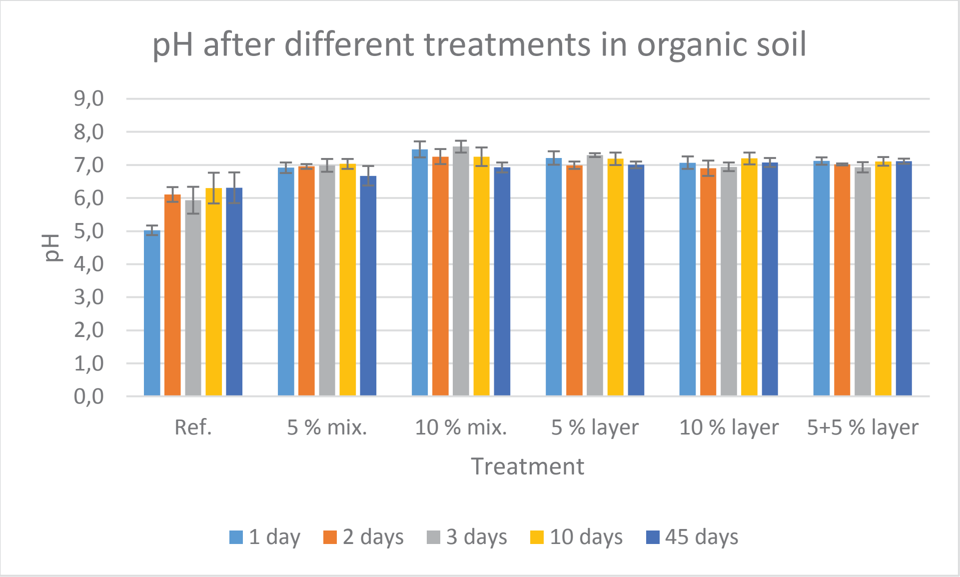


Figure 9. pH measured in water leachates from the columns with differently added sorbent in organic soil. The time between extractions going from 1 day to 45 days.

It can be seen in Figure 9 that the pH changes in leachates during the experimental period was only significant in leachates from the reference soil.

3.3.2 DOC

DOC concentrations from leachates analyzed from mineral soil are shown in Figure 10. The highest concentrations come from the untreated mineral soil. The concentrations decrease with increasing amount of sorbent added. There is also an increase in the DOC concentration in the last measurement (after 45 days) in all treatments.

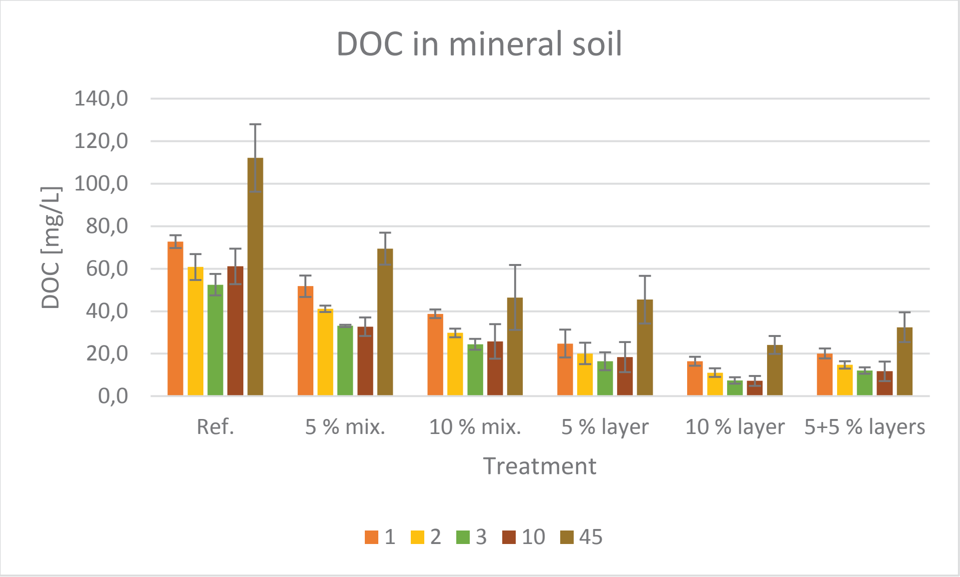


Figure 10. DOC concentrations measured in leachates from mineral soil after different sorbent treatments over the course of 45 days.

DOC concentrations in leachates from the organic soil are depicted in Figure 11. The maximum concentration is 14,4 mg/L DOC in the untreated soil leachates, while the minimum concentration is at a 0,8 mg/L DOC in the leachates from the treatment with a 10 % layer of sorbent is added. The tendency seem to be the same as in mineral soil; the amount of DOC decreases with an increasing amount of sorbent added.

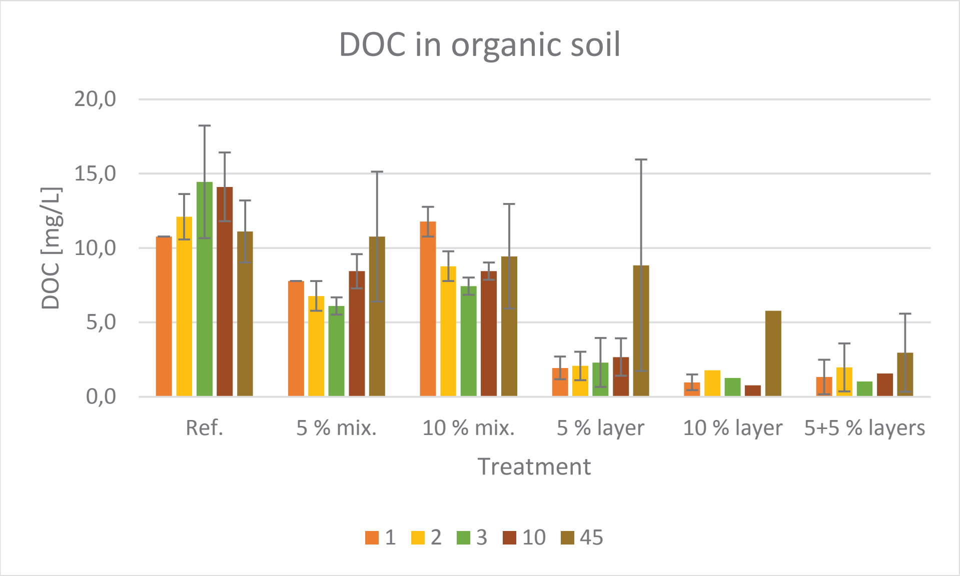


Figure 11. DOC concentrations measured in leachates from organic soil after different sorbent treatments over the course of 45 days. Standard deviations are missing for values from the treatment ’10 % layer’ and ‘5+5 % layers’ for the reason that one of the replicas were smaller than the blanc measured for the DOC (<4,2 mg/L), and therefore the author chose to leave them out.

3.3.3 Sulphate (SO_4^{2-})

The inorganic anions in leachates measured were chloride (Cl^-), nitrate (NO_3^-) and sulphate SO_4^{2-}. Since Cl- and NO_3^- were not significant for the variation in concentrations of Pb and Sb they are excluded from the figures. The concentrations of Cl^- varied from 20,3 mg/L to 58,3 mg/L in leachates from the mineral soil, and from 2,2 mg/L to 24,3 mg/L in leachates from the organic soil. The concentrations of nitrate varied from 23,5 to 86,9 mg/L in mineral soil, and from 19,2 to 86,9 mg/L in organic soil.

In mineral soil, the concentration of sulphate seemed to have a slight increase with increased amount of sorbent (see Figure 12.) The same goes for organic soil (see Figure 13.) In organic soil the untreated soil had far lower concentrations of sulphate than the untreated mineral soil. This inclines that the mineral soil naturally had higher amounts of sulphate.

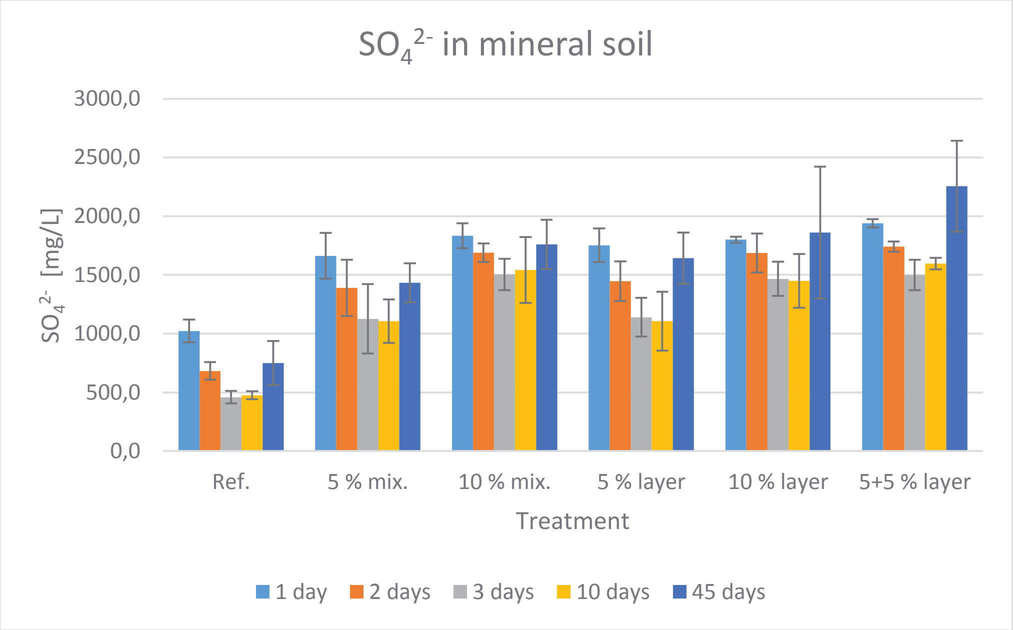


Figure 12. Concentrations of $[SO_4^{2-}]_w$ in leachates from mineral soil. Different treatments shown for the time of the study.

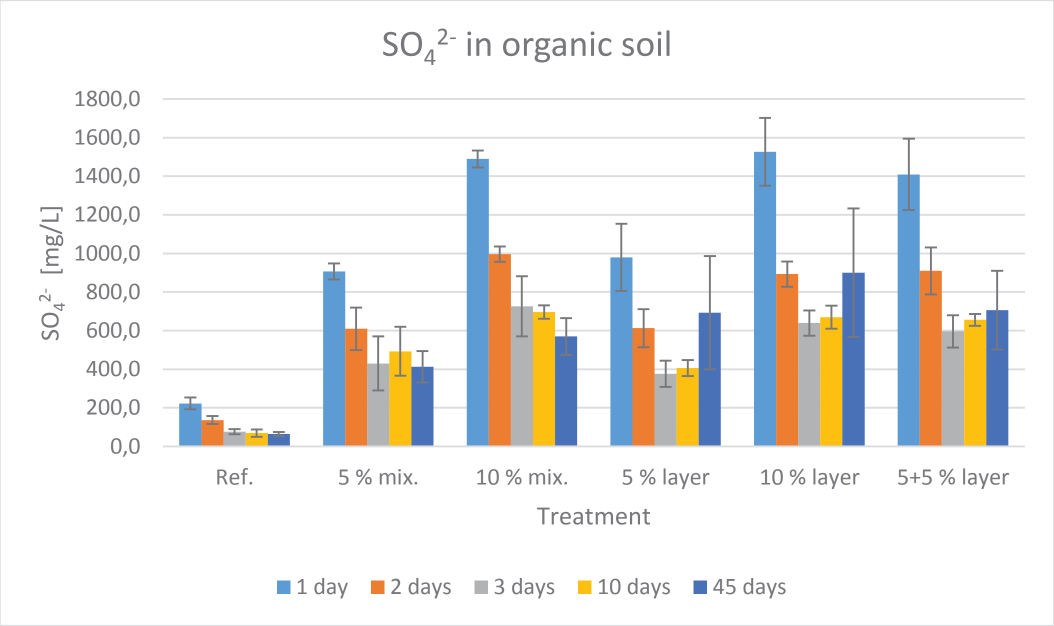


Figure 13. Concentrations of $[SO_4^{2-}]_w$ in leachates from organic soil. Different treatments shown for the time of the study.

3.5 Statistics and correlations

3.5.1 Influences on the concentrations of Pb and Sb

For the *organic soil* leachates, pH was the only parameter that correlated with the concentrations of Pb (see the regression plot in figure 14.) The plot shows a negative correlation between Pb and pH in the leachates, as the concentration of Pb decreases with an increasing pH.

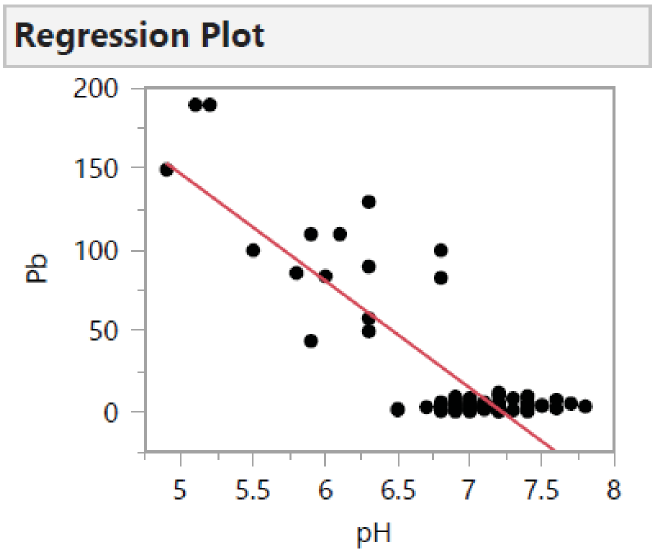


Figure 14. Multiple regression plot of concentration of Pb_w versus the pH in organic soil.

For *mineral soil* leachates, Pb was positively correlated with pH (see Figure 15). Lead was also a positive correlation with the concentration of DOC (see Figure 16.)

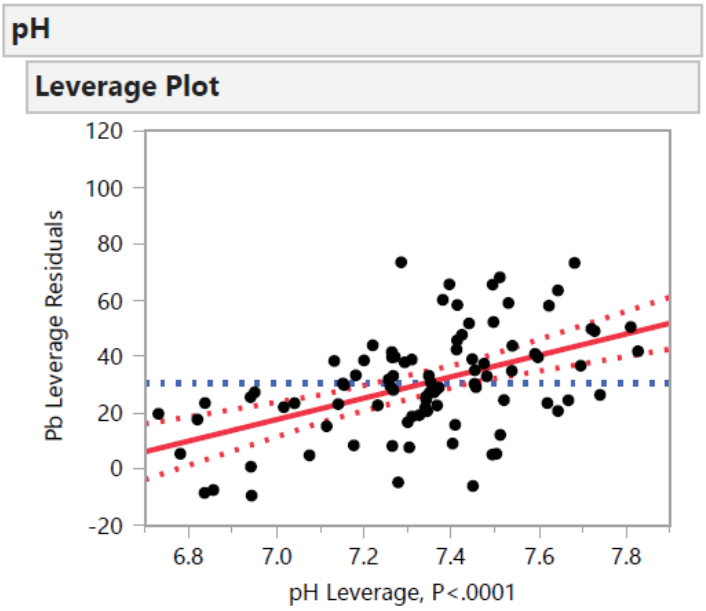


Figure 15. Plot of Pb_w versus pH in mineral soil.

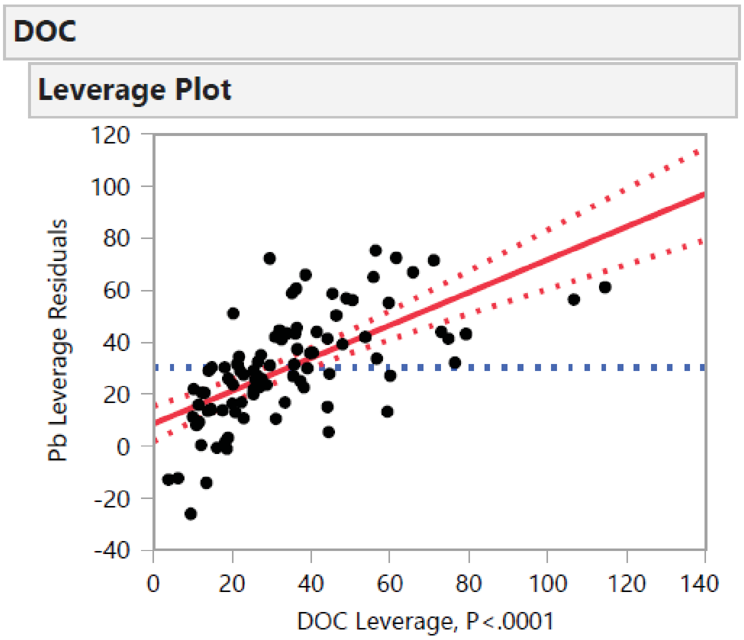


Figure 16. Plot of Pb_w versus DOC concentrations in mineral soil leachates.

Figure 17 shows the negative correlation Pb has with SO_4^{2-} in mineral soil. This negative correlation is related to treatment effects. The higher leaching of SO_4^{2-} is a response to treatment and the strongest leaching of SO_4^{2-} was collected from columns where the sorbent was located close to bottom of columns. In those columns, the adsorption of Pb was also most effective.

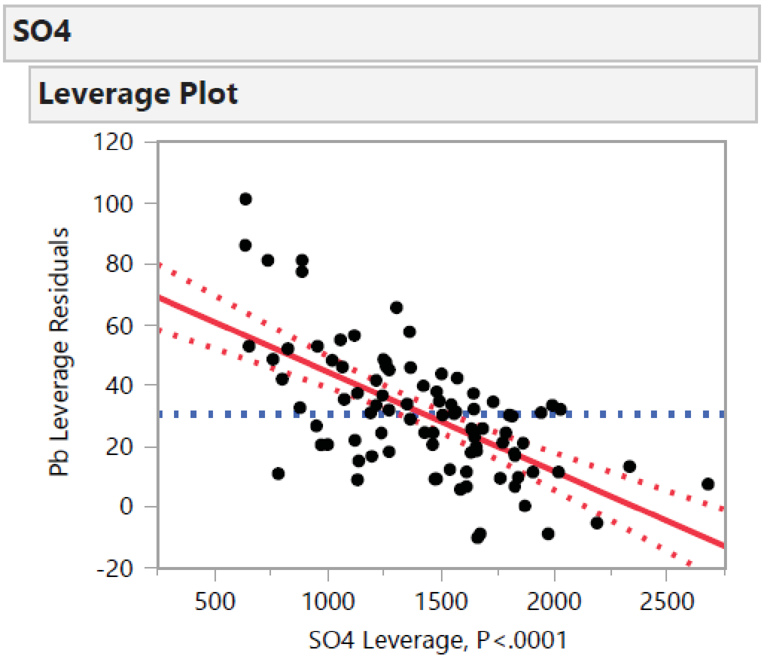


Figure 17. Plot of Pb_w versus SO_4^{2-} concentrations in mineral soil leachates.

Antimony in *organic soil* leachates was influenced by the pH (see Figure 18). The concentration of antimony increases when pH increases. Further, Sb in organic soil leachates was positively correlated with DOC (see Figure 19) and SO_4^{2-} (shown in Figure 20).

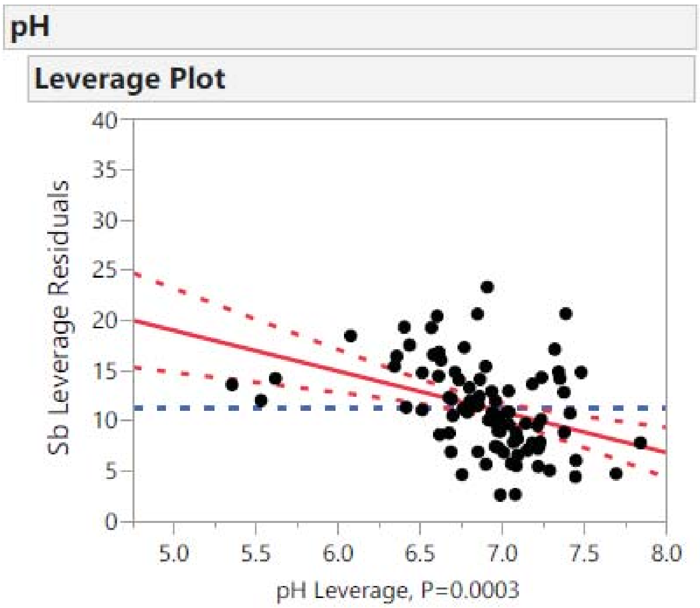


Figure 18. Plot of the concentrations of Sb_w plotted against the pH values in organic soil leachates.

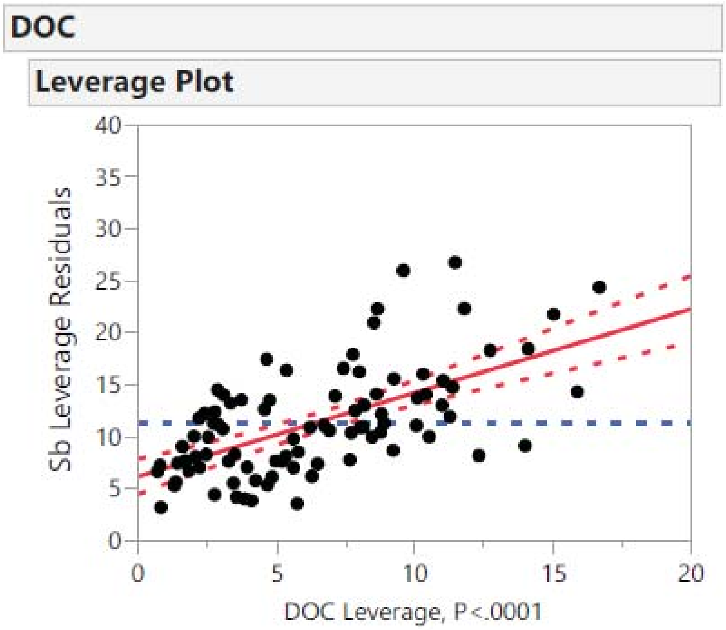


Figure 19. Plot of the concentrations of Sb_w plotted against the DOC values in organic soil leachates.

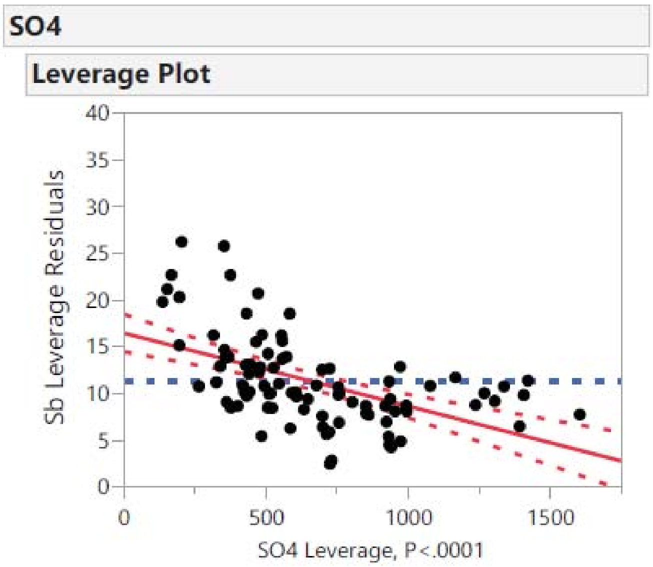


Figure 20. Plot of the concentrations of Sb_w against the concentrations of SO_4^{2-} in organic soil leachates.

Antimony in *mineral soil* leachates was influenced by pH, as seen in Figure 21. The concentration of Sb was positively correlated with pH. Antimony from leachates in mineral soil was also positively correlated with DOC (Figure 22) and SO_4^{2-} (Figure 23).

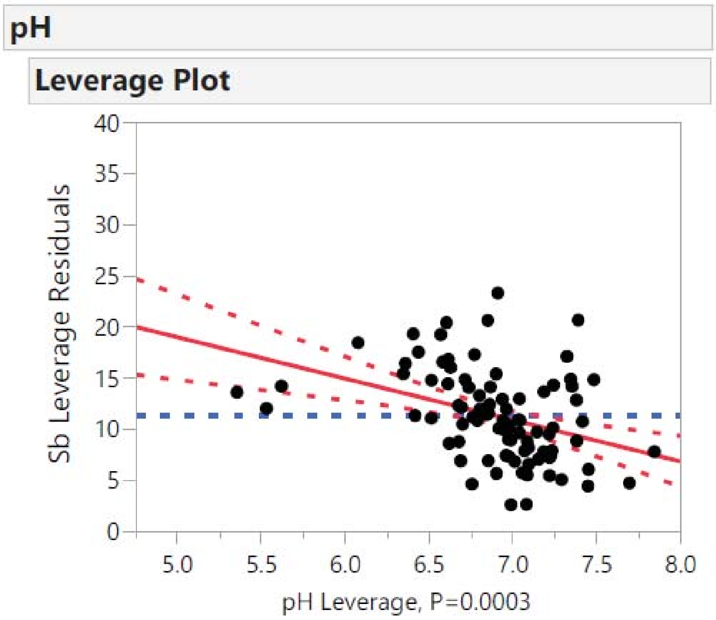


Figure 21. Plot of the concentration of Sb_w versus the pH in mineral soil leachates.

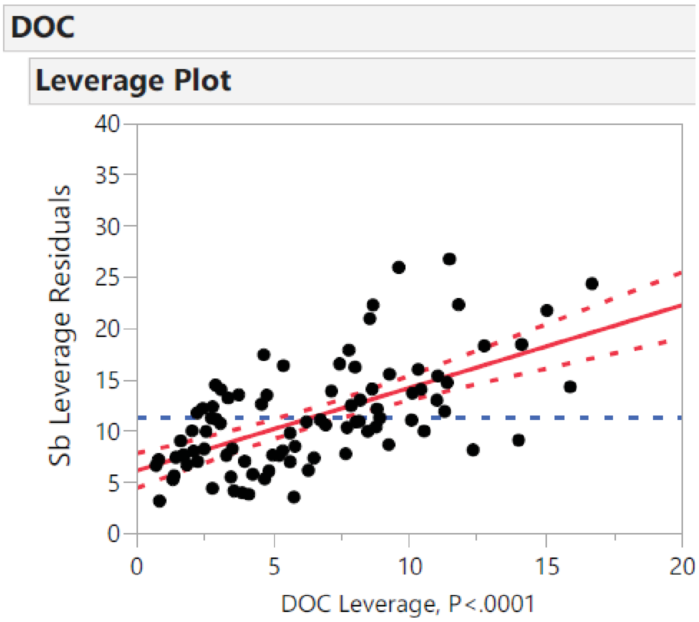


Figure 22. Plot of the concentration of Sb_w versus the DOC in mineral soil leachates.

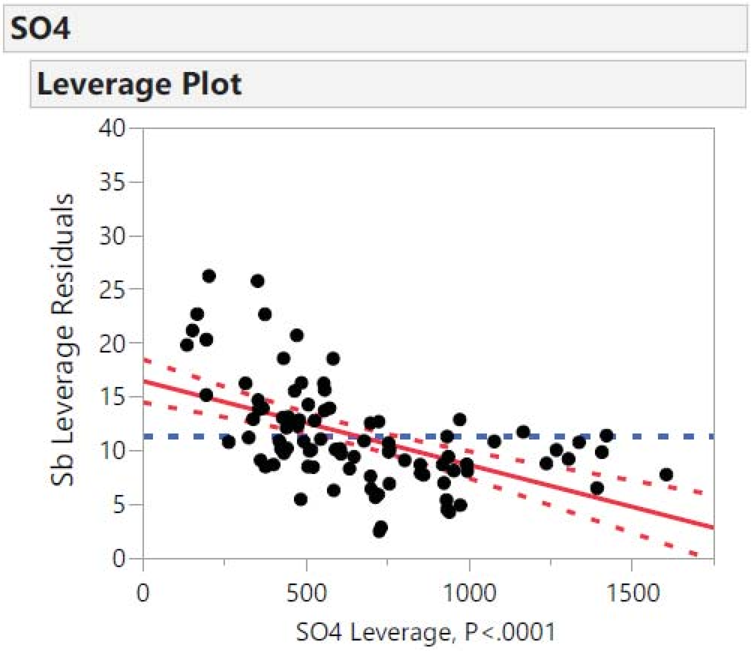


Figure 23. Plot of the concentration of Sb_w versus the concentration of SO_4^{2-} in mineral soil leachates.

3.5.2 The effect of time on immobilization

With Sb, the only significant effect time had is on the untreated mineral soil leachates (seen in Figure 24.) The time of study had little effect on Pb in mineral and organic soil leachates.

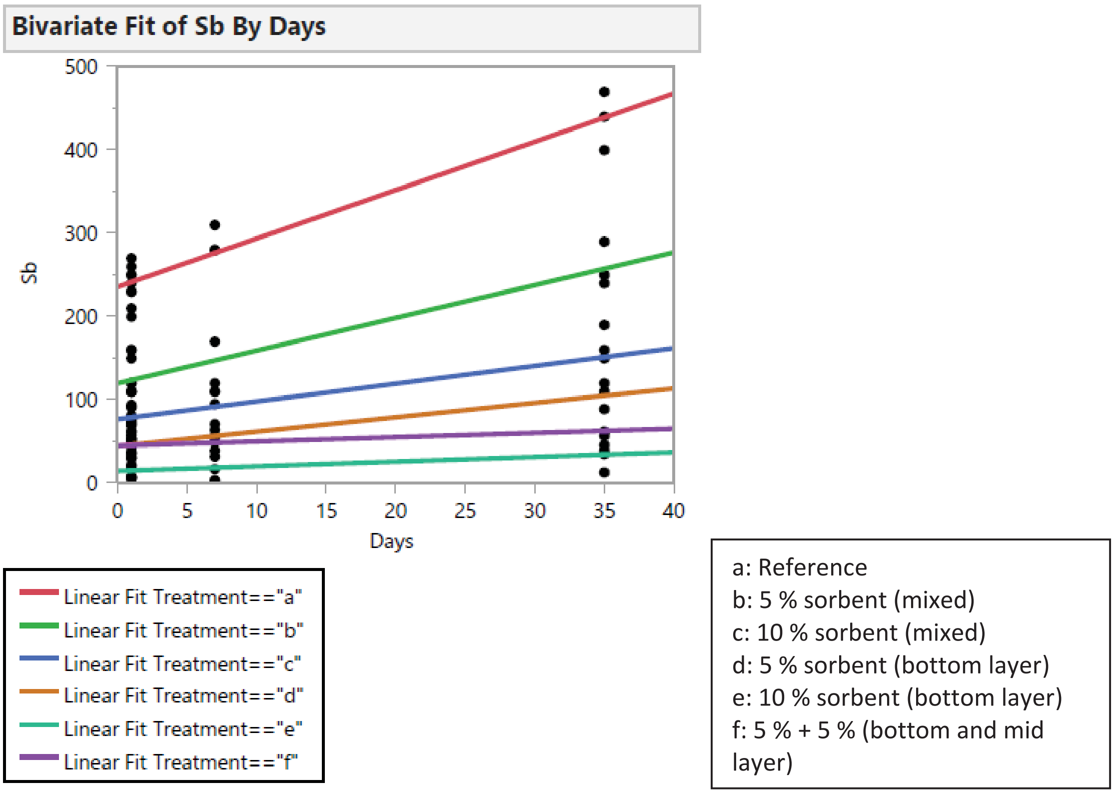


Figure 24. Bivariate fit of Sb_w according to days in leachates from mineral soil.

3.5.3 Repeated extractions

In *organic soil* leachates, Pb extractions were not far apart (Figure 25). The untreated soil is the treatment with the most variation after repeated extractions on Pb concentrations. For Sb (Figure 26) the variations were bigger. As with Pb the most noticeable variation is within the untreated soil leachates, but there is also variation found in the treatment d (with 5 % added sorbent in bottom layer).

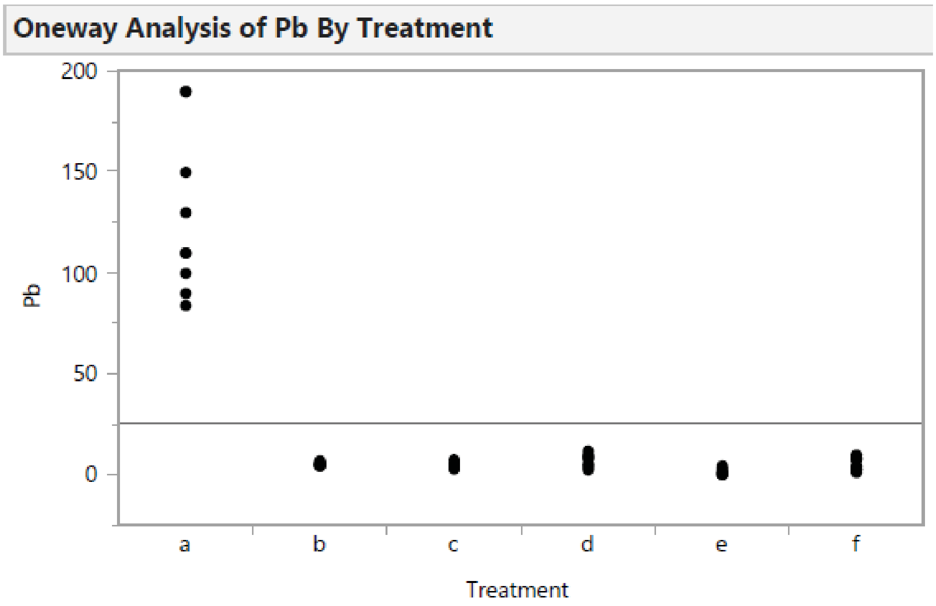


Figure 25. Oneway analysis of Pb_w by treatment in leachates from organic soil.

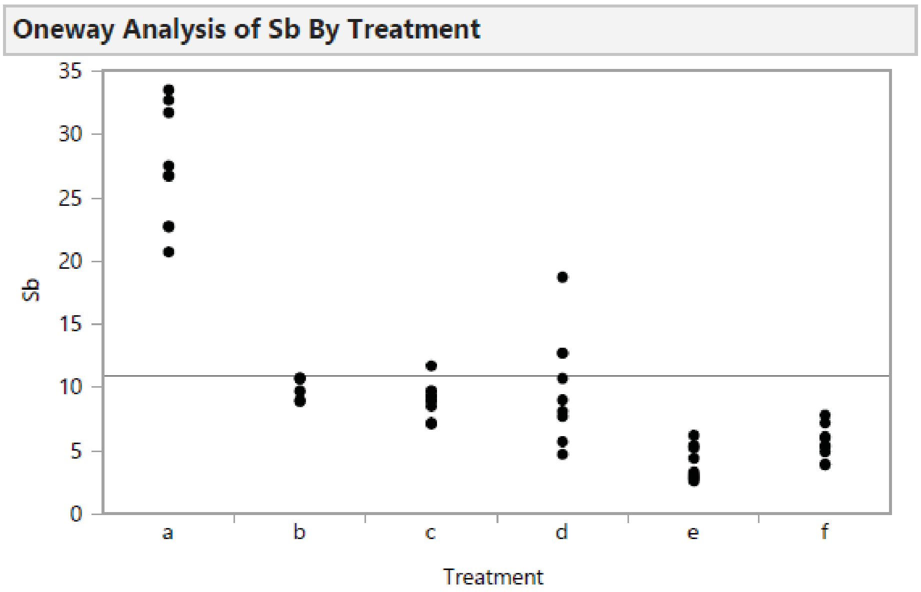


Figure 26. Oneway analysis of Sb_w by treatment in leachates from organic soil.

In *mineral soil* leachates, the variations were once again not very high for Pb (see Figure 27). Antimony had the highest variations the untreated soil and the soil with 5 % added sorbent (mixed in), as depicted in Figure 28.

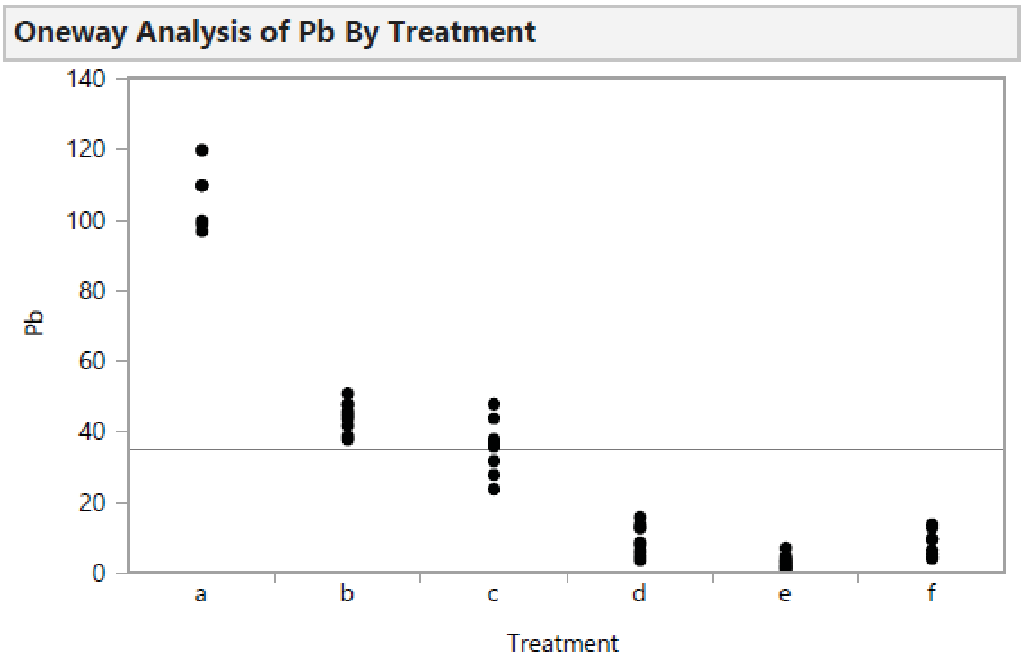


Figure 27. Oneway analysis of Pb_w by treatment in leachates from mineral soil.

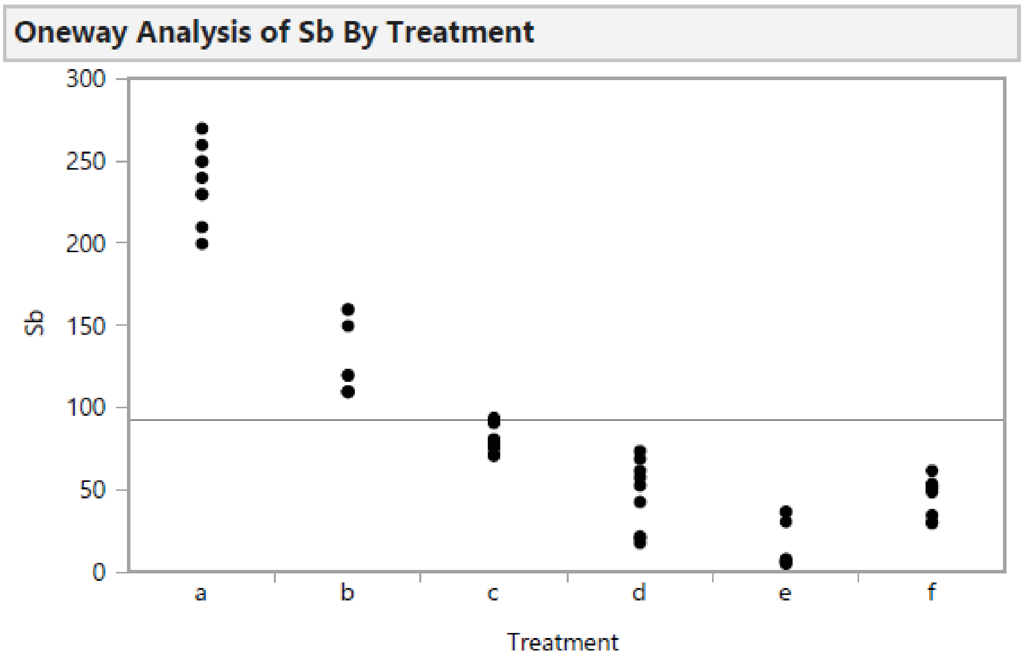


Figure 28. Oneway analysis of Sb_w by treatment in leachates from mineral soil.

4. Discussion

4.1 Immobilization effect of the sorbent and different treatments on lead and antimony

The column experiment showed that the sorbent works well on both polluted mineral soil and organic soil. There was a significant difference between extracts from the untreated reference columns and the columns with added sorbent for both trace metals. The results showed that both elements were immobilized to a high extent, as was expected from previous studies with Fe-sorbents (Okkenhaug et al. 2013; Sipos et al. 2008; Strømseng et al. 2011)

The study implies that how you add the sorbent affects the results. For organic soil, the amount of added sorbent was positively correlated with the SO_4^{2-} concentration, and also the concentration of antimony. For organic soil, antimony was positively correlated with the SO_4^{2-} concentration, which in turn was positively correlated with the amount of sorbent added. The increase in SO_4^{2-} is most likely an indirect effect of the treatment. The treatment is the biggest factor influencing antimony that leached from the column experiment.

The different treatments also affect other parameters like pH, DOC and SO_4^{2-}. This is a result of the Fe-based sorbent added. Where the treatment was added in layers of either a 5 % bottom layer, a 10 % bottom layer or a 5 % bottom layer in addition to a 5 % mid layer, the concentration of DOC in organic soil was the lowest in the leachates. The layers seemed to have worked as blocking agents, keeping the DOC in the soil. Adding sorbent in layers may therefore be the best way of remediating shooting range soil with this kind of a sorbent.

For mineral soil leachates, the increasing of pH affected the concentrations of DOC. This is because the solubility of DOC increases with an increasing pH, thus more DOC leaches from the mineral soil. Where the most sorbent is added, the pH is the highest, and here the most Pb leaches from the organic soil.

4.2 Stabilization effect over the time of the study

The pH in the leachates from the untreated organic soil increased significantly during the time of the study. This increase indicated the development of reducing conditions to some degree. For instance, the reduction of 1 mole $Fe(OH)_3$ to $Fe(OH)_2$ produces one mole of OH^-. The treatments with sorbent added buffered the pH to around pH 7, and hence any redox induced pH is not seen here. For the trace metals analyzed, the leachates in organic soil had little significance over time. Although, in untreated mineral soil, Sb seemed to break free from other binding agents in the soil after some time of the soil staying moist. For organic soil the difference was not significant.

Over time there were some leaching of Fe in mineral soil, and also SO_4^{2-}. When Fe leaches from soil, it takes SO_4^{2-} with it. In the treatments with bottom layers of 5 % and 10 % sorbent, Fe tended to increase over time. This could be because the sorbent is Fe-based and in the treatments with bottom layers of sorbent the sorbent is very close to the end of the column system. Some of the sorbent may have gone through the filter at the bottom since it has fine particles after sifting.

4.3 The effect of repeated extractions

According to the Anova analysis (see chapter 3.5 in Results), doing the extractions three days after each other didn't have a significant effect on the concentrations of Pb and Sb leached. The time for the study was set to 45 days, which needed to be longer to establish long-term effects.

In leachates from organic soil, Sb is more easily washed out in the untreated soil. This is likely to be because Sb does not have the same affinity for organic material, as does Pb. Lead is more easily bound to organic material in soil, and that might be the reason that it does not wash out as quickly.

Contact time for the sorbent and the soil were of little significance to the results of the trace metals in leachates from both soils. The exception here was untreated Sb in leachates from mineral soil.

4.4 Sources of error

The background concentrations of Pb, Sb and Fe in the shooting range soil were not taken into account. The results can therefore point towards a larger degree of contamination than is actually present.

Rhizon samplers were put into the columns to try and get a better picture of the local metal concentrations in the pore water of the soils. These could have helped in the speciation of the trace metals, but did not work in practice.

5. Conclusion

The results from the column experiment with added Fe-based sorbent show that stabilization with the industrial waste iron-based sorbent is a promising treatment to minimize leaching of lead and antimony from shooting-range soils, both organic and mineral. Contact time between sorbent and shooting range soil did not have significant effects apart from a raise in pH in untreated organic soil, which most likely is due to the development of reducing conditions. Repeated extractions in the columns experiment XXXXX. The most important fact that influenced the leaching of Pb and Sb in the study was the amount of sorbent added.

The result of the hypothesis:

I. *The sorbent will immobilize lead and antimony in both mineral and organic shooting-range soil.* The hypothesis cannot be rejected.

II. *Time will not influence the stabilization effect of the sorbent under field capacity conditions*. The hypothesis cannot be rejected nor validated.

III. *Repeated extractions and washing will not influence the stabilization effect of the sorbent.* The hypothesis cannot be rejected.

6. Further studies

The column experiment in this study went over the course of 5 weeks. Further studies on immobilization of Pb and Sb in shooting range soil should be done over a longer time period. This would help to get a better understanding of the possible changes over a long-time perspective, and would help with the work of stabilizing shooting range soils for years.

It could be an idea to make columns with a 50/50 content of each soil type (mineral and organic) before adding iron sorbent. This could be interesting in regards to making it easier to put shooting range soil in a landfill.

Speciation of the Sb and Pb in the extractions from the column experiments could help to answer the question of what fractions of the pollutants mobile and are transported.This could be helpful, since different oxidation states have a different level of toxicity for trace metals.

References

Alloway, B. J. (1995). *Heavy Metals in Soils*: Blackie Academic & Professional. 368 pp.

Almås, Å. R., Lofts, S., Tipping, E. & Mulder, J. (2007). Solubility of major cations and Cu, Zn and Cd in soil extracts of some contaminated agricultural soils near a zinc smelter in Norway: modelling with a multisurface extension of WHAM. *European Journal of Soil Science*, 58 (5): 1074-1086.

Blume, H. P., Horn, R., Brümmer, G. W., Kandeler, E., Kögel-Knabner, I., Kretzschmar, R., Stahr, K., Schachtschabel, P. & Wilke, B. M. (2010). *Scheffer/Schachtschabel: Lehrbuch der Bodenkunde*: Spektrum Akademischer Verlag.

Bradl, H. B. (2004). Adsorption of heavy metal ions on soils and soils constituents. *Journal of Colloid and Interface Science*, 277 (1): 1-18.

Brady, P. V. (1996). *The Physics and Chemistry of Mineral Surfaces*: Taylor & Francis.

Charlatchka, R. & Cambier, P. (2000). Influence of Reducing Conditions on Solubility of Trace Metals in Contaminated Soils. *Water, Air, and Soil Pollution*, 118 (1-2): 143-168.

Darling, C. T. R. & Thomas, V. G. (2003). The distribution of outdoor shooting ranges in Ontario and the potential for lead pollution of soil and water. *Science of The Total Environment*, 313 (1–3): 235-243.

Davies, B. E. (1995). Lead. In Alloway, B. J. (ed.) *Heavy Metals in Soils*: Blackie Academic & Professional.

Deliyanni, E. A., Peleka, E. N. & Matis, K. A. (2009). Modeling the sorption of metal ions from aqueous solution by iron-based adsorbents. *Journal of Hazardous Materials*, 172 (2–3): 550-558.

Filella, M., Belzile, N. & Chen, Y.-W. (2002). Antimony in the environment: a review focused on natural waters: II. Relevant solution chemistry. *Earth-Science Reviews*, 59 (1–4): 265-285.

Flynn, H. C., Meharg, A. A., Bowyer, P. K. & Paton, G. I. (2003). Antimony bioavailability in mine soils. *Environmental Pollution*, 124 (1): 93-100.

Forsvarsbygg. (2013). Tittelsnes SØF, Sveio kommune. Tiltaksplan, 138084.

Fränzle, S., Markert, B. & Wünschmann, S. (2012). *Introduction to Environmental Engineering*: Wiley.

Garmo, Ø. A. (2015). Overvåking av avrenning fra nedlagte skyte- og øvingsfelt. Årsrapport for 2014, NIVA-rapport; 6786. NIVA.

Gebel, T. (1997). Arsenic and antimony: comparative approach on mechanistic toxicology. *Chemico-Biological Interactions*, 107 (3): 131-144.

Gleyzes, C., Tellier, S. & Astruc, M. (2002). Fractionation studies of trace elements in contaminated soils and sediments: a review of sequential extraction procedures. *TrAC Trends in Analytical Chemistry*, 21 (6–7): 451-467.

Hansen, H. J. & Danielsberg, A. (2009). Classification of condition for contaminated sites, TA-2553/2009: Norwegian Pollution Control Authority. 27 pp.

Heier, L. S., Meland, S., Ljones, M., Salbu, B. & Stromseng, A. E. (2010). Short-term temporal variations in speciation of Pb, Cu, Zn and Sb in a shooting range runoff stream. *Sci Total Environ*, 408 (11): 2409-17.

Herzel, H. (2012). *Demobilisation of Metals in Shooting Range by Secondary Mineralisation and Sorption on iron Oxides*, Chemisch-geowissenschaftlichen Fakultät der Friedrich-Schiller-Universität Jena. 61 pp.

Ivezic, V., Almås, Å. R. & Singh, B. R. (2012). Predicting the solubility of Cd, Cu, Pb and Zn in uncontaminated Croatian soils under different land uses by applying established regression models. *Geoderma*, 170: 89-95.

Janssen, R. P. T., Peijnenburg, W. J. G. M., Posthuma, L. & Van Den Hoop, M. A. G. T. (1997). Equilibrium partitioning of heavy metals in dutch field soils. I. Relationship between metal partition coefficients and soil characteristics. *Environmental Toxicology and Chemistry*, 16 (12): 2470-2478.

Jardine, P. M., McCarthy, J. F. & Weber, N. L. (1989). Mechanisms of Dissolved Organic Carbon Adsorption on Soil. *Soil Sci. Soc. Am. J.*, 53 (5): 1378-1385.

Johnson, C. A., Moench, H., Wersin, P., Kugler, P. & Wenger, C. (2005). Solubility of antimony and other elements in samples taken from shooting ranges. *J Environ Qual*, 34 (1): 248-54.

Alloway, B. J. (1995). *Heavy Metals in Soils*: Blackie Academic & Professional. 368 pp.

Blume, H. P., Horn, R., Brümmer, G. W., Kandeler, E., Kögel-Knabner, I., Kretzschmar, R., Stahr, K., Schachtschabel, P. & Wilke, B. M. (2010). *Scheffer/Schachtschabel: Lehrbuch der Bodenkunde*: Spektrum Akademischer Verlag.

Bradl, H. B. (2004). Adsorption of heavy metal ions on soils and soils constituents. *Journal of Colloid and Interface Science*, 277 (1): 1-18.

Charlatchka, R. & Cambier, P. (2000). Influence of Reducing Conditions on Solubility of Trace Metals in Contaminated Soils. *Water, Air, and Soil Pollution*, 118 (1-2): 143-168.

Darling, C. T. R. & Thomas, V. G. (2003). The distribution of outdoor shooting ranges in Ontario and the potential for lead pollution of soil and water. *Science of The Total Environment*, 313 (1–3): 235-243.

Davies, B. E. (1995). Lead. In Alloway, B. J. (ed.) *Heavy Metals in Soils*: Blackie Academic & Professional.

Deliyanni, E. A., Peleka, E. N. & Matis, K. A. (2009). Modeling the sorption of metal ions from aqueous solution by iron-based adsorbents. *Journal of Hazardous Materials*, 172 (2–3): 550-558.

Filella, M., Belzile, N. & Chen, Y.-W. (2002). Antimony in the environment: a review focused on natural waters: II. Relevant solution chemistry. *Earth-Science Reviews*, 59 (1–4): 265-285.

Flynn, H. C., Meharg, A. A., Bowyer, P. K. & Paton, G. I. (2003). Antimony bioavailability in mine soils. *Environmental Pollution*, 124 (1): 93-100.

Forsvarsbygg. (2013). Tittelsnes SØF, Sveio kommune. Tiltaksplan, 138084.

Fränzle, S., Markert, B. & Wünschmann, S. (2012). *Introduction to Environmental Engineering*: Wiley.

Garmo, Ø. A. (2015). Overvåking av avrenning fra nedlagte skyte- og øvingsfelt. Årsrapport for 2014, NIVA-rapport; 6786. NIVA.

Gebel, T. (1997). Arsenic and antimony: comparative approach on mechanistic toxicology. *Chemico-Biological Interactions*, 107 (3): 131-144.

Gleyzes, C., Tellier, S. & Astruc, M. (2002). Fractionation studies of trace elements in contaminated soils and sediments: a review of sequential extraction procedures. *TrAC Trends in Analytical Chemistry*, 21 (6–7): 451-467.

Hansen, H. J. & Danielsberg, A. (2009). Classification of condition for contaminated sites, TA-2553/2009: Norwegian Pollution Control Authority. 27 pp.

Heier, L. S., Meland, S., Ljones, M., Salbu, B. & Stromseng, A. E. (2010). Short-term temporal variations in speciation of Pb, Cu, Zn and Sb in a shooting range runoff stream. *Sci Total Environ*, 408 (11): 2409-17.

Herzel, H. (2012). *Demobilisation of Metals in Shooting Range by Secondary Mineralisation and Sorption on iron Oxides*, Chemisch-geowissenschaftlichen Fakultät der Friedrich-Schiller-Universität Jena. 61 pp.

Jardine, P. M., McCarthy, J. F. & Weber, N. L. (1989). Mechanisms of Dissolved Organic Carbon Adsorption on Soil. *Soil Sci. Soc. Am. J.*, 53 (5): 1378-1385.

Johnson, C. A., Moench, H., Wersin, P., Kugler, P. & Wenger, C. (2005). Solubility of antimony and other elements in samples taken from shooting ranges. *J Environ Qual*, 34 (1): 248-54.

Krachler, M., Emons, H. & Zheng, J. (2001). Speciation of antimony for the 21st century: promises and pitfalls. *TrAC Trends in Analytical Chemistry*, 20 (2): 79-90.

Lambrechts, T., Couder, E., Bernal, M. P., Faz, Á., Iserentant, A. & Lutts, S. (2011). Assessment of Heavy Metal Bioavailability in Contaminated Soils from a Former Mining Area (La Union, Spain) Using a Rhizospheric Test. *Water, Air, & Soil Pollution*, 217 (1-4): 333-346.

Lovdata. (1983). *Kapittel 9. Deponering av avfall. Forskrift om gjenvinning og behandling av avfall (avfallsforskriften).*

Manaka, M. (2006). Amount of amorphous materials in relationship to arsenic, antimony, and bismuth concentrations in a brown forest soil. *Geoderma*, 136 (1–2): 75-86.

Mielke, H. W., Gonzales, C. R., Powell, E., Jartun, M. & Mielke Jr, P. W. (2007). Nonlinear association between soil lead and blood lead of children in metropolitan New Orleans, Louisiana: 2000–2005. *Science of The Total Environment*, 388 (1–3): 43-53.

Mitsunobu, S., Takahashi, Y. & Sakai, Y. (2008). Abiotic reduction of antimony(V) by green rust (Fe4(II)Fe2(III)(OH)12SO4 · 3H2O). *Chemosphere*, 70 (5): 942-947.

Nordberg, G. F., Fowler, B. A. & Nordberg, M. (2014). *Handbook on the Toxicology of Metals*: Elsevier Science.

Nordberg, M., Duffus, J. & Templeton, D. M. (2004). *Glossary of terms used in toxicokinetics (IUPAC Recommendations 2003)*. Pure and Applied Chemistry, 76, 5. p. 1033.

O'Day, P. A. & Vlassopoulos, D. (2010). Mineral-Based Amendments for Remediation. *Elements (Quebec, Quebec)*, 6 (6): 375-381.

Okkenhaug, G., Zhu, Y.-G., Luo, L., Lei, M., Li, X. & Mulder, J. (2011). Distribution, speciation and availability of antimony (Sb) in soils and terrestrial plants from an active Sb mining area. *Environmental Pollution*, 159 (10): 2427-2434.

Okkenhaug, G., Amstätter, K., Lassen Bue, H., Cornelissen, G., Breedveld, G. D., Henriksen, T. & Mulder, J. (2013). Antimony (Sb) Contaminated Shooting Range Soil: Sb Mobility and Immobilization by Soil Amendments. *Environmental Science & Technology*, 47 (12): 6431-6439.

Okkenhaug, G. & Breedveld, G. D. (2014). *Etablering av pilot-test for stabilisert skytefeltjord ved Oredalen avfallsanlegg*: NGI. Unpublished manuscript.

Paquin, P. R., Gorsuch, J. W., Apte, S., Batley, G. E., Bowles, K. C., Campbell, P. G., Delos, C. G., Di Toro, D. M., Dwyer, R. L., Galvez, F., et al. (2002). The biotic ligand model: a historical overview. *Comp Biochem Physiol C Toxicol Pharmacol*, 133 (1-2): 3-35.

Reistad, T., Fjellheim, K., Pryds, P. & Longva, K. (2013). Forsvarssektorens miljø- og klimaregnskap for 2012. In (FFI), F. F. (ed.). Forsvarets Forskningsinstitutt (FFI).

Santillan-Medrano, J. & Jurinak, J. J. (1975). The Chemistry of Lead and Cadmium in Soil: Solid Phase Formation1. *Soil Sci. Soc. Am. J.*, 39 (5): 851-856.

Sipos, P., Németh, T., Kis, V. K. & Mohai, I. (2008). Sorption of copper, zinc and lead on soil mineral phases. *Chemosphere*, 73 (4): 461-469.

Skoog, D. A., Crouch, S. R. & Holler, F. J. (2007). *Principles of instrumental analysis.* , vol. XV: Belmont, Calif.: Thomson. 1039 pp.

Strømseng, A. E., Ljønes, M. & Mariussen, E. (2011). Testing av jordforbedringsmidler for stabilisering av tungmetaller i jord, 2010/00696: FFI.

USEPA. (2013). *Appendix A to Part 423-126 Priority Pollutants. Protection of Environment. Chapter 1 - Environmental Protection Agency (Continued). Subchapter N - Effluent guidelines and standards.* Available at: http://www.gpo.gov/fdsys/pkg/CFR-2013-title40-vol30/xml/CFR-2013-title40-vol30-part423.xml.

Vega, F. A., Covelo, E. F. & Andrade, M. L. (2006). Competitive sorption and desorption of heavy metals in mine soils: Influence of mine soil characteristics. *Journal of Colloid and Interface Science*, 298 (2): 582-592.

Voie, Ø., Strømseng, A., Johnsen, A., Rossland, H. K., Karsrud, T. & Longva, K. (2010). Veileder for undersøkelse, risikovurdering, opprydning og avhending av skytebaner og øvingfelt, 2010/00116: FFI.

Kinniburgh, D. G., Milne, C. J., Benedetti, M. F., Pinheiro, J. P., Filius, J., Koopal, L. K. & Van Riemsdijk, W. H. (1996). Metal ion binding by humic acid: Application of the Nica-Donnan Model. *Environmental Science & Technology*, 30 (5): 1687-1698.

Krachler, M., Emons, H. & Zheng, J. (2001). Speciation of antimony for the 21st century: promises and pitfalls. *TrAC Trends in Analytical Chemistry*, 20 (2): 79-90.

Lambrechts, T., Couder, E., Bernal, M. P., Faz, Á., Iserentant, A. & Lutts, S. (2011). Assessment of Heavy Metal Bioavailability in Contaminated Soils from a Former Mining Area (La Union, Spain) Using a Rhizospheric Test. *Water, Air, & Soil Pollution*, 217 (1-4): 333-346.

Lovdata. (1983). *Kapittel 9. Deponering av avfall. Forskrift om gjenvinning og behandling av avfall (avfallsforskriften)*.

Manaka, M. (2006). Amount of amorphous materials in relationship to arsenic, antimony, and bismuth concentrations in a brown forest soil. *Geoderma*, 136 (1–2): 75-86.

Mielke, H. W., Gonzales, C. R., Powell, E., Jartun, M. & Mielke Jr, P. W. (2007). Nonlinear association between soil lead and blood lead of children in metropolitan New Orleans, Louisiana: 2000–2005. *Science of The Total Environment*, 388 (1–3): 43-53.

Mitsunobu, S., Takahashi, Y. & Sakai, Y. (2008). Abiotic reduction of antimony(V) by green rust (Fe4(II)Fe2(III)(OH)12SO4 · 3H2O). *Chemosphere*, 70 (5): 942-947.

Nordberg, G. F., Fowler, B. A. & Nordberg, M. (2014). *Handbook on the Toxicology of Metals*: Elsevier Science.

Nordberg, M., Duffus, J. & Templeton, D. M. (2004). *Glossary of terms used in toxicokinetics (IUPAC Recommendations 2003)*. Pure and Applied Chemistry, 76, 5. p. 1033.

O'Day, P. A. & Vlassopoulos, D. (2010). Mineral-Based Amendments for Remediation. *Elements (Quebec, Quebec)*, 6 (6): 375-381.

Okkenhaug, G., Zhu, Y.-G., Luo, L., Lei, M., Li, X. & Mulder, J. (2011). Distribution, speciation and availability of antimony (Sb) in soils and terrestrial plants from an active Sb mining area. *Environmental Pollution*, 159 (10): 2427-2434.

Okkenhaug, G., Amstätter, K., Lassen Bue, H., Cornelissen, G., Breedveld, G. D., Henriksen, T. & Mulder, J. (2013). Antimony (Sb) Contaminated Shooting Range Soil: Sb Mobility and Immobilization by Soil Amendments. *Environmental Science & Technology*, 47 (12): 6431-6439.

Okkenhaug, G. & Breedveld, G. D. (2014). *Etablering av pilot-test for stabilisert skytefeltjord ved Oredalen avfallsanlegg*: NGI. Unpublished manuscript.

Santillan-Medrano, J. & Jurinak, J. J. (1975). The Chemistry of Lead and Cadmium in Soil: Solid Phase Formation1. *Soil Sci. Soc. Am. J.*, 39 (5): 851-856.

Sipos, P., Németh, T., Kis, V. K. & Mohai, I. (2008). Sorption of copper, zinc and lead on soil mineral phases. *Chemosphere*, 73 (4): 461-469.

Skoog, D. A., Crouch, S. R. & Holler, F. J. (2007). *Principles of instrumental analysis.* , vol. XV: Belmont, Calif.: Thomson. 1039 pp.

Smolders, E., Oorts, K., van Sprang, P., Schoeters, I., Janssen, C. R., McGrath, S. P. & McLaughlin, M. J. (2009). Toxicity of Trace Metals in Soil As Affected by Soil Type and Aging After Contamination: Using Calibrated Bioavailability Models to Set Ecological Soil Standards. *Environmental Toxicology and Chemistry*, 28 (8): 1633-1642.

Strømseng, A. E., Ljønes, M. & Mariussen, E. (2011). Testing av jordforbedringsmidler for stabilisering av tungmetaller i jord, 2010/00696: FFI.

Temminghoff, E. J. M., Plette, A. C. C., Van Eck, R. & Van Riemsdijk, W. H. (2000). Determination of the chemical speciation of trace metal in aqueous systems by the Wageningen Donnan Membrane Technique. *Analytica Chimica Acta*, 417: 149-157.

Tipping, E. (1998). Humic ion-binding Model VI: An improved description of the interactions of protons and metal ions with humic substances. *Aquatic Geochemistry*, 4: 3-48.

Tipping, E., Rieuwerts, J., Pan, G., Ashmore, M. R., Lofts, S., Fargo, M. & Thornton, I. (2003). The solid solution partitioning of heavy metals (Cu, Zn, Cd, Pb) in upland soils of England and Wales. *Environmental Pollution*, 125 (2): 213-225.

USEPA. (2013). *Appendix A to Part 423-126 Priority Pollutants. Protection of Environment. Chapter 1 - Environmental Protection Agency (Continued). Subchapter N - Effluent guidelines and standards.* Available at: http://www.gpo.gov/fdsys/pkg/CFR-2013-title40-vol30/xml/CFR-2013-title40-vol30-part423.xml.

Vega, F. A., Covelo, E. F. & Andrade, M. L. (2006). Competitive sorption and desorption of heavy metals in mine soils: Influence of mine soil characteristics. *Journal of Colloid and Interface Science*, 298 (2): 582-592.

Voie, Ø., Strømseng, A., Johnsen, A., Rossland, H. K., Karsrud, T. & Longva, K. (2010). Veileder for undersøkelse, risikovurdering, opprydning og avhending av skytebaner og øvingfelt, 2010/00116: FFI.

Attachments

Table 1. Raw data from organic (green column) and mineral (yellow column) leachates. Pb, Sb, Fe, pH, DOC, Cl, NO_3^- and SO_4^{2-}.

ORGANIC	Time	Treatment	Replicate	Pb	Sb	Fe	pH	DOC	Cl	NO3	SO4
	1 day	A	a	189,7	22,7	65,9	5,2	10,8	26,6	74,9	249,6
			a	149,7	22,7	90,9	4,9	10,8	20,6	53,9	189,6
			a	189,7	20,7	75,9	5,1	10,8	25,6	69,9	229,6
		B	b	6,8	10,7	63,9	6,9	7,8	22,6	79,9	919,6
			b	5,7	9,0	241,9	7,1	7,8	17,6	64,9	859,6
			b	5,8	10,7	78,9	6,8	7,8	21,6	77,9	939,6
		C	c	5,4	9,3	84,9	7,2	11,8	24,6	109,9	1439,6
			c	5,0	8,5	371,9	7,7	10,8	23,6	92,9	1509,6
			c	7,3	11,7	621,9	7,6	12,8	23,6	94,9	1519,6
		D	d	2,4	8,1	161,9	7,0	1,8	23,6	52,9	1059,6
			d	9,6	9,0	241,9	7,4	2,8	18,6	44,9	779,6
			d	5,0	4,7	131,9	7,2	1,3	21,6	47,9	1099,6
		E	e	0,1	2,6	31,9	6,9	0,6	25,6	50,9	1669,6
			e	2,3	5,4	101,9	7,2	1,6	20,6	47,9	1329,6
			e	0,3	2,8	25,9	7,2	0,8	23,6	51,9	1579,6
		F	f	1,1	3,9	52,9	7,0	1,2	17,6	38,9	1399,6
			f	9,7	5,4	261,9	7,2	2,6	16,6	39,9	1229,6
			f	2,5	3,9	73,9	7,2	0,3	20,6	47,9	1599,6
	2 days	A	a	129,7	26,7	87,9	6,3	10,8	16,6	45,9	159,6
			a	109,7	27,5	141,9	5,9	13,8	10,6	28,9	119,6
			a	109,7	26,7	111,9	6,1	11,8	11,6	31,9	129,6
		B	b	5,1	9,7	48,9	7,0	5,8	14,6	59,9	719,6
			b	5,0	8,9	251,9	6,9	6,8	7,8	29,9	499,6
			b	4,6	10,7	62,9	7,0	7,8	10,6	40,9	609,6
		C	c	3,9	8,9	54,9	7,1	8,8	15,6	68,9	1039,6
			c	3,8	7,2	321,9	7,5	7,8	9,6	41,9	959,6
			c	4,5	9,7	591,9	7,2	9,8	10,6	45,9	989,6
		D	d	3,4	10,7	111,9	7,1	1,7	14,6	37,9	679,6
			d	9,2	12,7	271,9	6,9	3,2	10,6	28,9	499,6
			d	7,7	5,7	181,9	6,9	1,4	11,6	28,9	659,6
		E	e	1,1	3,1	32,9	6,9	3,8	11,6	25,9	959,6
			e	2,8	5,2	171,9	6,7	1,8	10,6	26,9	829,6
			e	0,4	2,9	30,9	7,2	4,2	10,6	25,9	889,6
		F	f	4,3	5,3	91,9	7,0	1,1	9,4	20,9	849,6
			f	8,2	6,1	231,9	7,0	2,9	10,6	23,9	829,6
			f	2,6	4,9	42,9	7,1	4,3	9,6	23,9	1049,6

3 days	A	a	89,7	31,7	131,9	6,3	11,8	7,1	21,9	91,6
		a	99,7	33,5	241,9	5,5	18,8	4,1	13,9	68,6
		a	83,7	32,7	161,9	6,0	12,8	4,2	13,9	67,6
	B	b	4,3	9,7	49,9	7,2	5,8	9,6	42,9	589,6
		b	4,7	8,9	261,9	6,9	6,8	4,4	18,9	369,6
		b	4,3	10,7	53,9	6,9	5,8	3,9	18,9	329,6
	C	c	3,8	9,7	69,9	7,5	7,8	9,6	46,9	899,6
		c	2,8	7,1	221,9	7,4	6,8	4,7	20,9	599,6
		c	3,3	9,0	551,9	7,8	7,8	5,8	24,9	679,6
	D	d	4,8	12,7	101,9	7,4	1,7	8,3	21,9	429,6
		d	11,7	18,7	311,9	7,2	4,2	5,0	14,9	299,6
		d	8,2	7,7	181,9	7,3	1,1	6,2	16,9	399,6
	E	e	0,5	4,4	59,9	6,8	4,2	8,1	19,9	699,6
		e	4,3	6,2	201,9	7,1	1,3	5,3	13,9	569,6
		e	0,2	3,3	27,9	7,0	3,8	6,9	16,9	649,6
	F	f	7,8	7,2	161,9	7,0	0,6	5,2	12,9	529,6
		f	7,5	7,8	241,9	7,0	1,5	5,7	14,9	569,6
		f	3,7	6,0	48,9	6,8	3,8	4,8	12,9	689,6
1 week	A	a	82,7	32,7	111,9	6,8	12,8	6,4	22,9	91,6
		a	85,7	35,5	201,9	5,8	16,8	2,2	11,9	59,6
		a	57,7	35,7	141,9	6,3	12,8	2,6	12,9	57,6
	B	b	3,6	10,7	26,9	6,9	7,8	7,5	40,9	629,6
		b	2,9	9,7	72,9	7,2	9,8	4,8	28,9	469,6
		b	3,1	9,7	46,9	7,0	7,8	4,3	25,9	379,6
	C	c	2,4	8,9	52,9	7,2	7,8	5,8	33,9	699,6
		c	2,0	7,1	471,9	7,6	8,8	5,4	28,9	659,6
		c	2,3	7,4	301,9	7,0	8,8	5,8	28,9	729,6
	D	d	3,1	10,7	131,9	7,1	1,7	10,6	24,9	439,6
		d	5,0	15,7	221,9	7,4	4,1	5,2	12,9	359,6
		d	5,1	8,6	141,9	7,0	2,3	6,5	15,9	419,6
	E	e	-0,1	5,8	21,9	7,1	0,1	8,8	15,9	669,6
		e	1,0	6,7	131,9	7,2	1,5	5,3	10,9	609,6
		e	0,1	3,6	23,9	7,4	3,1	8,9	14,9	729,6
	F	f	2,5	8,5	87,9	7,0	1,3	9,2	17,9	649,6
		f	5,0	8,9	151,9	7,1	1,9	6,7	14,9	629,6
		f	0,9	5,1	62,9	7,3	3,9	6,9	14,9	689,6
5 weeks	A	a	99,7	22,7	63,9	6,8	8,8	2,9	60,9	69,6
		a	43,7	23,5	101,9	5,9	12,8	1,1	36,9	52,6
		a	49,7	23,7	86,9	6,3	11,8	2,4	50,9	70,6
	B	b	1,5	11,7	14,9	7,0	8,8	3,7	65,9	449,6
		b	1,0	9,5	11,9	6,5	7,8	4,3	71,9	319,6
		b	1,7	13,7	50,9	6,5	15,8	7,9	84,9	469,6
	C	c	0,9	9,7	13,9	7,0	5,8	4,8	56,9	459,6
		c	0,7	9,3	19,9	6,8	12,8	6,8	85,9	619,6
		c	0,6	10,7	16,9	7,0	9,8	5,8	80,9	629,6

		D	d	-0,2	5,3	101,9	7,0	1,0	6,6	30,9	359,6
			d	1,1	11,7	611,9	6,9	10,8	8,8	26,9	809,6
			d	2,1	10,7	1091,9	7,1	14,8	9,0	20,9	909,6
		E	e	-0,2	3,8	84,9	7,0	4,2	7,9	17,9	759,6
			e	-0,2	7,6	2691,9	7,0	5,8	10,6	24,9	1279,6
			e	0,4	4,1	121,9	7,2	3,8	4,7	16,9	659,6
		F	f	1,4	6,3	401,9	7,1	2,6	9,0	34,9	659,6
			f	1,6	6,8	401,9	7,1	5,8	8,6	24,9	929,6
			f	0,8	6,6	131,9	7,2	0,6	4,4	24,9	529,6
MINERAL	1 day	A	a	109,7	229,7	30,9	7,6	69,8	49,6	55,9	939,6
			a	96,7	199,7	33,9	7,7	75,8	58,6	66,9	1129,6
			a	99,7	209,7	28,9	7,5	72,8	51,6	58,9	999,6
		B	b	50,7	109,7	71,9	7,5	46,8	49,6	61,9	1879,6
			b	45,7	149,7	50,9	7,2	56,8	48,6	57,9	1499,6
			b	44,7	119,7	58,9	7,6	51,8	48,6	57,9	1609,6
		C	c	37,7	80,7	111,9	7,4	38,8	50,6	63,9	1889,6
			c	47,7	80,7	111,9	7,6	36,8	42,6	52,9	1899,6
			c	43,7	93,7	51,9	7,7	40,8	44,6	57,9	1709,6
		D	d	12,7	42,7	211,9	7,4	25,8	54,6	62,9	1879,6
			d	6,1	17,7	91,9	7,4	17,8	42,6	48,9	1779,6
			d	15,7	61,7	171,9	7,5	30,8	46,6	53,9	1599,6
		E	e	2,2	6,2	141,9	7,2	15,8	42,6	46,9	1829,6
			e	2,9	6,8	131,9	7,4	14,8	49,6	53,9	1779,6
			e	7,0	30,7	161,9	7,3	18,8	42,6	44,9	1789,6
		F	f	9,4	29,7	221,9	7,2	18,8	40,6	48,9	1969,6
			f	12,7	52,7	311,9	7,2	22,8	46,6	59,9	1949,6
			f	13,7	48,7	141,9	7,2	18,8	39,6	48,9	1899,6
	2 days	A	a	109,7	239,7	30,9	7,6	56,8	29,6	31,9	629,6
			a	98,7	229,7	40,9	7,6	67,8	35,6	39,9	769,6
			a	109,7	249,7	33,9	7,5	57,8	28,6	32,9	649,6
		B	b	47,7	109,7	80,9	7,8	39,8	35,6	42,9	1599,6
			b	43,7	159,7	50,9	7,4	42,8	28,6	33,9	1129,6
			b	41,7	119,7	50,9	7,3	40,8	33,6	38,9	1439,6
		C	c	27,7	75,7	121,9	7,5	29,8	38,6	46,9	1779,6
			c	36,7	77,7	101,9	7,7	27,8	27,6	32,9	1639,6
			c	37,7	91,7	45,9	7,4	31,8	33,6	42,9	1649,6
		D	d	8,6	52,7	101,9	7,4	20,8	32,6	37,9	1369,6
			d	4,7	21,7	84,9	7,3	14,8	31,6	34,9	1639,6
			d	13,7	68,7	111,9	7,3	24,8	32,6	37,9	1329,6
		E	e	3,8	7,2	79,9	7,1	12,8	31,6	33,9	1739,6
			e	1,4	5,8	49,9	7,2	8,8	35,6	36,9	1819,6
			e	4,7	36,7	80,9	7,2	11,8	28,6	28,9	1499,6
		F	f	5,3	30,7	111,9	7,2	13,8	28,6	31,9	1769,6
			f	6,3	51,7	161,9	7,1	16,8	31,6	40,9	1759,6
			f	9,6	53,7	111,9	7,3	13,8	25,6	31,9	1689,6

3 days	A	a	119,7	269,7	33,9	7,4	51,8	21,6	21,9	479,6
		a	109,7	249,7	43,9	7,5	57,8	22,6	23,9	499,6
		a	119,7	259,7	36,9	7,8	47,8	16,6	18,9	399,6
	B	b	37,7	109,7	71,9	7,2	32,8	22,6	24,9	1419,6
		b	47,7	159,7	42,9	7,8	33,8	18,6	21,9	829,6
		b	38,7	109,7	49,9	7,4	32,8	21,6	23,9	1129,6
	C	c	23,7	70,7	241,9	7,1	24,8	27,6	30,9	1649,6
		c	31,7	71,7	76,9	7,3	21,8	16,6	18,9	1389,6
		c	35,7	90,7	57,9	7,4	26,8	25,6	31,9	1469,6
	D	d	8,0	57,7	71,9	6,8	17,8	20,6	22,9	1049,6
		d	3,5	20,7	44,9	7,1	11,8	21,6	22,9	1329,6
		d	12,7	73,7	79,9	7,2	19,8	21,6	22,9	1039,6
	E	e	1,5	8,0	49,9	6,7	8,8	21,6	21,9	1449,6
		e	0,9	5,2	24,9	6,9	5,8	24,6	23,9	1619,6
		e	3,2	36,7	43,9	6,8	7,8	20,6	18,9	1329,6
	F	f	4,0	34,7	73,9	6,9	10,8	21,6	22,9	1579,6
		f	4,3	50,7	111,9	6,8	13,8	22,6	26,9	1569,6
		f	9,5	61,7	101,9	7,0	11,8	16,6	19,9	1349,6
1 week	A	a	79,7	309,7	40,9	7,5	67,8	26,6	32,9	479,6
		a	90,7	279,7	37,9	7,6	63,8	24,6	31,9	509,6
		a	80,7	279,7	31,9	7,6	51,8	22,6	31,9	439,6
	B	b	42,7	109,7	48,9	7,6	30,8	17,6	18,9	1309,6
		b	58,7	169,7	30,9	7,5	37,8	28,6	35,9	949,6
		b	34,7	119,7	50,9	7,7	29,8	24,6	29,9	1059,6
	C	c	17,7	63,7	81,9	7,5	23,8	29,6	25,9	1699,6
		c	33,7	70,7	34,9	7,7	18,8	18,6	22,9	1219,6
		c	31,7	109,7	53,9	7,8	34,8	37,6	39,9	1709,6
	D	d	3,2	53,7	43,9	7,5	19,8	31,6	27,9	1209,6
		d	1,2	16,7	19,9	7,5	10,8	28,6	25,9	1289,6
		d	9,7	94,7	121,9	7,5	24,8	23,6	28,9	819,6
	E	e	0,3	3,4	1,9	7,1	6,8	29,6	18,9	1539,6
		e	0,5	2,9	1,5	7,3	5,2	29,6	20,9	1619,6
		e	1,1	38,7	19,9	7,4	9,8	23,6	17,9	1189,6
	F	f	0,6	31,7	48,9	7,0	6,8	22,6	18,9	1539,6
		f	1,8	48,7	65,9	7,3	15,8	28,6	28,9	1629,6
		f	3,0	56,7	60,9	7,3	12,8	27,6	29,9	1619,6
5 weeks	A	a	63,7	399,7	43,9	7,1	94,8	40,6	55,9	539,6
		a	75,7	439,7	67,9	7,0	125,8	56,6	94,9	809,6
		a	66,7	469,7	50,9	7,0	115,8	65,6	109,9	899,6
	B	b	27,7	249,7	47,9	7,3	60,8	35,6	49,9	1379,6
		b	32,7	289,7	43,9	7,0	74,8	67,6	99,9	1299,6
		b	23,7	239,7	49,9	7,1	72,8	62,6	90,9	1619,6
	C	c	14,7	109,7	22,9	7,6	29,8	36,6	55,9	1579,6
		c	18,7	159,7	50,9	7,3	49,8	47,6	68,9	1709,6
		c	15,7	189,7	68,9	7,3	59,8	71,6	91,9	1989,6

D	d	6,2	119,7	151,9	7,5	42,8	56,6	60,9	1559,6
	d	4,6	45,7	171,9	7,6	35,8	63,6	55,9	1889,6
	d	9,1	149,7	441,9	7,3	57,8	54,6	67,9	1479,6
E	e	2,4	34,7	381,9	7,3	28,8	48,6	44,9	1819,6
	e	0,8	12,7	78,9	7,3	22,8	55,6	45,9	2439,6
	e	2,4	56,7	191,9	7,3	20,8	30,6	36,9	1319,6
F	f	1,7	38,7	281,9	7,3	25,8	38,6	54,9	1989,6
	f	1,0	61,7	221,9	7,1	39,8	64,6	65,9	2699,6
	f	2,4	88,7	171,9	7,2	31,8	50,6	62,9	2079,6

Registrert **2014-12-16 13:33**
Utstedt **2014-12-23**

Lindum Egge AS
Katarzyna Krajewska
Lerpevn 155

N-3036 Drammen
Norge

Prosjekt
Bestnr

Analyse av faststoff

Deres prøvenavn **Oredalen Mineral**
Jord

Labnummer N00343365

Analyse	Resultater	Usikkerhet (±)	Enhet	Metode	Utført	Sign
Tørrstoff (DK)	**82.1**	8.21	%	1	1	JIBJ
As (Arsen)	**14**	1.96	mg/kg TS	1	1	JIBJ
Cd (Kadmium)	**0.52**	0.0728	mg/kg TS	1	1	JIBJ
Cr (Krom)	**19**	2.66	mg/kg TS	1	1	JIBJ
Cu (Kopper)	**907**	126.98	mg/kg TS	1	1	JIBJ
Hg (Kvikksølv)	**0.16**	0.0224	mg/kg TS	1	1	JIBJ
Ni (Nikkel)	**20**	2.8	mg/kg TS	1	1	JIBJ
Pb (Bly)	**3290**	460.6	mg/kg TS	1	1	JIBJ
Zn (Sink)	**292**	29.2	mg/kg TS	1	1	JIBJ
Sb (Antimon)*	**93**		mg/kg TS	1	1	JIBJ
PCB 28	**<0.0010**		mg/kg TS	1	1	JIBJ
PCB 52	**0.0011**	0.00022	mg/kg TS	1	1	JIBJ
PCB 101	**0.0072**	0.00144	mg/kg TS	1	1	JIBJ
PCB 118	**0.0025**	0.0005	mg/kg TS	1	1	JIBJ
PCB 138	**0.0078**	0.00156	mg/kg TS	1	1	JIBJ
PCB 153	**0.0052**	0.00104	mg/kg TS	1	1	JIBJ
PCB 180	**0.0025**	0.0005	mg/kg TS	1	1	JIBJ
Sum PCB-7*	**0.0263**		mg/kg TS	1	1	JIBJ
Naftalen	**0.057**	0.0171	mg/kg TS	1	1	JIBJ
Acenaftylen	**0.16**	0.048	mg/kg TS	1	1	JIBJ
Acenaften	**0.023**	0.0069	mg/kg TS	1	1	JIBJ
Fluoren	**0.042**	0.0126	mg/kg TS	1	1	JIBJ
Fenantren	**0.35**	0.105	mg/kg TS	1	1	JIBJ
Antracen	**0.13**	0.039	mg/kg TS	1	1	JIBJ
Fluoranten	**0.84**	0.252	mg/kg TS	1	1	JIBJ
Pyren	**0.76**	0.228	mg/kg TS	1	1	JIBJ
Benso(a)antracen^	**0.53**	0.159	mg/kg TS	1	1	JIBJ
Krysen^	**0.61**	0.183	mg/kg TS	1	1	JIBJ
Benso(b+j)fluoranten^	**0.63**	0.189	mg/kg TS	1	1	JIBJ
Benso(k)fluoranten^	**0.48**	0.144	mg/kg TS	1	1	JIBJ
Benso(a)pyren^	**0.64**	0.192	mg/kg TS	1	1	JIBJ
Dibenso(ah)antracen^	**0.14**	0.042	mg/kg TS	1	1	JIBJ
Benso(ghi)perylen	**0.37**	0.111	mg/kg TS	1	1	JIBJ
Indeno(123cd)pyren^	**0.37**	0.111	mg/kg TS	1	1	JIBJ
Sum PAH-16*	**6.13**		mg/kg TS	1	1	JIBJ
Bensen	**<0.010**		mg/kg TS	1	1	JIBJ
Toluen	**<0.010**		mg/kg TS	1	1	JIBJ
Etylbensen	**<0.010**		mg/kg TS	1	1	JIBJ

Deres prøvenavn	**Oredalen Mineral** **Jord**					
Labnummer	N00343365					
Analyse	**Resultater**	**Usikkerhet (±)**	**Enhet**	**Metode**	**Utført**	**Sign**
Xylener	**<0.010**		mg/kg TS	1	1	JIBJ
Sum BTEX*	**n.d.**		mg/kg TS	1	1	JIBJ
Fraksjon C5-C6	**<2.5**		mg/kg TS	1	1	JIBJ
Fraksjon >C6-C8	**<7.0**		mg/kg TS	1	1	JIBJ
Fraksjon >C8-C10	**<10**		mg/kg TS	1	1	JIBJ
Fraksjon >C10-C12	**<10**		mg/kg TS	1	1	JIBJ
Fraksjon >C12-C16	**<10**		mg/kg TS	1	1	JIBJ
Sum >C12-C35*	**150**		mg/kg TS	1	1	JIBJ
Fraksjon >C16-C35	**150**	45	mg/kg TS	1	1	JIBJ
TOC	**4.7**	0.705	% TS	2	1	JIBJ

Deres prøvenavn	**Oredalen organisk** **Jord**
Labnummer	N00343366

Analyse	Resultater	Usikkerhet (±)	Enhet	Metode	Utført	Sign
Tørrstoff (DK)	**41.7**	4.17	%	1	1	JIBJ
As (Arsen)	**3**	0.42	mg/kg TS	1	1	JIBJ
Cd (Kadmium)	**0.24**	0.04	mg/kg TS	1	1	JIBJ
Cr (Krom)	**20**	2.8	mg/kg TS	1	1	JIBJ
Cu (Kopper)	**316**	44.24	mg/kg TS	1	1	JIBJ
Hg (Kvikksølv)	**0.12**	0.02	mg/kg TS	1	1	JIBJ
Ni (Nikkel)	**13**	1.82	mg/kg TS	1	1	JIBJ
Pb (Bly)	**1500**	210	mg/kg TS	1	1	JIBJ
Zn (Sink)	**96**	9.6	mg/kg TS	1	1	JIBJ
Sb (Antimon)*	**26**		mg/kg TS	1	1	JIBJ
PCB 28	**<0.0010**		mg/kg TS	1	1	JIBJ
PCB 52	**<0.0010**		mg/kg TS	1	1	JIBJ
PCB 101	**<0.0010**		mg/kg TS	1	1	JIBJ
PCB 118	**<0.0010**		mg/kg TS	1	1	JIBJ
PCB 138	**<0.0010**		mg/kg TS	1	1	JIBJ
PCB 153	**<0.0010**		mg/kg TS	1	1	JIBJ
PCB 180	**<0.0010**		mg/kg TS	1	1	JIBJ
Sum PCB-7*	**n.d.**		mg/kg TS	1	1	JIBJ
Naftalen	**<0.010**		mg/kg TS	1	1	JIBJ
Acenaftylen	**0.012**	0.0036	mg/kg TS	1	1	JIBJ
Acenaften	**<0.010**		mg/kg TS	1	1	JIBJ
Fluoren	**<0.010**		mg/kg TS	1	1	JIBJ
Fenantren	**0.025**	0.0075	mg/kg TS	1	1	JIBJ
Antracen	**<0.010**		mg/kg TS	1	1	JIBJ
Fluoranten	**0.067**	0.0201	mg/kg TS	1	1	JIBJ
Pyren	**0.053**	0.0159	mg/kg TS	1	1	JIBJ
Benso(a)antracen^	**0.030**	0.009	mg/kg TS	1	1	JIBJ
Krysen^	**0.053**	0.0159	mg/kg TS	1	1	JIBJ
Benso(b+j)fluoranten^	**0.069**	0.0207	mg/kg TS	1	1	JIBJ
Benso(k)fluoranten^	**0.040**	0.012	mg/kg TS	1	1	JIBJ
Benso(a)pyren^	**0.046**	0.0138	mg/kg TS	1	1	JIBJ
Dibenso(ah)antracen^	**0.019**	0.0057	mg/kg TS	1	1	JIBJ
Benso(ghi)perylen	**0.039**	0.0117	mg/kg TS	1	1	JIBJ
Indeno(123cd)pyren^	**0.039**	0.0117	mg/kg TS	1	1	JIBJ
Sum PAH-16*	**0.492**		mg/kg TS	1	1	JIBJ
Bensen	**<0.010**		mg/kg TS	1	1	JIBJ
Toluen	**<0.010**		mg/kg TS	1	1	JIBJ
Etylbensen	**<0.010**		mg/kg TS	1	1	JIBJ
Xylener	**<0.010**		mg/kg TS	1	1	JIBJ
Sum BTEX*	**n.d.**		mg/kg TS	1	1	JIBJ
Fraksjon C5-C6	**<2.5**		mg/kg TS	1	1	JIBJ
Fraksjon >C6-C8	**<7.0**		mg/kg TS	1	1	JIBJ
Fraksjon >C8-C10	**<10**		mg/kg TS	1	1	JIBJ
Fraksjon >C10-C12	**<10**		mg/kg TS	1	1	JIBJ
Fraksjon >C12-C16	**<10**		mg/kg TS	1	1	JIBJ
Sum >C12-C35*	**78.0**		mg/kg TS	1	1	JIBJ
Fraksjon >C16-C35	**78**	23.4	mg/kg TS	1	1	JIBJ
TOC	**12.6**	1.89	% TS	2	1	JIBJ

Kromatogram: Humus/annet organisk materiale kan ikke utelukkes

* etter parameternavn indikerer uakkreditert analyse.
n.d. betyr ikke påvist.
n/a betyr ikke analyserbart.
< betyr mindre enn.
> betyr større enn.

	Metodespesifikasjon
1	**Bestemmelse av Normpakke (liten) for jord.** Metode: Metaller: DS259/ICP Tørrstoff: DS 204 PCB-7: GC/MS/SIM PAH: REFLAB 4:2008 BTEX: GC/MS/pentan Hydrokarboner: >C5-C6 GC/MS/SIM >C6-C35 REFLAB 1/VKI 2010 Rapporteringsgrenser: Metaller: LOD 0,01-5 mg/kg TS Tørrstoff: LOD 0,1 % PCB-7: LOD 0,001 mg/kg TS PAH: 0,01-0,04 mg/kg TS Måleusikkerhet: Metaller: relativ usikkerhet 14 % Tørrstoff: relativ usikkerhet 10 % PCB-7: relativ usikkerhet 20 % PAH: relativ usikkerhet 40 %
2	**Bestemmelse av TOC i jord** Metode: DS/EN ISO 13137 Måleprinsipp: TOC bestemmes ved å måle TC og IC (TOC = TC - IC). TC Bestemmelse av TC foregår ved brenning av prøve ved 1100 °C gjennom en katalysator, hvor all uorganisk og organisk materiale bli oksidert for å danne CO2. CO2 innholdet måles derretter i en IR-detektor. IC Bestemmelse av IC foregår ved å tilsette syre til prøven for derved å danne CO2 ved dekomponering av uorganisk komponenter. CO2 innholdet blir målt i samme IR-detektor. Rapporteringsgrenser: LOD 500 mg/kg TS

	Godkjenner
JIBJ	Jan Inge Bjørnengen

	Underleverandør[1]	
1	Ansvarlig laboratorium:	ALS Denmark A/S, Bakkegårdsvej 406A, 3050 Humlebæk, Danmark

---

[1] Utførende teknisk enhet (innen ALS Laboratory Group) eller eksternt laboratorium (underleverandør).

	Underleverandør[1]
	Akkreditering: DANAK, registreringsnr. 361

Måleusikkerheten angis som en utvidet måleusikkerhet (etter definisjon i "Evaluation of measurement data – Guide to the expression of uncertainty in measurement", JCGM 100:2008 Corrected version 2010) beregnet med en dekningsfaktor på 2 noe som gir et konfidensinterval på om lag 95%.

Måleusikkerhet fra underleverandører angis ofte som en utvidet usikkerhet beregnet med dekningsfaktor 2. For ytterligere informasjon, kontakt laboratoriet.

Denne rapporten får kun gjengis i sin helhet, om ikke utførende laboratorium på forhånd har skriftlig godkjent annet.

Angående laboratoriets ansvar i forbindelse med oppdrag, se aktuell produktkatalog eller vår webside www.alsglobal.no

Kopi sendt til:
Thomas Henriksen, Lindum Egge AS, N-3036 Drammen, Norge.
Hilmar Sævarsson, Lindum Egge AS, N-3036 Drammen, Norge.
+
frank.gabrielsen@lindum.no
thomas.henriksen@lindum.no

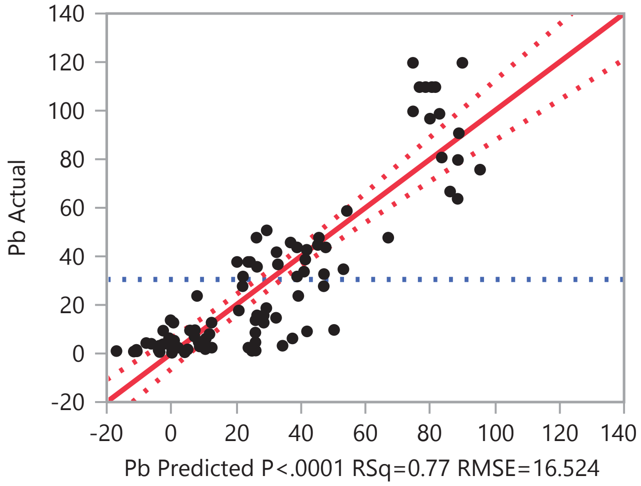
Den digitalt signert PDF-fil representerer den opprinnelige rapporten. Eventuelle utskrifter er å anse som kopier.

Jan-Inge Bjornengen
2014.12.23 12:46:06
Client Service
jan-inge.bjornengen@alsglobal.com

Response Pb

Whole Model

Actual by Predicted Plot



Summary of Fit

RSquare	0.768657
RSquare Adj	0.760586
Root Mean Square Error	16.52375
Mean of Response	30.48444
Observations (or Sum Wgts)	90

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	78017.26	26005.8	95.2473
Error	86	23480.94	273.0	**Prob > F**
C. Total	89	101498.20		<.0001 *

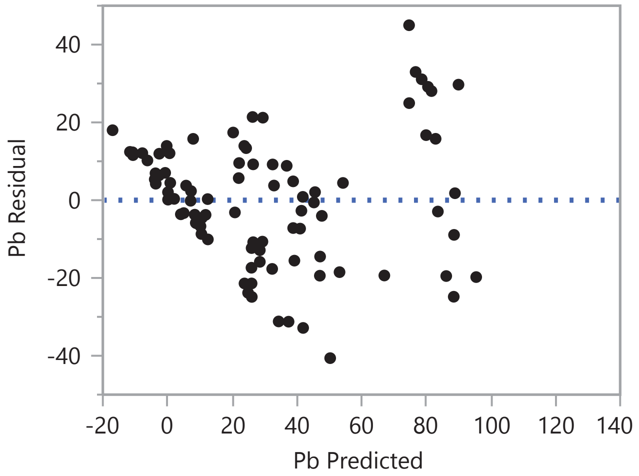
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-223.2765	54.82627	-4.07	0.0001 *
pH	37.969829	7.285368	5.21	<.0001 *
DOC	0.6318367	0.082868	7.62	<.0001 *
SO4	-0.032762	0.004358	-7.52	<.0001 *

Response Pb

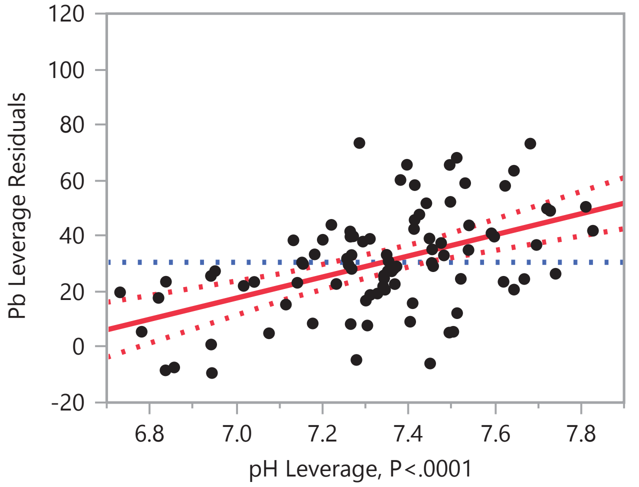
Whole Model

Residual by Predicted Plot



pH

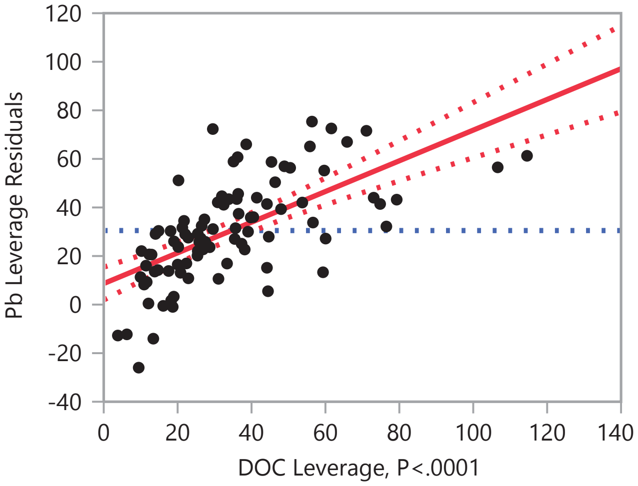
Leverage Plot



Response Pb

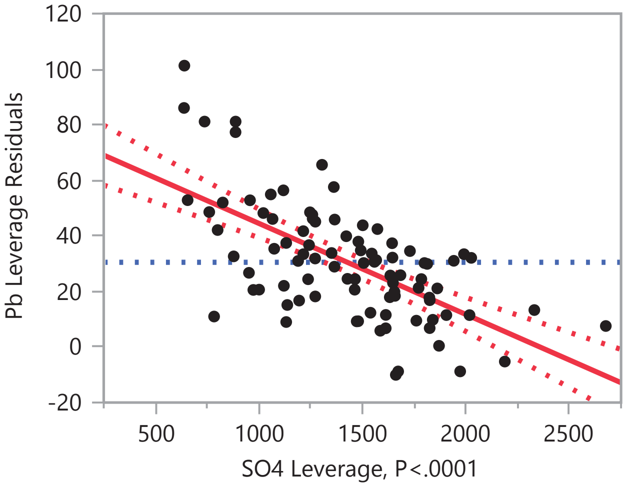
DOC

Leverage Plot



SO4

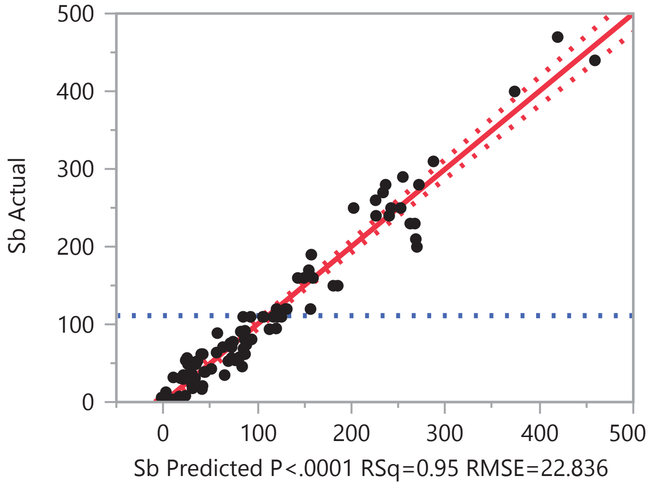
Leverage Plot



Response Sb

Whole Model

Actual by Predicted Plot



Summary of Fit

RSquare	0.950303
RSquare Adj	0.94916
Root Mean Square Error	22.83636
Mean of Response	111.1989
Observations (or Sum Wgts)	90

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	2	867565.53	433783	831.7989
Error	87	45370.46	521	**Prob > F**
C. Total	89	912935.99		<.0001 *

Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	86	44970.015	522.907	1.3058
Pure Error	1	400.445	400.445	**Prob > F**
Total Error	87	45370.460		0.6160
				Max RSq
				0.9996

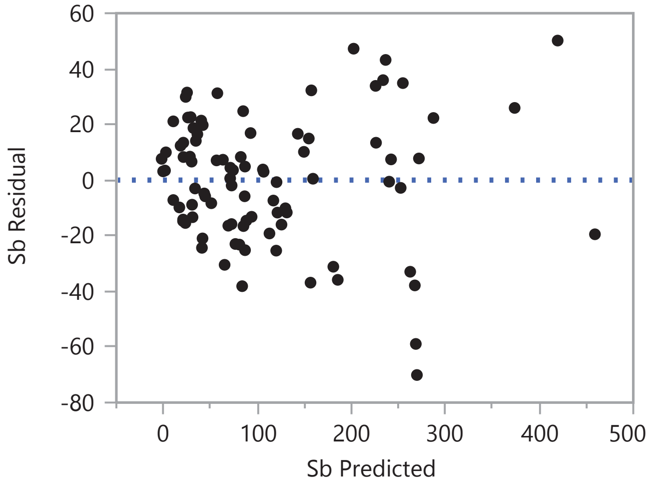
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	92.950707	11.13189	8.35	<.0001 *
DOC	3.3508231	0.113973	29.40	<.0001 *
SO4	-0.0683	0.005938	-11.50	<.0001 *

Response Sb

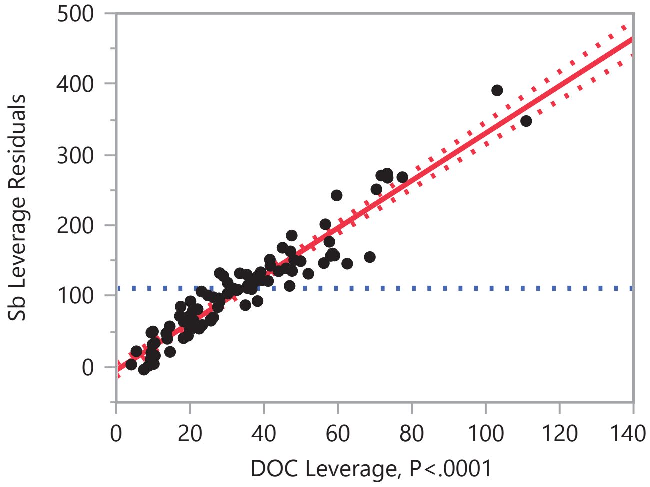
Whole Model

Residual by Predicted Plot



DOC

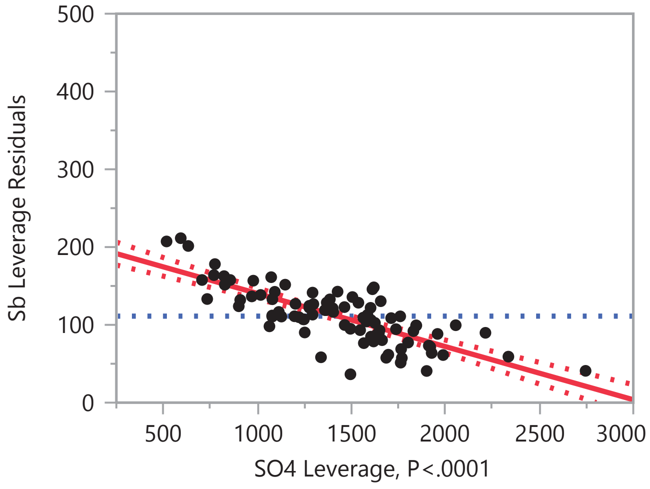
Leverage Plot



Response Sb

SO4

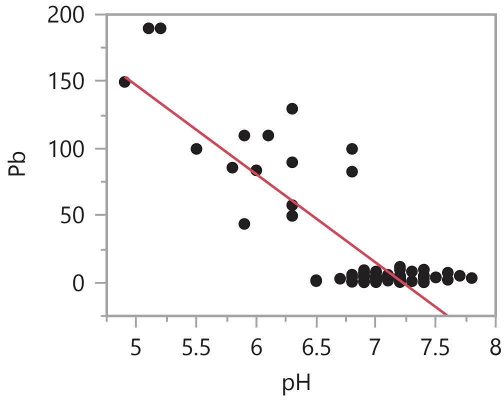
Leverage Plot



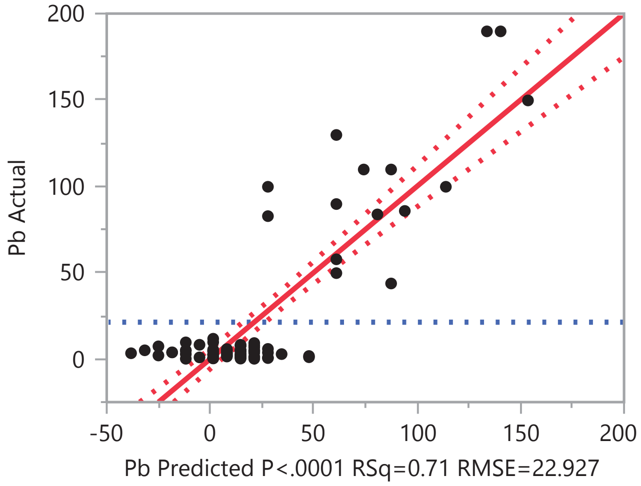
Response Pb

Whole Model

Regression Plot



Actual by Predicted Plot



Summary of Fit

RSquare	0.714361
RSquare Adj	0.71096
Root Mean Square Error	22.92686
Mean of Response	21.28837
Observations (or Sum Wgts)	86

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	1	110425.12	110425	210.0771
Error	84	44153.85	526	**Prob > F**
C. Total	85	154578.97		<.0001 *

Response Pb

Whole Model

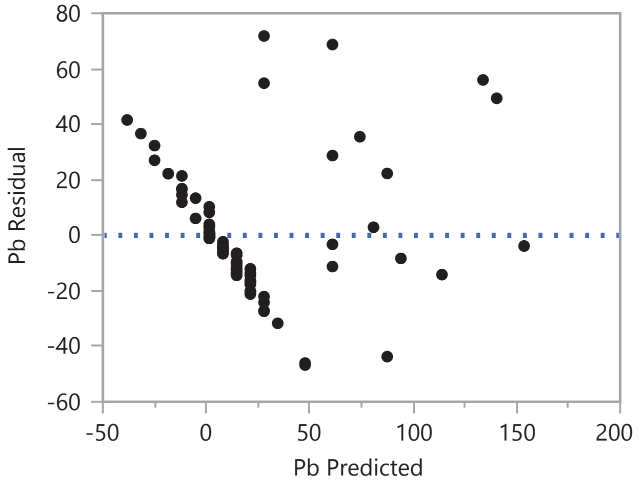
Lack Of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	20	26942.739	1347.14	5.0094
Pure Error	64	17211.108	268.92	**Prob > F**
Total Error	84	44153.846		<.0001 *
				Max RSq
				0.8887

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	477.68073	31.58519	15.12	<.0001 *
pH	-66.14382	4.56352	-14.49	<.0001 *

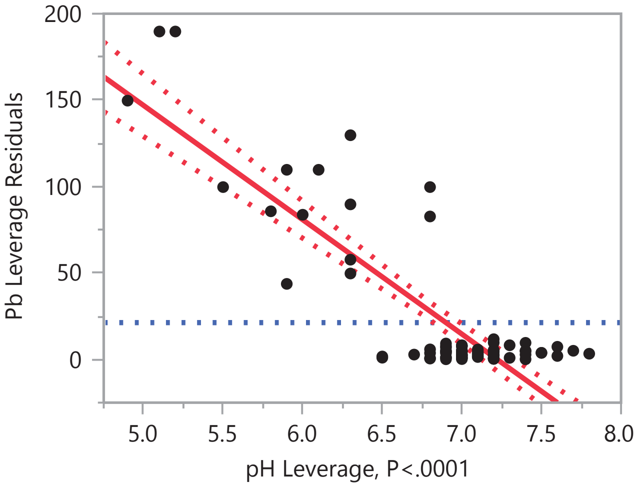
Residual by Predicted Plot



Response Pb

pH

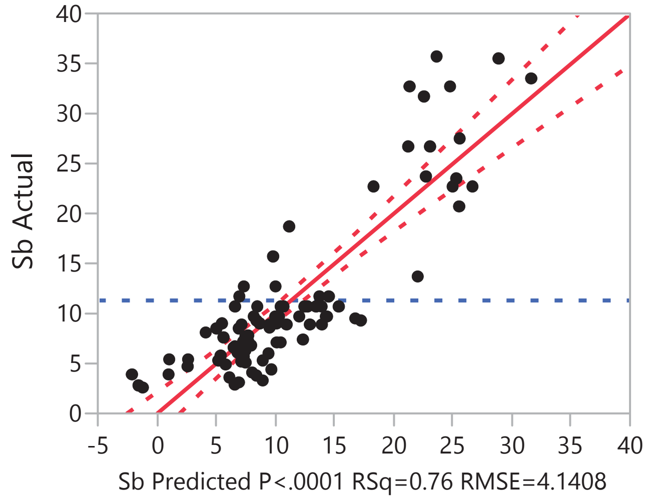
Leverage Plot



Response Sb

Whole Model

Actual by Predicted Plot



Summary of Fit

RSquare	0.756197
RSquare Adj	0.747693
Root Mean Square Error	4.140821
Mean of Response	11.33111
Observations (or Sum Wgts)	90

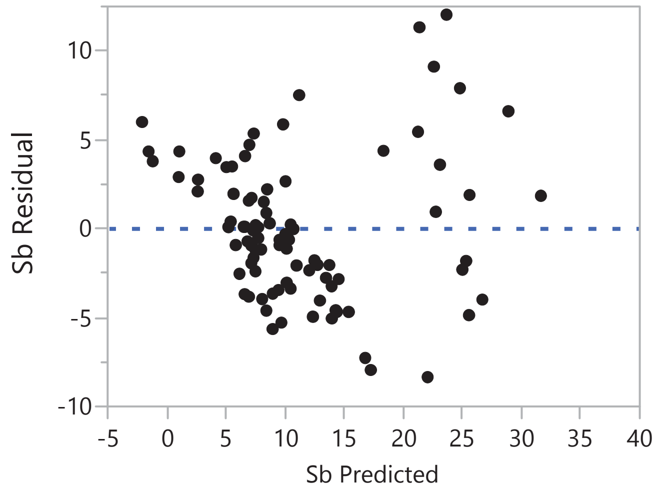
Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	4573.7029	1524.57	88.9148
Error	86	1474.5900	17.15	**Prob > F**
C. Total	89	6048.2929		<.0001*

Parameter Estimates

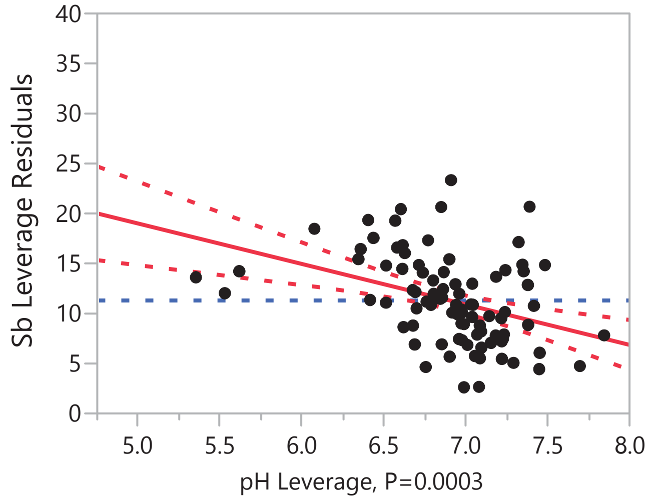
Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	39.315112	7.407767	5.31	<.0001*
pH	-4.049409	1.076581	-3.76	0.0003*
DOC	0.8063981	0.113554	7.10	<.0001*
SO4	-0.007807	0.001362	-5.73	<.0001*

Residual by Predicted Plot



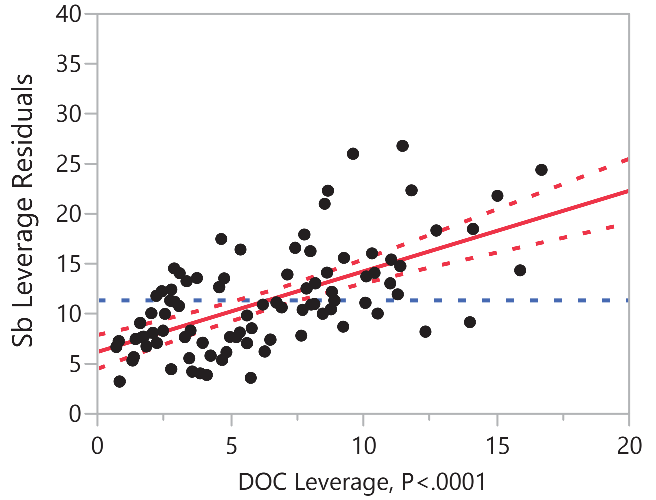
pH

Leverage Plot



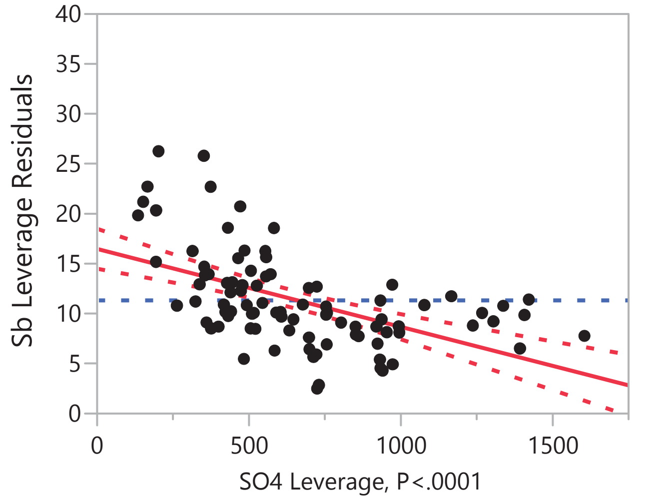
DOC

Leverage Plot



SO4

Leverage Plot



Norwegian University
of Life Sciences

Postboks 5003
NO-1432 Ås, Norway
+47 67 23 00 00
www.nmbu.no