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# Performance of road side gully pots in Trondheim for sediment removal

- evaluated based landuse and annual traffic loads

Master's thesis in Civil and Environmental Engineering

January 2020

**NTNU**  
Norwegian University of Science and Technology  
Faculty of Engineering  
Department of Civil and Environmental Engineering



Norwegian University of  
Science and Technology





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Master's thesis in Civil and Environmental Engineering

Supervisor: Tone Merete Muthanna, IBM

Co-supervisor: Prof. Dr.-Ing. Rita Hilliges, UAS Augsburg

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Norwegian University of Science and Technology

Faculty of Engineering

Department of Civil and Environmental Engineering

## Preface

This work has been carried out 2<sup>nd</sup> September 2019 to 26<sup>th</sup> January 2020 as the final assignment for the Degree of Master of Engineering at the Department of Civil and Environmental Engineering of the Norwegian University of Science and Technology in Trondheim, Norway. Home institution is the University of Applied Sciences in Augsburg, Germany.

The present study including research and laboratory work has been executed under the Supervision of Associate Professor Tone Merete Muthanna, University of Science and Technology Trondheim and under Co-supervision of Professor Dr.-Ing. Rita Hilliges, University of Applied Sciences Augsburg. Laboratory work was conducted in the analytical and hydrology laboratory at the Department of Civil and Environmental Engineering at NTNU.

The thesis has been conducted in collaboration with the Trondheim Municipality and the University of Applied Sciences in Augsburg in order to give an overview of the actual performance and the degree of heavy metal contamination of the existing road side gully pots as a first part of the stormwater system in Trondheim. Results can be used as a basis for further decisions regarding evaluation and selecting processes for the Municipality Trondheim. Based on consequences of current and further growing urbanization and climate change, treatment of urban stormwater before releasing into natural water streams, is gaining more and more in relevance and will force constant developments.

The structure of this work differs from the normal design of a Master thesis. It represents a manuscript for a potential further scientific paper in cooperation with another student from NTNU. Results not included in the manuscript can be found in the appendices.

I declare that I have written the present thesis by myself and without any external other support except the cited sources and literatures. I marked and listed literature I referred to, both literally and in context. I also state that this work has not been submitted or published in this or in a similar form before. At no time it was content of an examination or course content.

I would like to address special thanks to Associate Professor Tone Merete Muthanna for the professional supervision and support throughout the project. I also would like to extend special thanks to Professor Dr.-Ing. Rita Hilliges from my home institute in Germany for the excellent co-supervision and giving advice at any time.

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Trondheim, 26<sup>th</sup> January 2020

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## Sammendrag

Sandfangere i veikanten er det første viktige elementet i kloakksystemet som mottar avrenning av veggen. Foruten hydraulisk utladning, er hovedfunksjonen fjerning av forurensede sedimenter av avrenning før de går inn i avløpssystemet (Grottker, 1990). I Trondheim oppnås dette formålet ved rene sedimenteringsprosesser i rundt 15000 sandfanger (Information from Bydrift in Trondheim Kommune, 2019). Tidligere studier har vist at forekomsten av metaller festet til sedimenter øker med synkende partikkelstørrelser (Wang et al., 1998). Uten regelmessig vedlikehold kan disse forurensende partiklene potensielt skylles ut ubehandlet i miljøet, for eksempel i elver (Deletic et al., 2000).

Av denne grunn er kontinuerlig forskning og utvikling av renseanlegg av stor betydning. I dette bidraget undersøker den nåværende studien partikkelstørrelsesfordelingen i sedimenterende senger fra sandfanger i Trondheim, Norge for å evaluere dagens sedimentasjonsytelse for sandfanger i forhold til forskjellige partikkelstørrelser. Partikkelstørrelsesfordelingen bestemmes ved hjelp av en manuell våtsikting for partikler mellom 50 og 2000  $\mu\text{m}$  og en partikkelstørrelsesmåler LS230 for partikler  $<50 \mu\text{m}$ . Fordelingen av de ulike partikkelstørrelsene ble utført både med og uten dispersjon ved hjelp av natriumpyrofosfatdekahydrat. Den organiske fraksjonen innenfor de ulike partikkelstørrelsene ble bestemt ved å varme opp prøvene til  $550 \text{ }^\circ\text{C}$ . Organisk innhold i de siktede fraksjoner, samt tungmetaller og egenvekt ble også analysert. Metodikken er basert på en rekke tester utført før selve analysen. Det ble tatt prøver fra gågater uten motorisert trafikk, det ble tatt sedimentprøver fra klyper kommersielle og boligområder med ulik årlig daglig trafikkbelastning.

Resultatene fra den foreliggende studien viser en bemerkelsesverdig høy andel partikler i  $<50 \mu\text{m}$  området mellom 31 og 37 %. I kontrast har fraksjoner mellom 50 og 2000  $\mu\text{m}$  silpass mellom 3 og 18 %. Partikkelstørrelsesfordelingen var stort sett uavhengig av arealbruken, basert på trafikkfrie og trafikkbærende områder i Trondheim. De høyeste median tungmetallkonsentrasjonene i de siktede fraksjoner ble funnet for begge områdene (trafikkfrie og med trafikk) i de minste partiklene  $<50 \mu\text{m}$  for krom (220 og 155 mg/kg), kobber (91 og 43 mg/kg), bly (44 og 15 mg/kg) og delvis for jern i den trafikkfrie sonen (39944 mg/kg) og for sink i områdene med trafikk (142 mg/kg). For kobber, bly og sink var metallinnholdet i sedimentene høyere fra sedimentasjonssjakter i det fotgjengerfrie sentrum enn på gater.

Disse funnene representerer en vurdering av den faktiske ytelsen til sandfang for sedimentfjerning via ren sedimentasjon, analysert i Trondheim. Det gir også innsikt i forurensningsgraden av tungmetaller i sedimentene som er avsatt. Studieresultatene kan brukes som grunnlag for videre evaluering, valg og utvikling av egnede overvannshåndteringsapparater og forbedre drifts- og vedlikeholdsrutiner.

## Zusammenfassung

Ein Straßenablauf ist das erste wesentliche Element des Entwässerungssystems, das den Straßenabfluss bei Regenereignissen aufnimmt. Neben der hydraulischen Entleerung besteht die Hauptfunktion darin, verschmutzte Sedimente im Straßenabfluss vor dem Eintritt in das weiterführende Kanalsystem zu entfernen (Grottker, 1990). In Trondheim wird dieser Zweck durch reine Sedimentationsprozesse in ca. 15000 Absetzschächten erreicht (Information from Bydrift in Trondheim Kommune, 2019). Frühere Studien haben gezeigt, dass die Menge an partikelgebundenen Schadstoffen, wie z.B. Metallen mit abnehmender Partikelgröße zunimmt (Wang et al., 1998). Ohne ordnungsgemäße Wartung und Reinigung der Straßenabläufe gelangen diese verschmutzten, vor allem kleineren Partikel möglicherweise unbehandelt in das Kanalnetz und anschließend in aufnehmende Gewässer, wie Flüsse oder Seen (Deletic et al., 2000).

Aus diesem Grund ist eine stetige Forschung und Weiterentwicklung von Regenwasserbehandlungsanlagen von großer Bedeutung. Vorliegende Thesis untersucht die Partikelgrößenverteilung in abgelagerten Sedimentbetten von Straßenabläufen im norwegischen Trondheim, um die aktuelle Sedimentationsleistung von Straßenabläufen bezogen auf verschiedene Partikelgrößen zu bewerten. Die Partikelgrößenverteilung wird mit Hilfe einer manuellen Nasssiebung für Partikel zwischen 50 und 2000  $\mu\text{m}$  und einem Partikelgrößenzählerinstrument LS230 für Partikel  $<50 \mu\text{m}$  bestimmt. Die Partikelgrößenverteilung wurde sowohl mit als auch ohne Dispergierung mittels Natriumpyrophosphat Decahydrat durchgeführt. Der organische Anteil innerhalb der gesamten Partikelgrößenbreite wurde durch Erhitzen der Proben auf  $550 \text{ }^\circ\text{C}$  ermittelt. Organischer Anteil in den gesiebten Fraktionen, sowie Schwermetalle und das spezifische Gewicht wurden zudem analysiert. Die methodische Vorgehensweise basiert auf zahlreichen Tests, die vor Beginn der eigentlichen Analyse durchgeführt wurden. Sedimentproben von Straßenabläufen wurden aus Gewerbe- und Wohngebieten mit unterschiedlichen täglichen Verkehrslasten entnommen. Zudem wurden Proben aus Fußgängerbereichen ohne jeglichen Verkehrsanteil untersucht.

Ergebnisse der vorliegenden Studie zeigen einen bemerkenswerten hohen Anteil an Partikeln im Bereich  $<50 \mu\text{m}$  zwischen 31 und 37 %. Im Gegensatz hierzu, weisen Fraktionen zwischen 50 und 2000  $\mu\text{m}$  Siebdurchgänge zwischen 3 und 18 % auf. Die Partikelgrößenverteilung war weitgehend unabhängig von der Flächennutzung, basierend auf verkehrsfreie und verkehrsaufweisende Bereiche Trondheims. Die höchsten Schwermetallkonzentrationen in den gesiebten Fraktionen im Median wurden für bei beide Bereiche (jeweils verkehrsfrei und verkehrsaufweisend) in den kleinsten Partikeln  $<50 \mu\text{m}$  festgestellt für Chrom (220 und 155 mg/kg), Kupfer (91 und 43 mg/kg), Blei (44 und 15 mg/kg) und teilweise für Eisen in der verkehrsfreien

Zone (39944 mg/kg) und für Zink in den verkehrsaufweisenden Bereichen (142 mg/kg). Die Metallgehalte in den Sedimenten waren für Kupfer, Blei und Zink höher von Absetzschächten im fußgängerfreien Stadtzentrum als an Straßen.

Die Arbeit gibt eine Einschätzung der aktuellen Leistung von Straßenabläufen in Trondheim, kontaminierte Partikel durch reine Sedimentation aus dem Straßenablauf zu entfernen. Zudem gibt sie Erkenntnisse über den Verschmutzungsgrad von Schwermetallen in den abgesetzten Sedimenten. Die Studienergebnisse können als Grundlage für die weitere Bewertung, Auswahl und Entwicklung geeigneter Regenwasserbehandlungsanlagen und zur Verbesserung der Betriebs- und Wartungsmaßnahmen verwendet werden.



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## Abbreviations

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|      |                                  |
|------|----------------------------------|
| ADT  | Annual Daily Traffic             |
| DIS  | Dispersed                        |
| HGVT | Heavy Goods Vehicle Traffic      |
| NAT  | Natural                          |
| PSD  | Particle Size Distribution       |
| RSGP | Road Side Gully Pot              |
| SPD  | Sodium Pyrophosphate Decahydrate |
| XRF  | X-Ray Fluorescence               |

# Performance of road side gully pots in Trondheim for sediment removal – evaluated based landuse and annual traffic loads

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## Abstract

Road side gully pots (RSGP) are the first essential element of the sewer system receiving road runoff. Besides hydraulic discharge, main function is removal of polluted sediments of runoff before entering the drainage system. Previous studies have shown that the occurrence of metals attached to sediments increases with decreasing particle sizes. Without proper maintenance these pollutant attached particles are potentially flushed out untreated to receiving water streams.

The current study investigates the particle size distribution (PSD) in sediment beds from RSGP in Trondheim, Norway using wet sieving for particles between 50 and 2000  $\mu\text{m}$  and a Coulter counter LS230 instrument for particles  $<50 \mu\text{m}$ , both with and without a dispersant. Moreover, organic matter, heavy metals via X-ray fluorescence (XRF) analysis and specific gravity of fractions have been studied. Besides pedestrian areas, sediment samples were taken from gully pots in commercial and residential areas with different annual daily traffic (ADT) loads.

A remarkably high proportion of particles  $<50 \mu\text{m}$  between 31 and 37 % was found in contrast to fractions from 50 to 2000  $\mu\text{m}$  showing percentages between 3 and 18 %. PSD was mainly independent from different landuse, evaluated based traffic and no-traffic areas. Highest median heavy metal concentrations in sieved fractions were indicated in the smallest size range  $<50 \mu\text{m}$  in both groups, no traffic and traffic, respectively for Cr (220 and 155 mg/kg), Cu (91 and 43 mg/kg), Pb (44 and 15 mg/kg) and partly for Fe in the no-traffic group (39944 mg/kg) and for Zn in the traffic group (142 mg/kg).

These findings represent an evaluation of the actual sediment removal performance in RSGP via pure sedimentation and the degree of heavy metal contamination in sediment beds analyzed in Trondheim. Study results can be used as a basis for further evaluation, selection and development of suitable stormwater treatment devices and improving operation and maintenance routines.

**Keywords:** landuse, particle size distribution, road side gully pot, sediment removal, traffic load.



# 1 Introduction

Urbanization leads to a significant increase of impervious surface areas, such as roofs and roads. Roads and traffic activities are major source of particles, caused by tire abrasion, brake lining abrasion, leakages of hydrocarbons, residue from combustion and abrasions from roads (Muschack, 1990). These accumulated street sediments have a negative impact on receiving water bodies when flushed from the road by rain events and released from the sewer systems. This can cause not only siltation and clogging of natural waterbodies but also presents a risk on human health, flora and fauna concerning the water cycle. (Muschack, 1990; Sansalone et al., 1998)

In addition, rising occurrence of short-term heavy rain events due to climate change leads to higher runoff volumes. Especially in Norway, there was a 20 % increase of rainfall the last century. A further rise in rainfall of 10 – 20 % in the next 100 years is predicted. (Meteorologisk institutt, 2017) Hence the runoff retention time in RSGP decreases, which is related to a reduced settling efficiency of the particles and a direct discharge to the sewer system. Besides pollution of receiving water streams, the effectiveness of sewer systems can be reduced due to reduction of pipe cross sections caused by adhesion of sediments (Deletic et al., 2000).

Urban road runoff shows a broad size range consisting of organic and inorganic contaminants in dissolved, particulate and particulate-bound form including heavy metals, nutrients (Ball et al., 1998), de-icing salt (Sansalone and Buchberger, 1997) and Polycyclic Aromatic Hydrocarbons (PAH) (Bian and Zhu, 2009). Studies have shown that the occurrence of these contaminants varies, both in quantity and size distribution, with respect to landuse, traffic intensity and weather conditions (Miguel et al., 1997), (Bian and Zhu, 2009).

Both, hydraulic discharge and sediment removal are two operation tasks of a RSGP as the first part of the sewer system receiving road runoff (Grottker, 1990). Sediment removal is affected by several factors: Constitution of gully pots (Tang et al., 2016), (Post et al., 2016); retention time (Butler et al., 1995), (Tang et al., 2016); frequency of maintenance (Butler et al., 1995) regarding especially the first flush phenomenon and possible resuspension (Morgan et al., 2017), (Deletic et al., 2000); flow rate (Butler and Karunaratne, S. H. P. G., 1995), (Tang et al., 2016) and PSD (Sansalone et al., 1998), (Charters et al., 2015).

Sediment capture performance decreases with small particles (Butler and Karunaratne, S. H. P. G., 1995) and specific gravity (Ciccarello et al., 2012) regarding settling velocity (Tang et al., 2016). As a consequence, small particles tend to be more easily flushed towards receiving water streams. Lacking maintenance of RSGP reinforces the reduced sedimentation capability of small fractions due to possible resuspension and decreasing settling volumes. Furthermore,

smaller particles are attached by a higher bulk of heavy metals (Wang et al., 1998) due to the larger specific surface area of small particles (Sansalone et al., 1998).

Field studies investigating sediment beds of RSGP show a significant variance in PSD with median particle sizes from 21  $\mu\text{m}$  to 3000  $\mu\text{m}$ . However, there is the tendency of most sediment bed particles in reviewed literature being in the range between 200  $\mu\text{m}$  and 300  $\mu\text{m}$ . (Pratt and Adams, 1984), (Grottker, 1990), (Deletic et al., 2000), (Jartun et al., 2008), (Karlsson and Viklander, 2008), (Poletto et al., 2009)

Based on known influencing spatial and temporal factors (Monrabal-Martinez et al., 2018) in relation to different places and landuse and the variation of applied analytical methods, PSD of RSGP sediments need to be further observed in Trondheim. Furthermore, Clegg et al. (1992) reported that the composition of sediments show a broad variability with different properties with respect to organic content or specific gravity.

In Trondheim a large amount of gravel and sand is applied during winter operation. The applied amounts vary in the different winter seasons. But on average, about 17000 t of gravel and sand are used per season. (Information from Bydrift in Trondheim Kommune, 2019) Most of the anti-slip agents might end up in RSGP, depending on frequency of street sweeping (Sartor and Boyd, 1972).

In order to provide a basis of knowledge about the actual sediment removal performance of RSGP and the degree of contamination by heavy metals in sediment beds in the municipality of Trondheim, the present fieldwork-based study addresses following research questions:

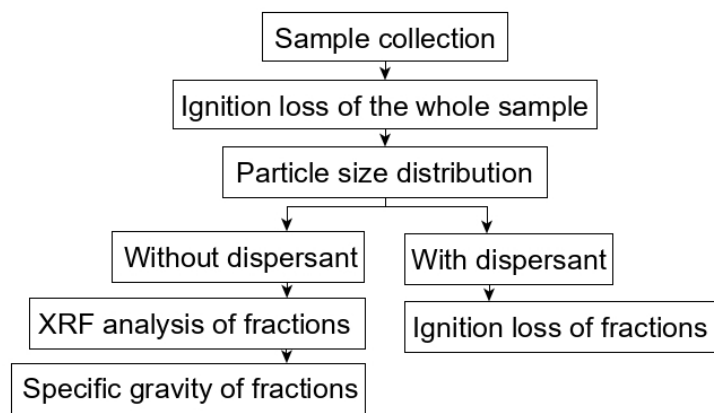
- What is the characterization of the particle size distribution in sediment beds in gully pots in Trondheim in areas with different landuse and traffic loads?
- To which extent are the sediment beds contaminated by heavy metals and is there a difference between different landuse and traffic loads?
- What suggestions can be derived for prospective stormwater treatment decisions for the municipality of Trondheim based on the findings from part 1 and 2?

The present study has been carried out in Trondheim, in the middle of Norway. Sample collection was performed between October and December 2019. However, temporal and seasonal changes, such as snowfall and snow melting events, affiliated salting and vegetation with related potential impact on PSD in sediment beds have been neglected for this investigation. Due to the fact, that sedimentation performance is dependent on the size, shape and hence on the distance wherein particles can settle (Tang et al., 2016) larger devices, such as detention ponds or catch basins are not considered for this paper.

## 2 Materials and Methods

The present study uses fieldwork-gained data to determine PSD in RSGP sediment beds based on different landuse and traffic loads in Trondheim.

Therefore, in total 14 sediment samples were collected and analyzed subsequently in the laboratory. Following analysis have been conducted: a) PSD using manual wet sieving for particles between 50 and 2000  $\mu\text{m}$  and a Coulter counter instrument LS230 for particles  $<50 \mu\text{m}$  – with and without using Sodium pyrophosphate decahydrate (SPD) as a dispersant, b) Ignition loss, c) Heavy metals Cr, Cu, Fe, Pb and Zn by using a X-ray fluorescence (XRF) analyzer and d) Specific gravity of fractions (see Figure 1).

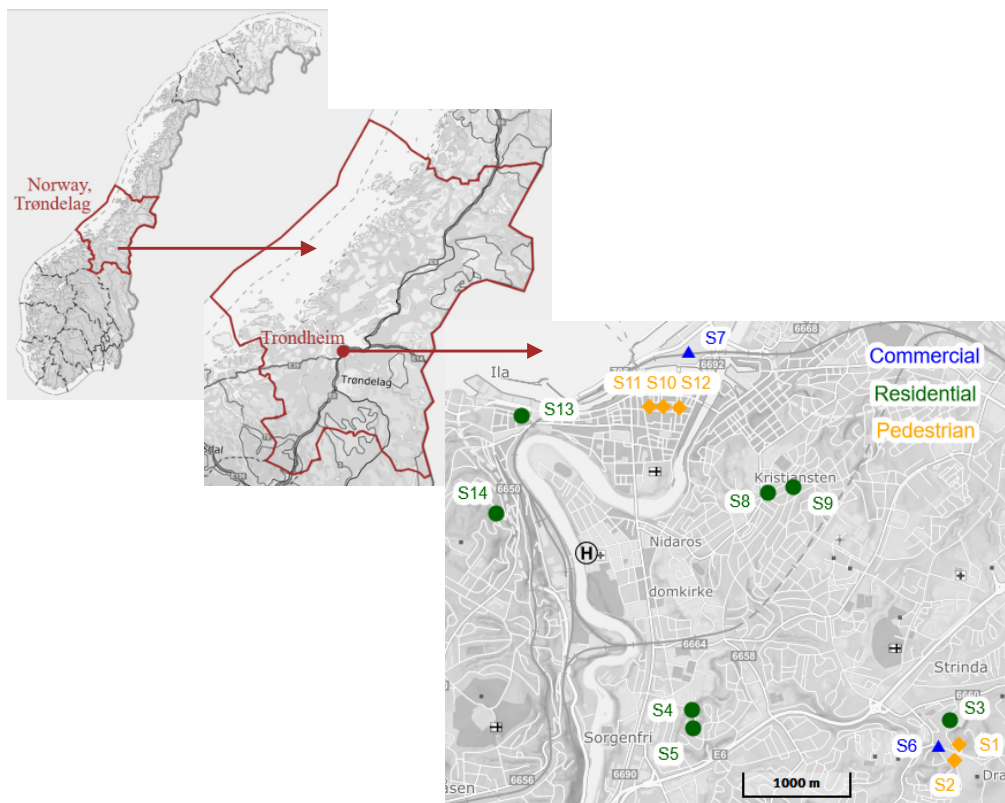


**Figure 1.** Flow chart describing the methodical approach.

## 2.1 Field sites

Trondheim, located at the mouth of the river Nidelva, is a city and a municipality in the province of Trøndelag in the middle of Norway. The mean annual precipitation is 884 mm (Merkel, 2019). Its moderate and humid climate with an annual maximum and minimum temperature in 2018/2019, respectively 14.9 °C and -2.1 °C (Meterologisk institutt, 2019), is attributed to the Gulf stream.

Samples of sediment beds in RSGP were collected in commercial, residential and pedestrian areas (see Figure 2).



**Figure 2.** Map of Trondheim, Trøndelag in Norway with marked sample locations in commercial (blue triangle), residential (green point) and pedestrian areas (orange rhombus). (GeoNorge; Norgeskart Kartverket)

According to Post et al. (2016), spatial characteristics for each sample with large impact on trap efficiency were noted for later precise analyzing approaches (see Table 1).

**Table 1.** List of samples with spatial characteristics: Landuse, street name, ADT [veh/day], HGVT [%], road surface, road slope, speed limit [km/h], impervious catchment area [m<sup>2</sup>] and sediment depth [m].

|  | Landuse     | Streetname           | ADT [veh/day]<br>HGVT [%]<br>*** | Road<br>Surface<br>Slope<br>Speed limit [km/h] | Imper-<br>vious<br>catch-<br>ment<br>area<br>[m <sup>2</sup> ] * | Sedi-<br>ment<br>depth<br>[m] |
|--|-------------|----------------------|----------------------------------|--|--|-------------------------------|
| S1   | Pedestrian  | Loholt alle          |                                  | Gravel; steep; -                               | 75   | 0.52                          |
| S2   | Pedestrian  | Loholt alle          |                                  | Gravel; steep; -                               | 110  | 0.08                          |
| S3   | Residential | Brit Grytbaks veg    | 100; 1                           | Asphalt; steep; 30                             | 740  | 0.36                          |
| S4   | Residential | Bakkehellet          | 700; 1                           | Asphalt; steep; 30                             | 470  | 0.94                          |
| S5   | Residential | Bakkehellet          | 700; 1                           | Asphalt; steep; 30                             | 585  | 0.56                          |
| S6   | Commercial  | Loholt alle          | 2500; 10                         | Asphalt; steep; 50                             | 470  | 0.38                          |
| S7   | Commercial  | Trondheim S          | 18250; 100**                     | Pavement; flat; 40                             | 330  | 0.83                          |
| S8   | Residential | Tyholtveien          | 2700; 2                          | Asphalt; flat; 30                              | 295  | *****                         |
| S9   | Residential | Strindvegen          | 600; 2                           | Asphalt; steep; 30                             | 340  | *****                         |
| S10  | Pedestrian  | Nordre Gate          | ****                             | Pavement; flat; -                              | 260  | *****                         |
| S11  | Pedestrian  | Thomas Angells Gate  | ****                             | Pavement; flat; -                              | 210  | *****                         |
| S12  | Pedestrian  | Thomas Angells Gate  | ****                             | Pavement; flat; -                              | 250  | *****                         |
| S13  | Residential | Mellomila            | 455; 7                           | Asphalt; flat; 30                              | 450  | *****                         |
| S14  | Residential | Fridtjof Nansens vei | 1000; 2                          | Asphalt; steep; 30                             | 630  | *****                         |
| * estimated  |             |                      |                                  |  |  |                               |
| ** (Information from AtB AS Trondheim, 2019)                         |             |                      |                                  |  |  |                               |
| *** (Statens vegvesen - vegkart)                                     |             |                      |                                  |  |  |                               |
| **** existing delivery traffic                                       |             |                      |                                  |  |  |                               |
| ***** not available (no emptying process using a truck was possible) |             |                      |                                  |  |  |                               |

## 2.2 Description of a RSGP

The city of Trondheim owns approximately 15000 RSGP receiving road runoff (Information from Bydrift in Trondheim Kommune, 2019). Figure 3 shows a typical RSGP used in Norway. The position of the outlet is located approximately 1 m above the bottom, providing a capacity for capturing particles via sedimentation flushed from roads during precipitation.



The inner diameter and the distance bottom – outlet of a RSGP in Trondheim, both are usually 1 m. However, the dimensions vary depending on appropriate requirements and conditions due to ground level. In Trondheim frequency of maintenance is mainly based on weather conditions and typically around once every three years. (Information from Bydrift in Trondheim Kommune, 2019)

A RSGP has a connected impervious catchment area in average of approximately 350 m<sup>2</sup> based on observations of the locations and calculations.

**Figure 3.** RSGP typical for Norway, Trondheim (BASAL, 2019).

## 2.3 Sample collection

Sample collection was carried out between October and December 2019. A grab composite sample of 1 liter was collected using a stainless cup welded on a stick. Sediment samples were taken during maintenance applying a suction vehicle by Trondheim Bydrift. In order to get a representative sample throughout the sediment depth, samples were taken from the top, middle and from the bottom layer of the sediment bed.

All samples were taken from the same horizontal placement within a gully pot, in this case from the center with respect to later comparability. Before sample extraction, water on top of the sediment bed was withdrawn by suction operated by an employee of Trondheim Bydrift. Between gully pots, the cup was washed in order not to contaminate the following sample. A clean, plastic bottle was used to store samples until taken directly to the laboratory where samples were prepared and analyzed subsequently. This sampling method was used for samples 1 to 7.

Due to limited possibilities of getting regularly samples supported by the vacuum vehicle, a manually operated grab sampler was used for the samples 8 to 14. This method was not ideal as the primarily applied method because of gaining solely samples from the sediment bed surface and not throughout the sediment depth.

## 2.4 Laboratory analyses

The table below shows the number of examined samples for each conducted laboratory test.

**Table 2.** Quantity of examined samples for each conducted laboratory test and corresponding sample numbers.

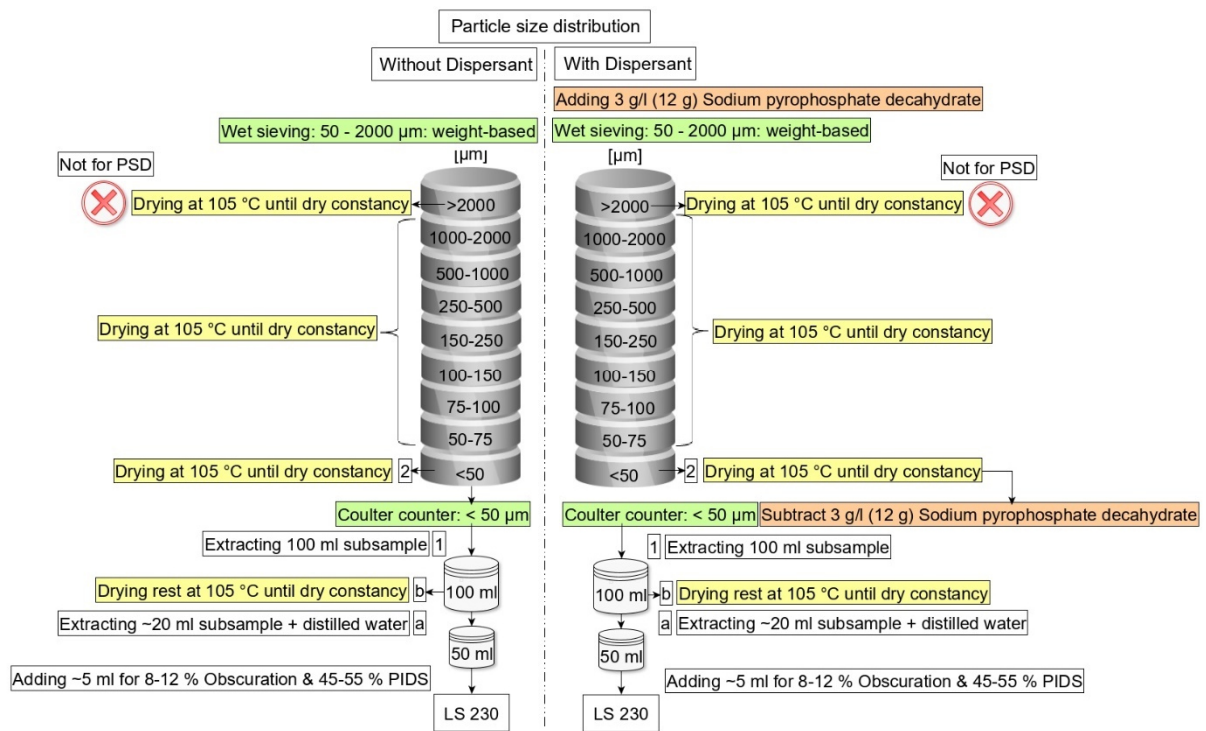
| Analysis type              | Quantity of samples   | Sample numbers |
|----------------------------|-----------------------|----------------|
| Particle size distribution | n = 14                | 1-14           |
| Ignition loss              | n = 14 (whole sample) | 1-14           |
|                            | n = 9 (fractions)     | 6-14           |
| Specific gravity           | n = 8 (fractions)     | 7-14           |
| Heavy metals               | n = 14 (whole sample) | 1-14           |
|                            | n = 8 (fractions)     | 7-14           |

### 2.4.1 Particle size distribution

The methodical approach of using the two methods for determining PSD, manual weight-based wet sieving for particles between 50 and 2000  $\mu\text{m}$  and additionally a Beckman Coulter counter instrument LS230 for small particles  $<50 \mu\text{m}$ , was selected based on prior conducted tests (see Appendix B.1 and Appendix B.2).

To indicate both, the natural appearance of particles in RSGP including agglomerations (NAT) and segregated particles breaking the natural agglomerations (DIS), a dispersant was used according to Slattery and Burt (1997). An inorganic sodium salt, Sodium Pyrophosphate Decahydrate:  $\text{Na}_4\text{P}_2\text{O}_7 \cdot 10\text{H}_2\text{O}$  (SPD) in crystal form was used as a gentle dispersant to minimize the risk of changing or destroying the particles more than merely separating from each other (Konert and Vandenberghe, 1997), (Roberts et al., 1988), (Appendix B.3.4).

The dosage of 3 g/l SPD was preliminary adapted from Konert and Vandenberghe (1997) and DIN EN ISO 17892-4 (2017), but was verified by measuring the zeta potential in this study (see Appendix B.5.2). Therefore, two equal subsamples were produced using a riffle splitter (Appendix B.4). Figure 4 shows the method flowchart for PSD analysis.



**Figure 4.** Flow chart describing the approach of particle size distribution; wet sieving for particles between 50 and 2000 µm and using a Coulter counter instrument for particles <50 µm; with and without a dispersant.

In order to prevent either overloaded or underloaded sieves, an initial weight in later dry condition of 100 g was intended (DIN EN ISO 17892-4, 2017), (Statens vegvesen Norway, 1997). An initial weight in wet condition between approximately 200 g and 300 g, depending on the coarseness, was found to provide this requirement.

Stainless steel sieves with a diameter of 200 mm were used. Sizes were chosen adapting on NJCAT Technology Verification (2015) for further purposes to: 50, 75, 100, 150, 250, 500, 1000 and 2000 µm. Manual wet sieving was carried out in 3 steps: 1) Pouring the sample diluted with 4 liter distilled water over the sieve stack in tray number 1; 2) Flushing each sieve step by step with 0.5 liter distilled water and pouring the through fraction over the remaining sieves on tray number 2; 3) Repeating step 2 over tray number 3. Particles were transferred carefully via distilled water from the sieves into beaker.

Drying samples was adapted to the European Standard DIN EN 12880 (2001). To ensure a dry sample, the dry mass after another hour at 105 °C should not differ >0,5 % from the dry mass before. Particles >2000 µm were neglected for determining the PSD in order not to underestimate the smaller fractions.

For analyzing particles <50 µm in the Coulter counter LS230, a representative small subsample was grabbed out of the first tray by a quick entire movement of a 100 ml clean plastic beaker directly after stirring and homogenizing the tray completely by hand. Approximately



20 ml were transferred via pipette into a 50 ml tube and was diluted with distilled water. Approximately 5 ml was added via pipette into the Coulter counter in order to get a suitable PIDS (Polarization Intensity Differential Scattering) between 45 % and 55 % and an obscuration between 8 % and 12 %. The extracted amounts are dependent on the concentration of the lowest fraction of each sample. Before each pipette abstraction the tube was inverted gently in order to prevent bubbles. For each sample three runs with a duration of 90 seconds each have been conducted subsequently. For a stable sample the variation of the three resulting PSD curves should be less than 1 %. This is influenced by pumping speed, which was adjusted experimentally to approximately 50 %. The device was flushed three times between samples to reduce particles in the background liquid using distilled water with a reflection index of 1.3330. Sample leftovers were transferred via distilled water into a beaker, dried at 105 °C and added calculational to the weight of particles <50 µm.

This procedure was repeated with the second subsample after adding 3 g/l SPD and soaking over night (Appendix B.5.1). For determining the PSD, 12 g was calculational subtracted from the mass of the particles <50 µm due to the added dispersant which will end up after flushing the sieves in the lowest fraction in tray number 1. As a background liquid in the Coulter counter the same concentration of 3 g/l SPD was used, measured a refractive index of 1.3335 by using a Mettler Toledo Refracto 30PX/GS.

The use of a light scattering particle size analyzer, a Coulter counter LS230 using the optical model Fraunhofer for analyzing the small fraction of particles was selected for its precise measurement in a total size range from 0.04 to 2000 µm. PIDS enables the LS230 to measure small particles from 0.04 µm to 0.4 µm. It measures distributions in 116 size channels both, differential and cumulative number-, volume-, or surface-based. (Beckman Coulter, 2011b).

#### **2.4.2 Ignition loss**

The determination of the organic matter in the samples provides a basis for an appropriate evaluation of the particle size distributions in relation to decomposition process caused by biological activity as a potential reason for small particles. It also points out the availability of oil.

Therefore, ignition loss of sediment bed samples was determined both, for the whole sample and for fractions after sieving. For the ignition loss on the whole sample three subsamples were taken using a spoon after sediments were rinsed out of the bottle and homogenized by hand. These subsamples were taken to get close to a sample quantity of 15 g or 30 g for fine soils and sand, respectively (DIN 18128, 2002).

Drying samples was adapted to the European Standard DIN EN 12880 (2001). Hence, to ensure a dry sample, the dry mass after another hour at 105 °C should not differ >0.5 % from the

dry mass before. Before glowing at 550 °C for 2 h, the drying layer caused by drying process was broken using a mortar (DIN 18128, 2002).

The standard for ignition loss was selected due to similarity of sediment and soil samples. Fireclay crucibles were heated at 550 °C for 1 h and weighed after cooling for 30 min in a desiccator prior to usage.

The average of the three subsamples was calculated to produce a representative value throughout the tray.

### **2.4.3 Specific gravity of fractions**

The specific gravity of the fractions <50, 50-75, 75-100, 100-150, 150-250, 250-500, 500-1000 and 1000-2000 µm was determined using a pycnometer. Fractions after wet sieving without a dispersant and XRF-analysis were used.

The approach was adapted to the International Standard ISO 17892-3 (2015) with some modifications caused by previous methodical steps related to the PSD analysis. Due to hydrophobic properties of once dried sediments which was experienced in earlier conducted methodical tests (see Appendix B.3), the fractions were resuspended into distilled water the day before analysis. Before this, the applied dry weight was noted (a). After soaking over night, the suspended particles were completely transferred via distilled water into the pycnometer and were de-aired for 1 h using a vacuum pump. Then the pycnometer including the particles was filled up with previously de-aired distilled water and was weighed together with the plug (b). The same pycnometer was weighed with de-aired and distilled water, but without particles (c). For determining the specific gravity of the fractions  $\rho_s$ , following formula with a water density  $\rho_w$  of 0.998 g/cm<sup>3</sup> was used:

$$\rho_s = \frac{a}{(c + a - b) * \rho_w} \text{ [g/cm}^3\text{]}^* \quad (1)$$

\* (ISO 17892-3, 2015)

### **2.4.4 Heavy metals**

Heavy metals were analyzed for the size ranges <50, 50-75, 75-100, 100-150, 150-250, 250-500, 500-1000 and 1000-2000 µm. A non-destructive handheld XRF analyzer was used to investigate heavy metals in the sediment samples focusing on chrome (Cr), copper (Cu), iron (Fe), lead (Pb) and zinc (Zn). The XRF analyzer was applied on fractions after wet sieving procedure and drying to dry constancy according to DIN EN 12880 (2001). XRF method measures both, dissolved and bonded metals.

Measurements have been done on prepared approximately 2-4 mm flat fractions since the effective measurement area of the XRF instrument is around 2 mm. Measured values within

one fraction varied to some extent related to the angle the radiation struck atoms in a sample. To minimize this effect and to ensure a representative value throughout each fraction, 7 measurement runs each in a length of minimum 60 seconds have been applied on different spots. For the same reason, fractions  $>2000 \mu\text{m}$  have not been analyzed due to an increase of this effect with increasing particles sizes. The measurement window was cleaned between each measurement run.

## **2.5 Data analyses**

For statistical evaluation of investigated parameters, samples were divided into two groups with different landuse: no traffic (Sample 1,2,10,11,12) and traffic (Sample 3,4,5,6,7,8,9,13,14).

Box-and-whisker plots were applied to evaluate the parameters within the two groups. In order to assess the correlation between investigated parameters among each other and with catchment characteristics, the rank-based Spearman correlations have been calculated. This statistical correlation method was chosen due to the non-normal distribution of most of the data. The Shapiro-Wilk normality test was applied for determining the normality of the datasets. Only statistically significant correlations with a p-value  $< 0.05$  were included for evaluation. For statistical analysis the program RStudio was used.

## **2.6 Methodical limitations**

Due to the necessity of keeping the lowest fraction  $<50 \mu\text{m}$  for further PSD in the Coulter counter when wet sieving, the amount of water used for flushing sieves in order to get a most appropriate distribution, is limited. If the smallest fraction would not have been kept, flushing procedure could have been repeated as much as needed. Hence, some smaller particles could remain on not appropriate sieves sizes due to the limited flushing possibility. However, it is assumed that this amount is not affecting the result of PSD significantly due to an almost clear water in the third tray after the last flush round during sieving procedure.

Another weakness of using wet sieving is the difficulty estimating a suitable initial start weight for sieving procedures due to the water content. It should be enough sediments in order to receive fair amounts of fractions after the drying process. At the same time the sediment mass has to be limited in order not to clog the sieves. Furthermore, there is no control of weight loss during sieving process due to the lack of initial dry weight.

It is recognized that there is a minor deficit in the weight for the particle size distribution in the smallest fraction due to the extraction of particles for further analysis in the Coulter counter LS230.

The fact, that getting samples from the field was dependent on working plans of Trondheim Bydrift implicated three shortcomings: 1) Less than ideal samples in number could be analyzed, 2) it was impossible to get samples from roads with a high ADT and 3) Sample locations could not be selected, but were determined by the management of Trondheim Bydrift. Furthermore, the unavoidable change of sampling strategy may cause inaccuracy in results.

For determining the specific gravity of the wet sieved and dried fractions by using a pycnometer there was the need of deviating from the Standard when applying less than the recommended minimum 10 g sample for some fractions. This is based on the available varying amounts of fractions resulting from limited initial amounts for previous sieving procedures in order not clogging the sieves. Furthermore, it was impossible to determine the specific gravity for the fraction >2000  $\mu\text{m}$  due to opening width of the pycnometer. In this investigation one is aware of potential trapped air in the pycnometer samples which may be caused by agglomerations related to prior experienced hydrophobic properties of the sediments. This potential effect is a limited factor and will be minimized by resuspending the dry particles one day before analysis.

For a meaningful and proper statistical analysis this investigation has less than ideal amounts of samples. Nevertheless, statistical tests were used to get an overview of the raw data obtained by laboratory tests. However, the statistically obtained results must be viewed with caution and may not be appropriate for clear conclusions. Therefore, more samples are necessary.

### **3 Results**

The results are divided into parts corresponding to the methodical approach, consisting of a) particle size distribution in relation to organic loss and specific gravity and b) heavy metal content found in sediment beds.

#### **3.1 Size characterization of particles in sediment beds**

Results of particle size distribution, ignition loss and specific gravity of fractions are demonstrated in the following paragraphs.

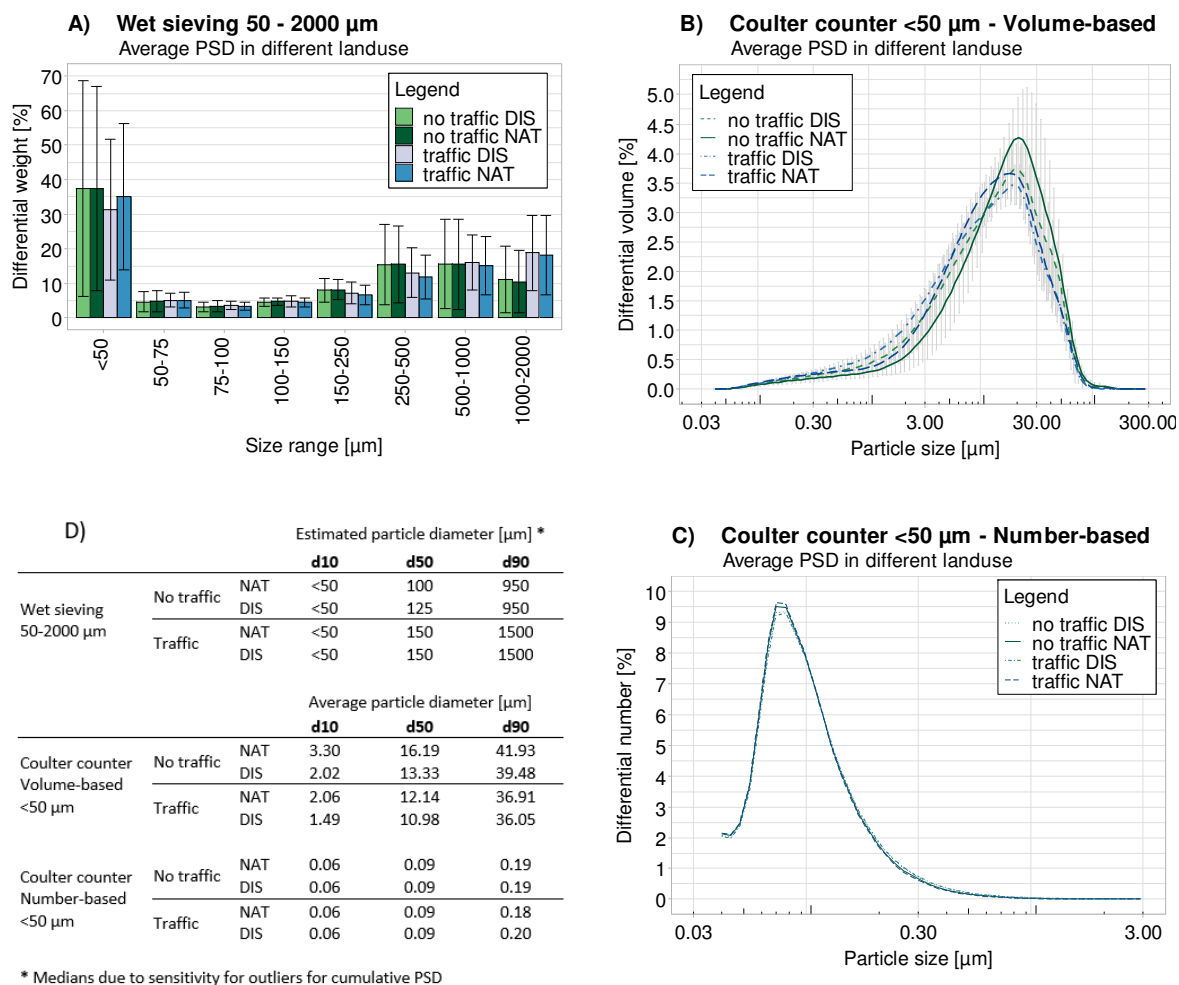
##### **3.1.1 Particle size distribution**

Particle size distribution of sediment bed samples was investigated applying wet sieving for particles between 50 and 2000  $\mu\text{m}$  since it is an important factor influencing sedimentation rate.

As can be seen from Figure 5, A), there was hardly any difference between not dispersing and dispersing with SPD prior to sieving procedure throughout all size ranges for both groups, with and without traffic activities. Furthermore, the particle size distributions in areas with and

without traffic activities differed not much from each other except for the size range 1000 to 2000  $\mu\text{m}$ . There was a difference of around one half between both groups. The mean values of fractions in the size ranges <50, 50-75, 75-100, 100-150, 150-250, 250-500, 500-1000 and 1000-2000  $\mu\text{m}$  were between 31.27 and 37.41 %, 4.65 and 5.12 %, 3.23 and 3.60 %, 4.51 and 4.79 %, 6.67 and 8.13 %, 11.93 and 15.49 %, 15.16 and 16.11 %, 10.55 and 18.77 %, respectively.

By using a Coulter counter instrument particles <50  $\mu\text{m}$  have been analyzed for providing a more detailed view for smaller particles, both volume-based (see Figure 5, B)) and number-based (see Figure 5, C)). Volume-based particle size distributions illustrated with standard deviations showed left-skewed unimodal distributions with peaks centered around 20  $\mu\text{m}$  with differential percentages between 3.38 % (traffic area, dispersed) and 4.25 % (no-traffic area, not dispersed). Within and for both groups there was a horizontal shift towards small particles for the dispersed subsamples which caused a decreasing percentage in the range between 10 and 50  $\mu\text{m}$  and 5 and 50  $\mu\text{m}$  and an increasing percentage in the range between 0.3 and 10  $\mu\text{m}$  and 0.3 and 5  $\mu\text{m}$ , respectively for the no-traffic and traffic group. The vertical difference between dispersed and undispersed subsamples varied with particle size but was highest with 0.50 %. Number-based distributions represent right-skewed unimodal curve progressions with peaks at around 0.08  $\mu\text{m}$  and differential percentage between 9.25 % for dispersed samples and 9.50 % for undispersed samples, very similar for both groups. Figure 5, D) shows the particle size characteristics  $d_{10}$ ,  $d_{50}$  and  $d_{90}$  for cumulative distribution.



**Figure 5.** Particle size distribution results for no-traffic and traffic group with (DIS) and without (NAT) dispersant. **A)** Wet sieving for particles between 50 and 2000  $\mu\text{m}$  showing averaged differential weights [%] for sieving size ranges  $\pm$ S.D. displayed as errorbars; **B)** Coulter counter for particles <50  $\mu\text{m}$  volume-based results showing averaged differential PSD [%]  $\pm$ S.D. displayed as errorbars; **C)** Coulter counter for particles <50  $\mu\text{m}$  number-based results showing averaged differential PSD [%]; **D)** Listed particle diameter d10, d50 and d90 [ $\mu\text{m}$ ].

### 3.1.2 Organic loss in particle size fractions

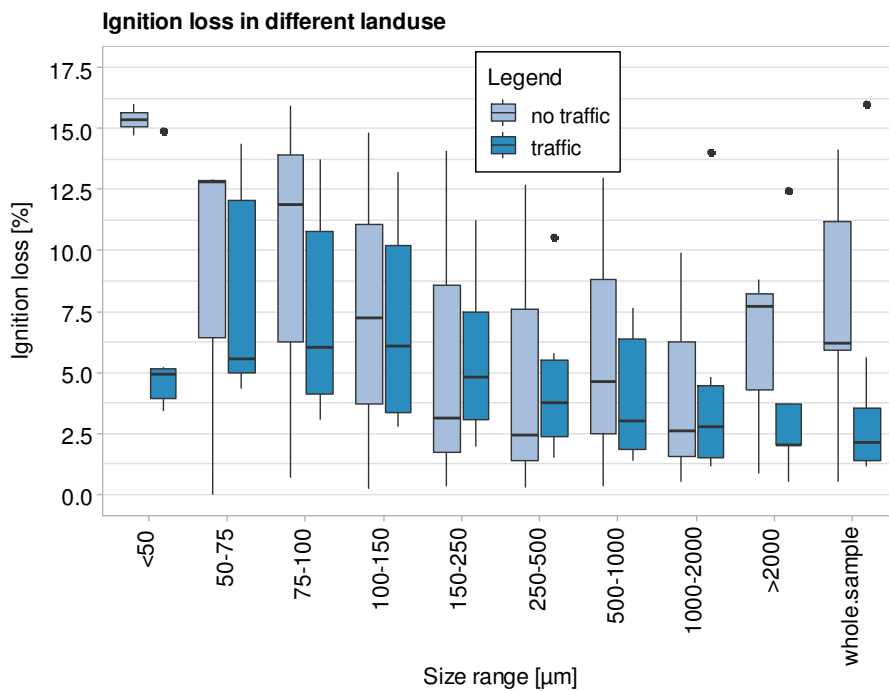
Organic loss was examined in the size ranges <50, 50-75, 75-100, 100-150, 150-250, 250-500, 500-1000, 1000-2000, >2000  $\mu\text{m}$  and for the whole sample for areas with and without traffic activities by glowing samples at 550  $^{\circ}\text{C}$  (see Figure 6).

Median values for the no-traffic group were higher than the medians for the traffic group for the size ranges <50 (15.36 and 4.95 %), 50-75 (12.84 and 5.57 %), 75-100 (11.86 and 6.04 %), 100-150 (7.23 and 6.06 %), 500-1000 (4.62 and 3.03 %), >2000  $\mu\text{m}$  (7.68 and 2.02 %) and for the whole sample (6.22 and 2.16 %). The median values for the no-traffic areas were lower than in traffic areas in the size ranges 150-250  $\mu\text{m}$  (3.12 and 4.84 %) and 250-500  $\mu\text{m}$  (2.44 and 3.74 %). In the size range 1000-2000  $\mu\text{m}$  the medians were similar: 2.61 and 2.76 %, respectively for the no-traffic and traffic area. In all size ranges except in the lowest fraction

<50  $\mu\text{m}$  the interquartile ranges were widely spread, but with a decrease in span width with increasing particle size for the traffic group.

The organic matter distribution among size ranges varied within the groups. Regarding the group without traffic, the highest median with 15.36 % was found to be in the smallest size fraction <50  $\mu\text{m}$ . The organic matter content decreased to a median of 2.44 % in the size range 250-500  $\mu\text{m}$  and increased fluctuating up to a median of 7.68 % in the largest size range >2000  $\mu\text{m}$ . For traffic areas the observed organic matter increased from a median of 4.95 % in the smallest fraction <50  $\mu\text{m}$  to a median of 6.06 % in the fraction 100-150  $\mu\text{m}$ . From this range there was an even decrease in organic matter until a median of 2.02 % in the largest particle size range >2000  $\mu\text{m}$ . Concerning the observed organic matter fraction in the whole sample, it can be seen a higher percentage with a widely spread interquartile range in the no-traffic group.

There were outliers detected in the size ranges <50, 250-500, 1000-2000, >2000  $\mu\text{m}$  and in the whole sample for the traffic group reaching values around 15 %.



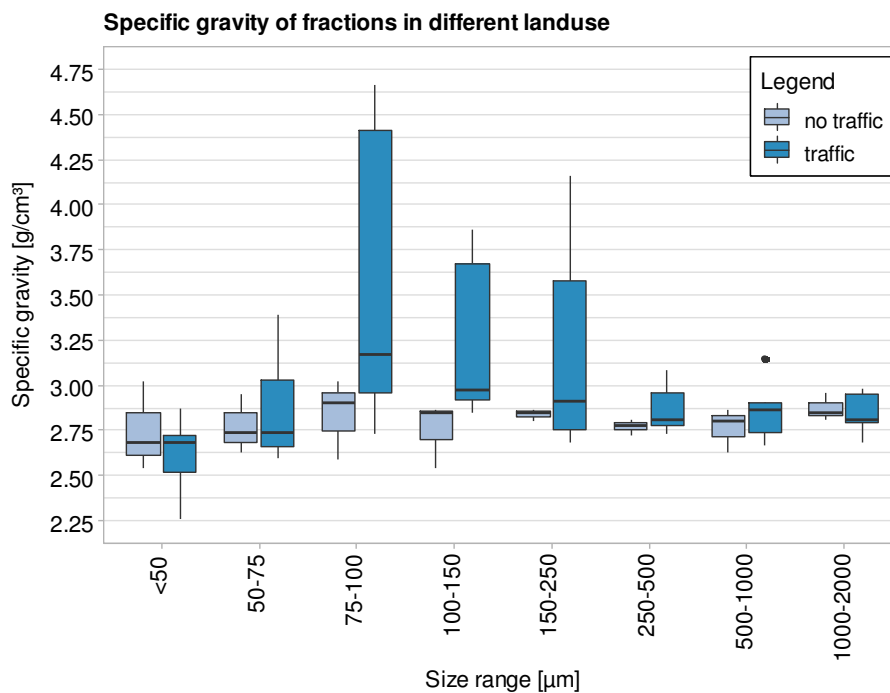
**Figure 6.** Ignition loss results [%] in sieved size ranges including >2000  $\mu\text{m}$  and whole sample displayed as a box-and-whisker plot for the no-traffic and traffic group.

### 3.1.3 Specific gravity of particle size fractions

Specific gravity of sediments in the sieved size ranges <50, 50-75, 75-100, 100-150, 150-250, 250-500, 500-1000 and 1000-2000  $\mu\text{m}$  were measured using a pycnometer (see Figure 7).

Comparing the two area types, it can be seen that the medians were exactly the same, namely 2.68  $\text{g}/\text{cm}^3$  for particles <50  $\mu\text{m}$  and 2.74  $\text{g}/\text{cm}^3$  for the size fraction 50-75  $\mu\text{m}$ . Specific gravity medians in the traffic group were higher than the values of the no-traffic group in the size ranges 75-100 (3.17 and 2.90  $\text{g}/\text{cm}^3$ ), 100-150 (2.97 and 2.85  $\text{g}/\text{cm}^3$ ), 150-250 (2.91 and 2.85  $\text{g}/\text{cm}^3$ ), 250-500 (2.81 and 2.78  $\text{g}/\text{cm}^3$ ) and 500-1000  $\mu\text{m}$  (2.86  $\text{g}/\text{cm}^3$  and 2.80  $\text{g}/\text{cm}^3$ ). For the largest fraction 1000-2000  $\mu\text{m}$  specific gravity median of the traffic group was lower than the median of the no-traffic group (2.81 and 2.85  $\text{g}/\text{cm}^3$ ). The right-skewed interquartile ranges of the traffic group in the size ranges 75-100, 100-150 and 150-250  $\mu\text{m}$  are widely spread with whiskers up to 4.66, 3.86 and 4.16  $\text{g}/\text{cm}^3$ , respectively.

In general, the lowest specific gravity was found to be in the smallest size range <50  $\mu\text{m}$  with a minimum of 2.26  $\text{g}/\text{cm}^3$  and medians at 2.68  $\text{g}/\text{cm}^3$ . The highest values with medians between 2.85 and 3.17  $\text{g}/\text{cm}^3$  were detected in the size ranges 75-100, 100-150 and 150-250  $\mu\text{m}$ .

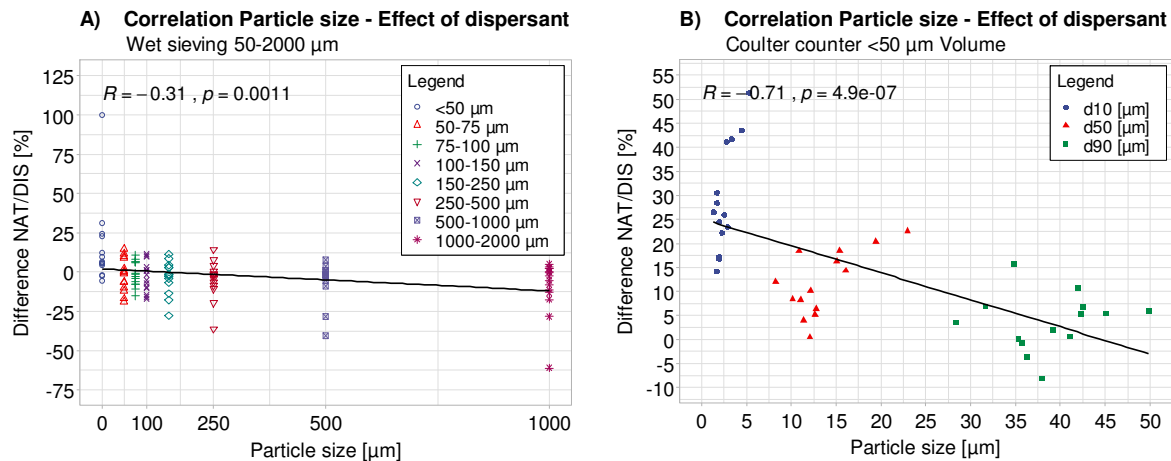


**Figure 7.** Specific gravity of fractions results [ $\text{g}/\text{cm}^3$ ] in sieved size ranges displayed as a box-and-whisker plot for the no-traffic and traffic group.



### 3.1.4 Correlation between particle size and effect of a dispersant

The correlation between the particle size and the difference in particle size distribution using a dispersant or not was analyzed. Figure 8, A) displays the difference for wet sieving procedure for particles between 50 and 2000  $\mu\text{m}$ . The percentage difference was calculated using the weight-based differential fractions from each size range (see Legend). As can be seen, there was a statistically significant negative weak correlation between those two variables with a correlation coefficient of  $R = -0.31$  and  $p = 0.0011$ .

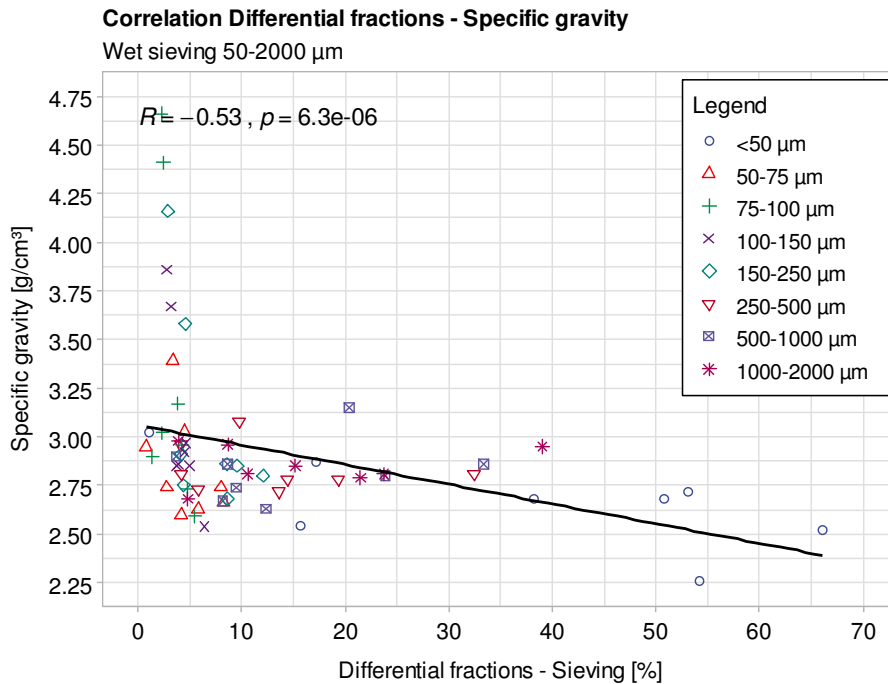


**Figure 8.** Spearman rank-order correlation between particle size [ $\mu\text{m}$ ] and the effect of the dispersant calculated as a percentage difference between: **A)** the differential weight-based sieving fractions with and without a dispersant from wet sieving for particles between 50 and 2000  $\mu\text{m}$ ; **B)** percentage difference between d10, d50 and d90 with and without a dispersant for Coulter counter for particles <50  $\mu\text{m}$  Volume-based results; (<50  $\mu\text{m}$  is equal to 0 etc.).

The correlation between the same variables, but for particles <50  $\mu\text{m}$  analyzed by using a Coulter counter instrument is displayed in plot B). The percentage difference was calculated for the particle size characteristics d<sub>10</sub>, d<sub>50</sub> and d<sub>90</sub>. There was a negative strong correlation with a statistical significance between the particle size and the percentage difference between using a dispersant or not. The correlation coefficient was  $R = -0.71$  with a p-value of  $4.9 \times 10^{-7}$ .

### 3.1.5 Correlation between differential particle size distribution and specific gravity

The correlation between the differential fractions from wet sieving procedure in the different size ranges and the specific gravity of each fraction is illustrated in Figure 9. It was found a statistically significant negative moderate correlation with a correlation coefficient of  $R = -0.53$  and a p-value of  $p = 6.3 \times 10^{-6}$ .



**Figure 9.** Spearman rank-order correlation between differential fractions [%] from wet sieving for particles between 50 and 2000 µm and specific gravity of fractions [g/cm³].

## 3.2 Characterization of heavy metals in sediment beds

Heavy metal concentrations found in investigated sediment beds are given in the following subchapters.

### 3.2.1 Content of heavy metals

Heavy metals focusing on Cr, Cu, Fe, Pb and Zn in RSGP sediment beds were analyzed using an XRF instrument in the size ranges <50, 50-75, 75-100, 100-150, 150-250, 250-500, 500-1000 and 1000-2000 µm (see Figure 10).

The median values for chrome (Figure 10, A) for no-traffic and traffic areas were found to be similar in the size ranges 50-75 (81.64 and 82.04 mg/kg), 75-100 (96.29 and 91.26 mg/kg), 100-150 (56.79 and 64.80 mg/kg) and 150-250 µm (35.21 and 36.46 mg/kg). For the smallest particle size fraction <50 µm the median value for samples in areas without traffic was higher (220.29 mg/kg) than the median for samples in areas with traffic (154.93 mg/kg). For the size ranges 250-500, 500-1000 and 1000-2000 µm median values for no-traffic areas were lower than for areas with traffic: 0.00 and 37.01 mg/kg, 31.78 and 55.14 mg/kg, 26.72 and

71.38 mg/kg. The lower quartile for both, traffic and no-traffic group were tending to be at zero for larger size ranges from 100 to 2000  $\mu\text{m}$  with one median in the size range 250-500  $\mu\text{m}$  located at zero. The highest values for Cr were in the smallest size range <50  $\mu\text{m}$  with a maximum of 381.28 mg/kg and a median of 220.29 mg/kg. For both groups, values decreased with an increasing particle size to around 30 mg/kg. Values increased again in the two larger size ranges 500-1000 and 1000-2000  $\mu\text{m}$  to 71.38 mg/kg for the traffic group.

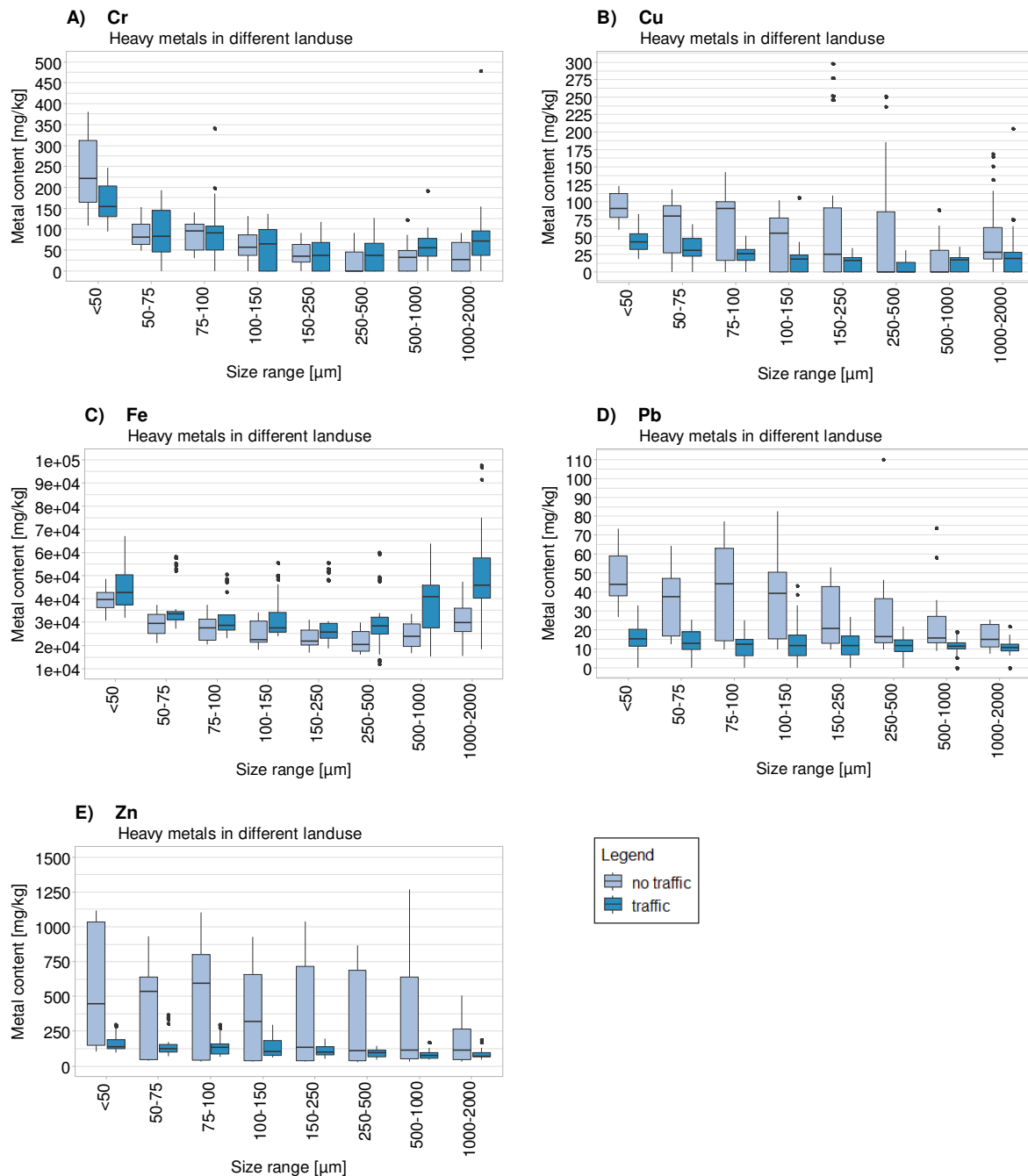
For copper (Figure 10, B)) median values were found to be higher in the no-traffic group than in the traffic group for the size ranges <50 (90.91 and 42.62 mg/kg), 50-75 (79.43 and 31.17 mg/kg), 75-100 (90.22 and 26.35 mg/kg), 100-150 (55.82 and 18.57 mg/kg), 150-250 (24.86 and 16.53 mg/kg) and 1000-2000  $\mu\text{m}$  (28.37 and 19.31 mg/kg). In the size range 250-500  $\mu\text{m}$  both medians were 0.00 mg/kg. In the range 500-1000  $\mu\text{m}$  the median for the traffic area was 16.78 mg/kg whereas the median for no-traffic areas was 0.00 mg/kg. In general, interquartile ranges for no-traffic areas were more widely scattered than for the traffic group. There were highest values in the small size ranges and dropping towards zero with an increase in particle size but with increased values in the largest fraction 1000-2000  $\mu\text{m}$ . In all size ranges, except in the smallest range there was either a minimum or a lower quartile at 0.00 mg/kg. Outliers were detected in particle sizes from 150  $\mu\text{m}$  located between 125 and 300 mg/kg.

For iron (Figure 10, C)) the median values in areas without traffic were lower throughout all size ranges with the highest difference in the two largest size ranges. The interquartile ranges for the group with traffic in the size ranges with the highest medians, namely <50, 500-1000 and 1000-2000  $\mu\text{m}$  are more widely spread. Moreover, there were outliers detected in size ranges with smaller values in the traffic group reaching values up to 60000 mg/kg, but still in the range of values of the size ranges with the highest values. In the size range <50  $\mu\text{m}$  medians were detected of 39943.90 mg/kg for the no-traffic group and 42861.12 mg/kg for the traffic group. Both groups followed the same manner in decreasing iron concentration with increasing particle size up to 150-250  $\mu\text{m}$  and 250-500  $\mu\text{m}$ , respectively for traffic and no-traffic areas with a minimum displayed as outlier of 11839.47 mg/kg. From 500  $\mu\text{m}$  there was a rise in iron content up to the largest fraction with the highest whisker at 75000 mg/kg and an outlier at around 97000 mg/kg.

For lead (Figure 10, D)) median values for no-traffic areas occurred constantly above the medians for traffic areas throughout all size ranges. There was found a large difference between the no-traffic and the traffic areas in the size ranges <50 (43.95 and 15.15 mg/kg), 50-75 (37.44 and 12.88 mg/kg), 75-100 (44.32 and 12.30 mg/kg) and 100-150  $\mu\text{m}$  (39.23 and 11.77 mg/kg) and a small difference in the size ranges 150-250 (20.69 and 11.63 mg/kg), 250-500 (16.19

and 11.59 mg/kg), 500-1000 (15.51 and 11.36 mg/kg) and 1000-2000  $\mu\text{m}$  (14.85 and 10.52 mg/kg). The minimum values of the traffic group in each size range remained at zero, whereas minimum values of the no-traffic group never reached zero, but were around 10 mg/kg. The interquartile ranges for the no traffic group were widely scattered, especially in the smaller size ranges with a maximum score of 82.83 mg/kg. Outliers in this group were found in the size ranges 250-500 and 500-1000  $\mu\text{m}$  with values around 60, 75 and 110 mg/kg. In contrast the normal-distributed interquartile ranges in the traffic group were closely together. The lower quartile of the no-traffic group stayed above the median values of the traffic group throughout all size ranges. In general, the content of Pb was higher in the small particle sizes with a steep drop towards the larger size fractions for the no-traffic group and a smooth decrease for the traffic group.

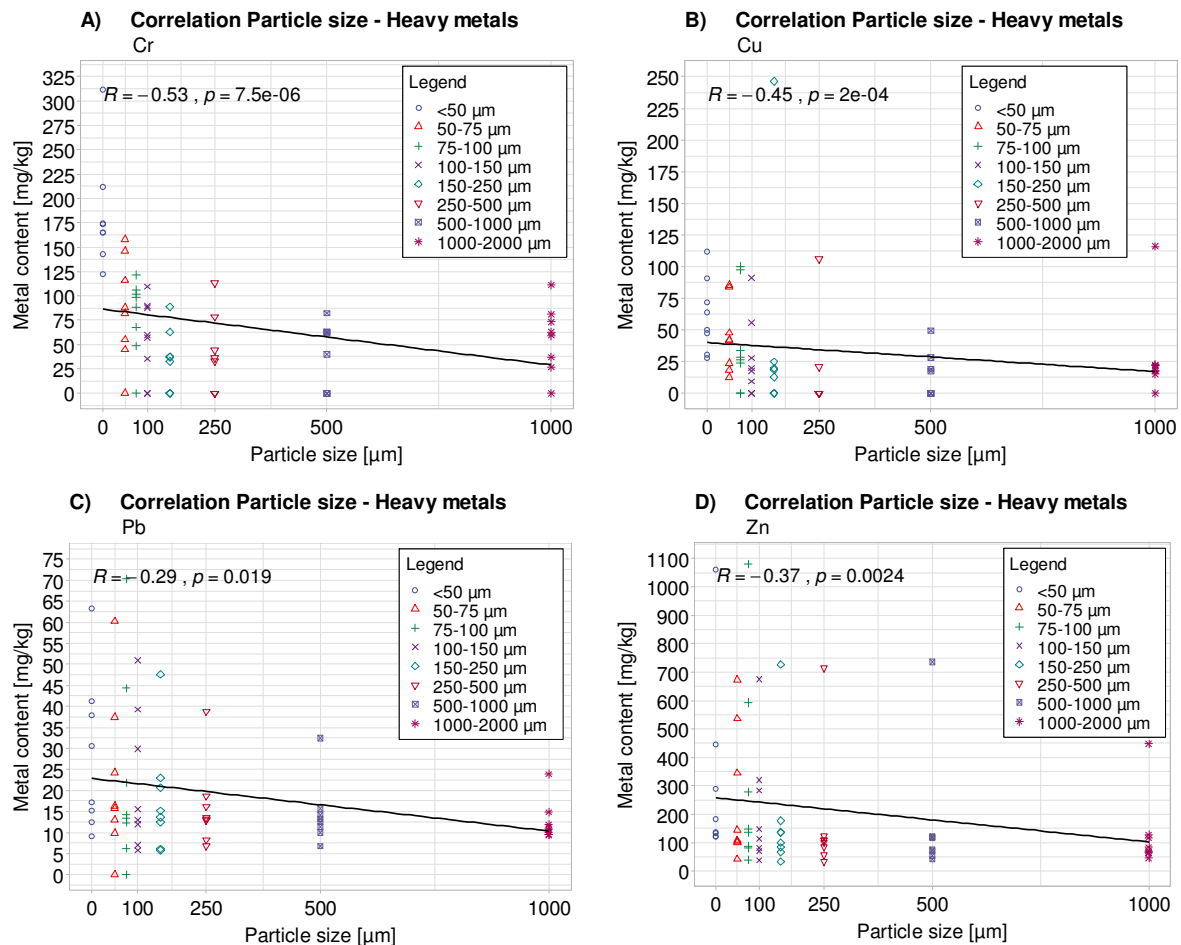
For zinc (Figure 10, E) a similar manner as lead was observed. The median values for the no-traffic group were higher than the traffic group throughout all size ranges. There was a large difference detected in median values in the size ranges <50 (446.11 and 142.09 mg/kg), 50-75 (535.57 and 127.44 mg/kg), 75-100 (593.50 and 134.76 mg/kg) and 100-150  $\mu\text{m}$  (319.96 and 106.98 mg/kg) and a small difference in the size ranges 150-250 (135.36 and 101.57 mg/kg), 250-500 (109.90 and 96.05 mg/kg), 500-1000 (117.38 and 76.18 mg/kg) and 1000-2000  $\mu\text{m}$  (116.16 and 72.06 mg/kg). For all size ranges except the smallest fraction the minimal scores for the no-traffic group were located around 30 mg/kg and 60 mg/kg for the traffic group. In the size range <50  $\mu\text{m}$  the minimal scores for both groups were located around 100 mg/kg. The maximum score for the traffic group was found as an outlier around 375 mg/kg in the size range 50-75  $\mu\text{m}$  and for the no-traffic group at 1270 mg/kg in the size range 500-1000  $\mu\text{m}$ . The interquartile ranges in the group without traffic were widely spread throughout all size ranges reaching values with the upper quartile between 625 and 1000 mg/kg. However, the lower quartiles were found to be below the lower quartiles of the group with traffic, means below around 60 mg/kg. In contrast, interquartile ranges for the group with traffic are close together with a maximum span width of around 130 mg/kg and squeezed in the size ranges 500-1000 and 1000-2000  $\mu\text{m}$ . In general, there was a higher metal content in small size ranges for both groups with a smooth decrease from <50  $\mu\text{m}$  with increasing particle sizes for the traffic group. In the no-traffic group median values increased from <50  $\mu\text{m}$  to 75-100  $\mu\text{m}$  and dropped abruptly.



**Figure 10.** Heavy metal content [mg/kg] in sieved size ranges displayed as a box-and-whisker plots for the no-traffic and traffic group. The median value as a representative value of all 7 XRF-shots were used as input values for each size range. **A)** Cr; **B)** Cu; **C)** Fe; **D)** Pb and **E)** Zn.

### 3.2.2 Correlation between particle size and heavy metal content

Correlations between particle size and the heavy metals Cr, Cu, Fe, Pb and Zn were calculated. There were found correlations between particle size and Cr, Cu, Pb and Zn, as shown in Figure 11. All correlations were found to be statistically significant with negative correlation coefficients in a moderate range from  $R = -0.29$  to  $R = -0.53$ . There was no statistically significant correlation between particle size and Fe.



**Figure 11.** Spearman rank-order correlation between particle size [μm] and heavy metal content [mg/kg] for sieved size ranges from wet sieving for particles between 50 and 2000 μm; (<50 μm is equal to 0 etc.).

## 4 Discussion

In this paper there have been addressed the following research questions, which will be discussed in the next chapters.

- What is the characterization of the particle size distribution in sediment beds in gully pots in Trondheim in areas with different landuse and traffic loads?
- To which extent are the sediment beds contaminated by heavy metals and is there a difference between different landuse and traffic loads?
- What suggestions can be derived for prospective stormwater treatment decisions for the municipality of Trondheim based on the findings from part 1 and 2?

### 4.1 Size characterization of particles in sediment beds

Both applied methodical approaches for determining particle size characteristics, wet sieving for particles between 50 and 2000 μm and measurements using a Coulter counter instrument for particles <50 μm cannot be combined, but they can be considered as a complement. This

is attributable to different basics of each procedure measuring the distribution of particles: weight-based for wet sieving and volume- or number-based recordings for the Coulter counter each with another method defining an equivalent spherical diameter of non-spherical particles (Jennings and Parslow, 1988). As can be seen in Figure 5, B) and C), the particle size distributions of volume- and number-based recordings differed extremely from each other. The differential number-based curves showed the most particles being in the range between 0.06 and 0.10  $\mu\text{m}$  for applied particles  $<50 \mu\text{m}$ , whereas the volume-based differential curves indicated the most particles in the range from 5 to 40  $\mu\text{m}$ . It is thought that the number-based determination is more appropriate for the purpose of focusing on the small particles using the Coulter counter instrument due to a believed underestimation of the smaller particles using the volume-based results. This assumption is in line with Drapper et al. (2000).

In general, there was a little difference observed between the particle size distributions using a dispersant or not, both for wet sieving and measurements in the Coulter counter (Figure 5, A), B) and C)). However, measurements of zeta-potentials of dispersed and non-dispersed particles (see Appendix B.5.2) demonstrated the effectiveness of the applied dispersant in form of high negative zeta-potentials for dispersed particles. It could be inferred therefore that potential agglomerations are not strong enough and are easily separated by applied movements and flushing of the sieves and the power of the circulation pump in the Coulter counter. This, in turn, could point the importance of frequently maintained gully pots with minimized turbulences to provide an appropriate settling chance. However, the weak negative correlation for sieving procedure and the strong negative correlation for the Coulter counter between particle size and the effect of a dispersant (Figure 8, A) and B)) infer that there is a tendency for a higher effect of a dispersant for smaller particles than for the large fractions.

As evident from Figure 5, A), a striking differential percentage in the range from 31 to 37 % for particles  $<50 \mu\text{m}$  in comparison to larger fractions were found mostly independent from different landuse. The estimated particle diameter  $d_{50}$  was found to be between  $<50$  and 700  $\mu\text{m}$ , but mostly in the range between  $<50$  and 350  $\mu\text{m}$  (see Figure 5, D)). Similar findings were reported by Jartun et al. (2008) and Poletto et al. (2009). However, findings from studies conducted by Pratt and Adams (1984), Deletic et al. (2000) and Karlsson and Viklander (2008) are not in line with results of the current study. They noted particle size distributions in sediment beds with particle diameter  $d_{10}$  between 80 and 120  $\mu\text{m}$  and medians in the range between 200 and 1750  $\mu\text{m}$ . Furthermore, Grottker (1990) found differential fractions in the range from 0.5 to 1.5 % for particles  $<25 \mu\text{m}$  and 8.0 to 13.0 % for particles between 25 and 80  $\mu\text{m}$ , with similar divided size ranges. These significant variations in investigated particle size distributions among mentioned studies is assumed to be related to both spatial and temporal differences (Monrabal-Martinez et al., 2018) and different applied sampling strategies, particle size

analyzing methods and different selected mesh sizes for sieving or a different applied particle size range for using a Coulter counter instrument. In this investigation, prior conducted tests in search of a robust and efficient method (Appendix B) have shown a considerable importance in accuracy, e.g. when flushing the sieves due to a bulk of attached fine particles on large particles  $>2000\ \mu\text{m}$ . Therefore, but also because of a lack of aforementioned information in several reports about applied methods it is difficult and imprecise to compare the results.

There is the assumption that the bulk of fine particles might either originate from biological activities due to organic matter in sediment beds or as a result of an efficient sedimentation. The latter case may be either related to cohesive properties of particles with respect to organic matter and specific gravity (Clegg et al., 1992) or to hydrophobic properties with respect to available oil on roads with associated agglomeration effects.

Comparing Figure 5, A) and Figure 6 considering the size range  $<50\ \mu\text{m}$  in the traffic and no-traffic group there was a slight difference in differential sieving amounts, but an extremely high variation in ignition loss in evidence. This relation raises concern about the organic matter and biological activity related to decomposition of sediments being the reason for the bulk of small particles found.

The low specific gravity of the smallest fraction  $<50\ \mu\text{m}$  in comparison to the other size ranges (see Figure 7) might argue for an enhanced sedimentation based on cohesive properties (Clegg et al., 1992) as a reason for the high bulk of small particles found. Moreover, inspection of Figure 9 might support this potential relation. However, results provided by current investigation do not indicate to what extent oil would be a responsible factor in case of an enhanced sedimentation. But the fact remains, that there were hydrophobic effects experienced in the test phase of current study (see Appendix B.3).

Drapper et al. (2000) formulated the hypothesis in their study that heavy metals could possibly be a responsible factor for an enhanced sedimentation of the smallest particles. However, for adapting on this assumption it needs to be proved in a more detailed way.

Further studies in which road runoff samples before entering a gully pot will be taken are necessary for investigating the origin of small particles found in RSGP.

## **4.2 Characterization of heavy metals in sediment beds**

For evaluation of heavy metals it must be mentioned that “no traffic” samples were taken from the pedestrian area in the middle of the city center from Thomas Angells Gate. “Traffic” samples were mostly from residential areas with two commercial areas including bus traffic.



There are both similarities and differences in investigated contents of heavy metals in sediment beds in comparison to previous studies conducted by Grottker (1990), Jartun et al. (2008) and Karlsson and Viklander (2008). All these studies reported heavy metal contents for the whole size range <2000 µm in one area mainly with traffic with the exception of Karlsson and Viklander (2008) who divided into two groups, namely “housing areas” and “road areas” and into different size ranges. Hence, heavy metals investigated in this study will be compared in quantity regardless different size ranges using the mean of the size range medians for each group (not displayed).

Jartun et al. (2008) and Karlsson and Viklander (2008) applied destructive ICP-AES (Inductively Coupled Plasma Atomic Emission Spectrometer) with prior sample modification using nitric acid, whereas no information about applied method was given in Grottker’s (1990) study.

The results in current study for chrome in a range from 69 (no traffic) to 74 mg/kg (traffic) were closer to what Grottker (1990) found (mean 51 mg/kg), but differed more from Jartun et al. (2008) and Karlsson and Viklander’s (2008) results (both in the range between 13 and 25 mg/kg).

The mean values of the size range medians for copper (21 mg/kg for traffic and 46 mg/kg for no traffic) were in accordance with the results (19 mg/kg for “housing area” and 44 mg/kg for “road areas”) from Karlsson and Viklander (2008), but changed within the groups. However, copper values from Grottker (1990) (394 mg/kg) and Jartun et al. (2008) (97 mg/kg) are in contrast to findings of current study.

Both Grottker (1990) and Jartun et al. (2008) have demonstrated values of iron which are more or less in line with values around 27000 to 34200 mg/kg from present study. Karlsson and Viklander (2008) did not report about iron.

Values for lead were found to be between 12 mg/kg for the traffic area and 29 mg/kg for the no-traffic area. These results are in close agreement with those obtained by Karlsson and Viklander (2008) (28 mg/kg for “housing areas” and “road areas”). However, these values vary to some extent from findings reported by Jartun et al. (2008) (61 mg/kg), but significantly from lead values Grottker (1990) demonstrated (1170 mg/kg).

For the heavy metal zinc, the medians in the two groups traffic and no-traffic were calculated to 107 mg/kg and 297 mg/kg, respectively. Similar findings with some variance were reported by Jartun et al. (2008) (403 mg/kg) and Karlsson and Viklander (2008) (65 mg/kg for “housing area” and 110 mg/kg for “road areas”). In contrast to these findings Grottker (1990) observed a value for zinc around 2000 mg/kg.

It would appear that most deviations in results among the different studies are either attributed to the choice of sampling strategy and analytical method determining heavy metals in samples or to differences in spatial characteristics of each gully pot. Grottker (1990) reported heavy metal concentrations in sediments investigated from merely three gully pots. This increases the chance of evaluating exceptional cases due to high temporal and spatial variations for each site (Monrabal-Martinez et al., 2018) what could be a reason for the frequent deviations from his study. A study conducted by Rouillon and Taylor (2016) showed that XRF-analysis measurements are comparable to results achieved using the ICP-AES method in terms of heavy metal polluted soils. However, comparison of results from different methods should be considered with caution.

With respect to a qualitative evaluation within the size ranges it could be identified that the smallest particle fraction  $<50\ \mu\text{m}$  for both groups contains the highest bulk of metals Cr, Cu and Pb. For Fe in the no-traffic group and Zn in the traffic group the highest concentration of metals was also found in the smallest particle fraction  $<50\ \mu\text{m}$ . For Fe in the traffic group the highest bulk of metals was detected in the largest size fraction  $>2000\ \mu\text{m}$ , whereas for Zn for the no-traffic group the metal content was highest in the fraction from 75 to  $100\ \mu\text{m}$ . This strong tendency of highest heavy metal concentrations being mostly in the smallest particle size fractions which was proved by a statistically significant correlation (see Figure 11) was confirmed by results from Karlsson and Viklander (2008). This result is also verified by early studies indicating the relation to the large particle surface area from small particles (Wang et al., 1998), (Sansalone et al., 1998). It is most likely that in larger fractions the grit by itself is measured whereas smaller fractions might tend to indicate attached matter.

With regard to differences in heavy metal content in the two different areas, it can be said that heavy metal concentrations were higher in the no-traffic group for Cu, Pb and Zn. For Fe and Cr metal values were higher in the traffic group. However, Karlsson and Viklander (2008) reported higher metal values in the "road areas" except for Pb. The difference might be related to spatial differences.

Concerning comparisons in heavy metal content within the size ranges there could be a striking tendency of heavy metal distribution observed in both groups for Cr, Cu and Fe. Concentrations were the highest in the smallest fractions, decreased with increasing particle size until the size range 250-500  $\mu\text{m}$  and then increased again in the last two size ranges (see Figure 10, A), B) and C)). Pb and Zn showed an equal metal distribution. In traffic areas the metal concentrations were almost the same within sizes with a slight decrease with increasing particle size, whereas in the no-traffic group concentrations were equal high in the first size ranges to 100  $\mu\text{m}$  and then dropped without increasing for both metals (see Figure 10, D) and E)).

These observations, except Zn are in an exceptional agreement with heavy metal distributions reported by Karlsson and Viklander (2008) with comparable similar chosen size ranges.

In general, there was a higher variety within the size ranges in the no traffic group for Cu, Pb and Zn. This observed trend is in accordance with Karlsson and Viklander (2008).

Table 3 shows investigated heavy metals in urban soils measured in soil samples distributed throughout Trondheim and along main roads in 2004 by Andersson et al. (2006). Heavy metals were analyzed by using the nitric acid-based ICP-AES method on <2000 µm samples taken between 1-2 cm from the soil surface (Andersson et al., 2006).

A comparison between heavy metal content found in sediment bed samples of RSGP (see also Table 3) and metal content in urban soils may provide information about additive anthropogenic pollution. However, it must be mentioned that anthropogenic pollution (Muschack, 1990) is already present in urban soils, especially for Cu, Cr, Pb and Zn (Andersson et al., 2006).

**Table 3. Columns 2 and 3:** Means of median values of 7 XRF-shots for each size range of heavy metal content [mg/kg] in sediment beds from RSGP in traffic and no-traffic areas; **Column 4:** Highest median of 7 XRF-shots from size ranges for each metal; **Columns 5 and 6:** Median values of heavy metal content [mg/kg] in urban soils sampled 2004 all over the city and along main roads (Andersson et al., 2006) for Cr, Cu, Fe, Pb and Zn.

| Metal                      | Median [mg/kg]  |  |   |  |   |
|----------------------------|---|--|---|--|---|
|                            | Sediments<br>Recent study<br>No-traffic area<br>n = 3 | Sediments<br>Recent study<br>Traffic area<br>n = 5 | Sediments<br>Recent study<br>Highest value<br>n = 8 | Urban soils<br>External study *<br>Whole city<br>n = 321 | Urban soils<br>External study *<br>Along main roads<br>n = 16 |
| Cr                         | 69  | 74   | 220 (<50 µm)  | 58   | 60  |
| Cu                         | 46  | 21   | 91 (<50 µm)   | 32   | 34  |
| Fe                         | 26952   | 34199  | 45898 (1-2 mm)                                      | 28063  | 27144   |
| Pb                         | 29  | 12   | 44 (<50 µm)   | 32   | 54  |
| Zn                         | 297   | 107  | 594 (75-100 µm)                                     | 80   | 112   |
| * (Andersson et al., 2006) |   |  |   |  |   |
| ** n = quantity of samples |   |  |   |  |   |

Following discussion parts concern columns 2, 3, 5 and 6 from Table 3.

Cr values are slightly increased for the RSGP sediment samples which means that there is a low additive pollution compared to urban soils. This observation is confirmed by Ljung et al. (2006) who found a mainly natural origin for Cr. The difference in traffic areas may be explainable by tire and brake pad wear as a most common source for chrome (Afsaneh et al., 2018).

Drainage from copper roof paneling in the town's oldest district, the city center appears to be a possible reason for the higher values in the no-traffic area compared to urban soils (Förster, 1996), (He et al., 2001).

In general, most of the iron is of natural origin (Tijhuis et al., 2002), (Ljung et al., 2006). However, Table 3 shows a minor increased value in metals between urban soils and gully pot sediments in traffic areas which may indicate an additive input of iron more related to traffic activities than to city center surrounding characteristics which may be explained by contents of iron in gravel applied as anti-slip agents in Trondheim (Norges geologiske undersøkelse, 2019) or metal corrosion products related to traffic activities (Miguel et al., 1997). Amemiya et al. (1984) could prove the relation between an increase in dust concentration and the use of studded tires. This could indicate another source of Fe on roads related either to an abrasion of asphalt (Amemiya et al., 1984) or wear of studded tires (Fukuzaki et al., 1986), (Hääl et al., 2004).

Regarding the traffic area, both for urban soils and RSGP sediments there is a significant difference in lead content evident. It seems plausible that this might be related to a slowly but steady decrease in still existing lead leftovers due to the use of leaded petrol in the past (Nriagu, 1990), (Callender and Rice, 2000). This observation correlates with the study from Andersson et al. (2006) who also noted a decrease in lead contents in comparison to urban soil values from 1994. The remarkable increased lead content in RSGP sediments in the city center compared to roads could be explained by drainage of leaded roof paneling (Gromaire-Mertz et al., 1999), in the older city part (Garnaud et al., 1999) and remained leaded paintings on old buildings from the past before restriction regulations (Davis and Burns, 1999).

The observed zinc value in sediment beds in traffic areas is in line with reported zinc contents in urban soils (Andersson et al., 2006). However, the zinc value in sediment beds in the city center are considerably increased. This might be attributable to “corrosion of galvanized metal” (Callender and Rice, 2000) in the form of roof paneling together with a fairly high runoff due to the high degree of impervious surfaces in the city center (Förster, 1996). Increased zinc values related to roof runoff in comparison to road runoff was confirmed by several studies, e.g. Gromaire-Mertz et al. (1999), Gnecco et al. (2005) and Afsaneh et al. (2018).

There was a significant difference between heavy metal content in urban soils and the highest values found, concerning size ranges for all heavy metals, except for Pb (see Table 3, column 4). This suggests that there is a higher degree in additive anthropogenic pollution mainly in the smaller size fractions.

## **5 Conclusion**

This work studied sediment removal performance of RSGP within different landuse and ADT in Trondheim, Norway. Therefore, PSD in sediment beds from RSGP have been investigated by applying manual wet sieving for particles between 50 and 2000  $\mu\text{m}$  and a Coulter counter

LS230 instrument for particles <50 µm. Moreover, organic matter, heavy metals via XRF analysis and specific gravity of fractions have been studied.

Main result was a remarkably high differential weight-based percentage of particles <50 µm between 31 and 37 % in contrast to fractions from 50 to 2000 µm showing percentages between 3 and 18 %. Particle size distribution was mainly independent from different landuse, evaluated based traffic and no-traffic areas. The magnitude of small particles is most likely originating either from biological activities due to organic matter or as a result of an efficient sedimentation due to cohesive properties or hydrophobic properties of particles associated with agglomeration effects from available oil on roads. As a further step, road runoff entering the examined RSGP from this study needs to be analyzed in order to investigate the origin of small particles <50 µm found in sediment beds.

Another main finding was that the highest median concentrations of heavy metals were found in the smallest size range <50 µm in both groups (no traffic and traffic, respectively) for Cr (220 and 155 mg/kg), Cu (91 and 43 mg/kg), Pb (44 and 15 mg/kg) and partly for Fe in the no-traffic group (39944 mg/kg) and for Zn in the traffic group (142 mg/kg). Metal concentrations were higher in the city center (no-traffic group) than on roads (traffic group) for Cu, Pb and Zn. Concerning the degree of additive anthropogenic pollution in comparison to urban soil there was found a marginal increase in metal concentration (combined values regardless particle size) in RSGP sediment beds for Cr, Cu, Fe and Zn for the traffic group. Concentration of Zn were found to be increased more than double in the city center (no-traffic group). Pb values in both groups (traffic and no-traffic) were appreciable lower than measured concentration in urban soils by Andersson et al. (2006). The highest metal concentrations mainly in the smallest size range <50 µm exceeded the urban soil values almost more than three times for all metals, except for Pb. These findings indicate a presumably drainage of metal roof paneling and lead paintings from the past in the city center into RSGP and a steady decrease in lead related to traffic activities.

In a final analysis, these results indicate a better performance of RSGP in Trondheim than probably expected. If the enhanced sedimentation caused by agglomerations of small particles will be verified by further investigations concerning the supply to RSGP, there is no need in investing in more expensive and further developed treatment devices suggested. However, this presumes a regular emptying of existing RSGP. If there will be a confirmation of more efficient BMP (Best Management Practices), such as Vortex Separators which is currently under field examination, based on current findings it is suggested to install them preferably in the older parts of the city with metal roof paneling and still available leaded paintings on buildings directly draining into gully pots due to a high degree of impervious areas. Since current results

are covering a little section of the existing 15000 RSGP in Trondheim in a narrow temporal span which allows only a rough evaluation, further analyses of additional samples are needed to verify findings of this study.

## Acknowledgements

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The following reference list includes literature cited in the manuscript and in the appendices.

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# Appendix A – Raw data

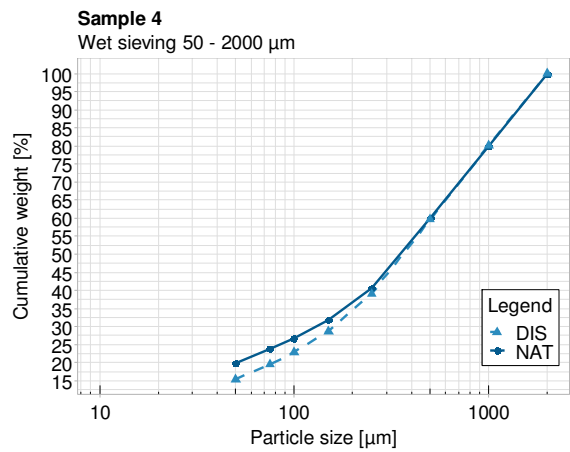
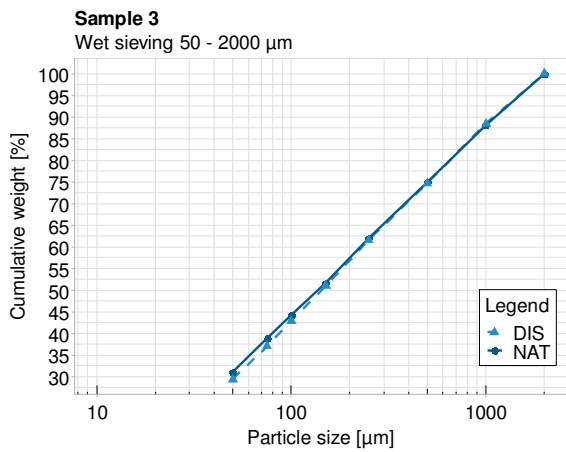
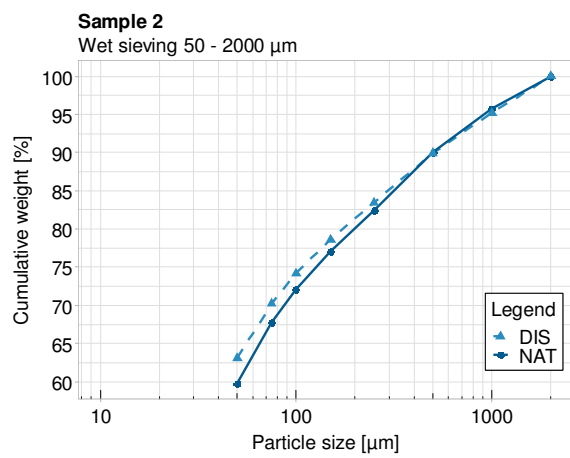
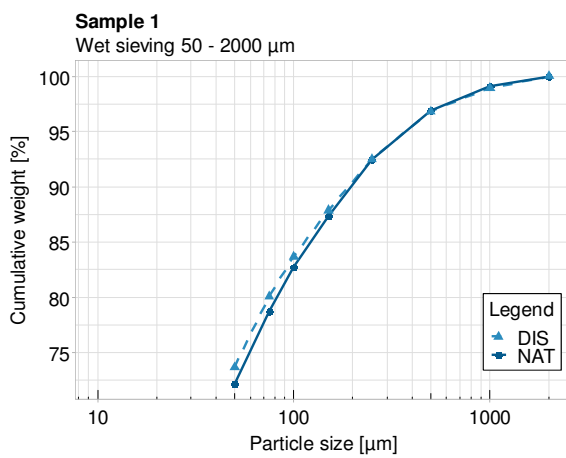
This appendix contains raw data of conducted laboratory tests.

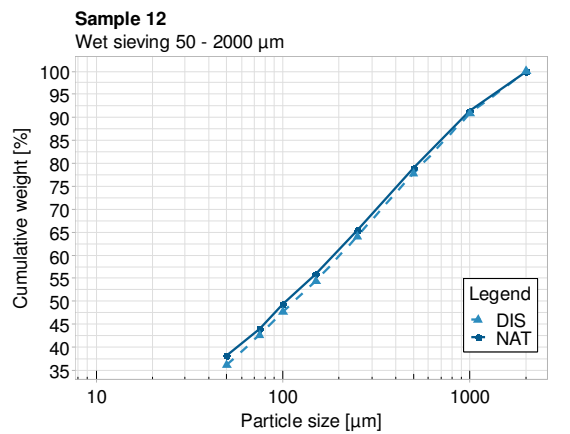
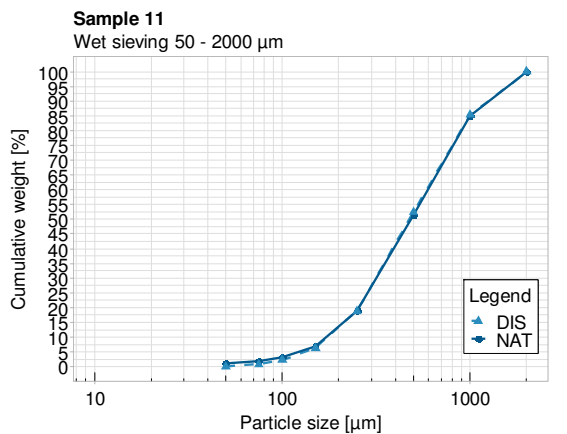
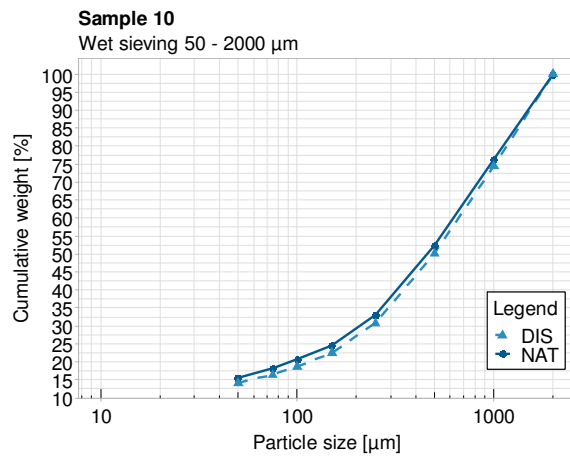
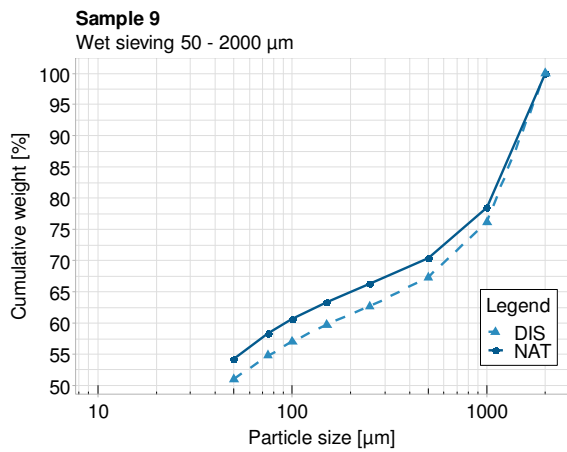
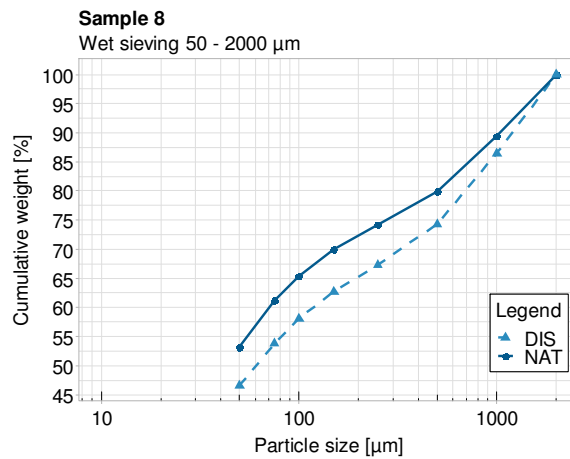
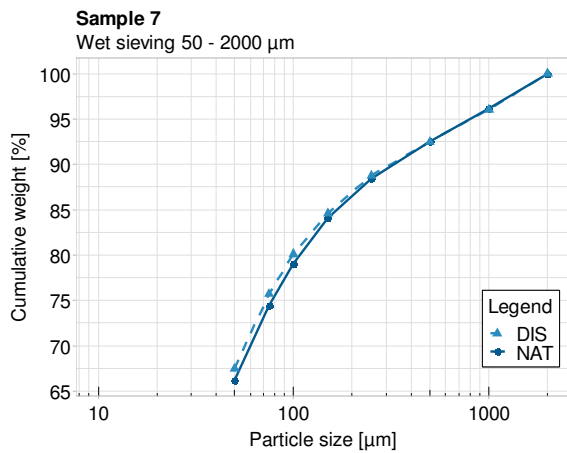
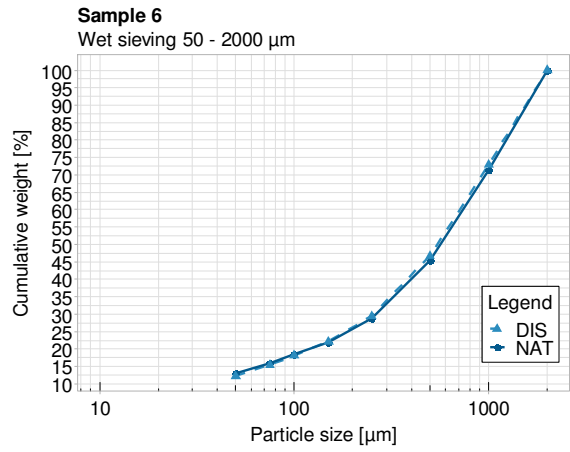
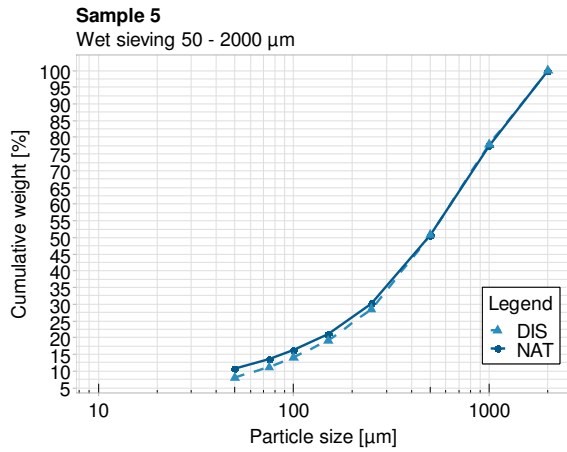
## A.1. Particle size distribution

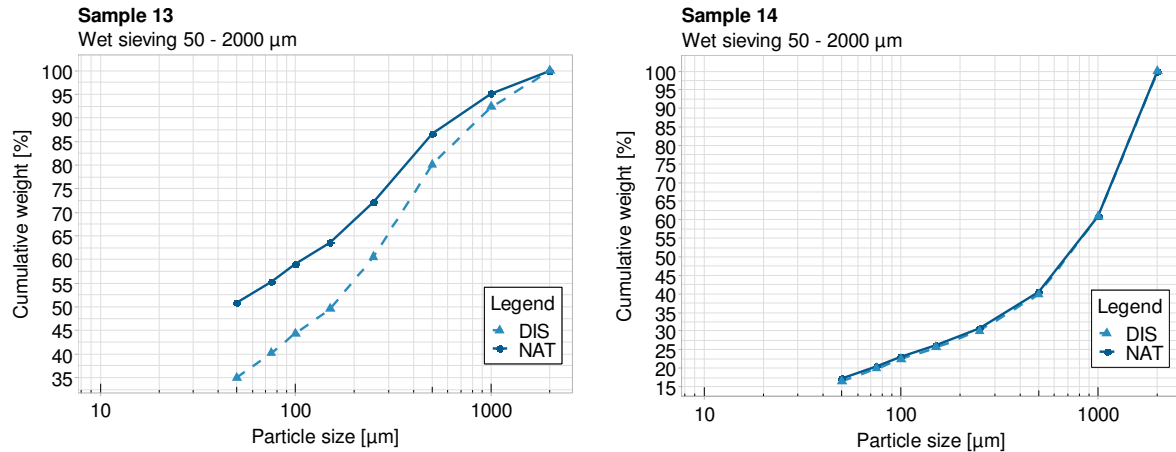
The results for PSD are illustrated in the following paragraphs.

### A.1.1. Wet sieving 50 – 2000 $\mu\text{m}$

The cumulative particle size distribution from the wet sieving procedure for particles between 50 and 2000  $\mu\text{m}$  are shown in the diagrams below. The points indicate the sieve mesh sizes 50, 75, 100, 150, 250, 500, 1000 and 2000  $\mu\text{m}$ .







**Figure 12.** Appendix A.1.1. Cumulative particle size distribution [%] for wet sieving for particles between 50 and 2000 µm, with and without a dispersant.

Table 4 shows the differential fractions from wet sieving for particles between 50 and 2000 µm and estimated particle diameters  $d_{10}$ ,  $d_{50}$  and  $d_{90}$  [µm] for each sample sorted by groups.

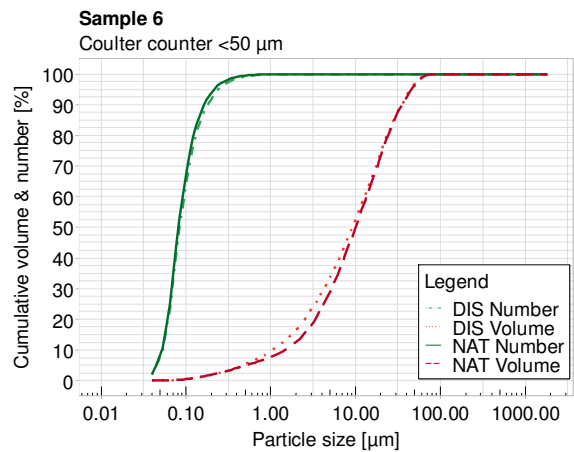
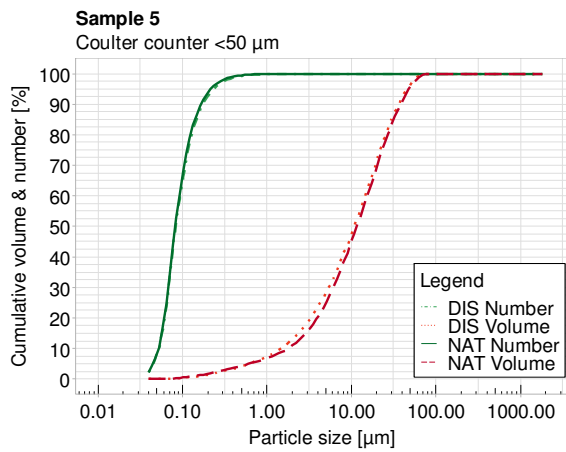
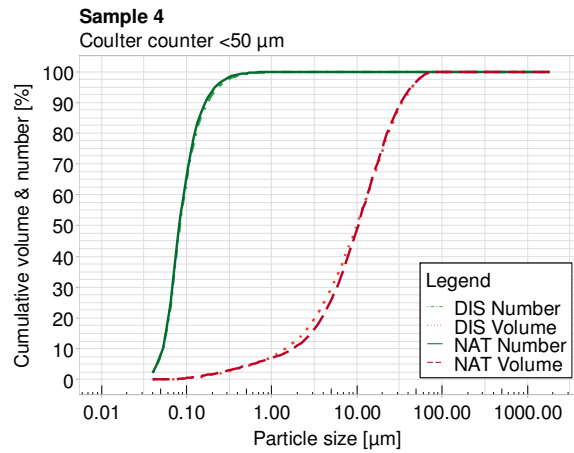
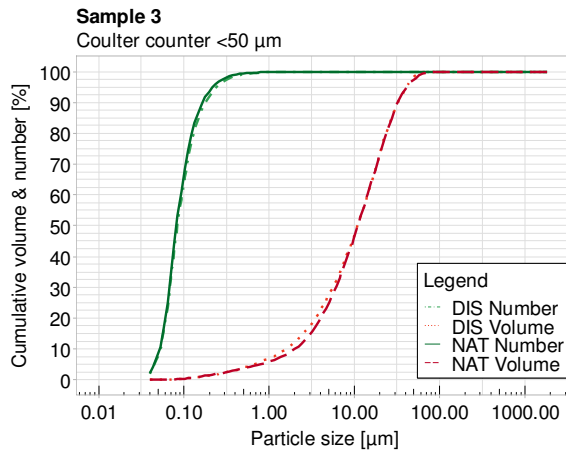
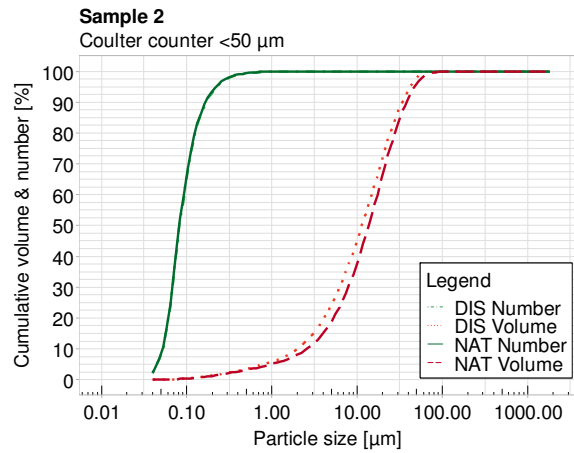
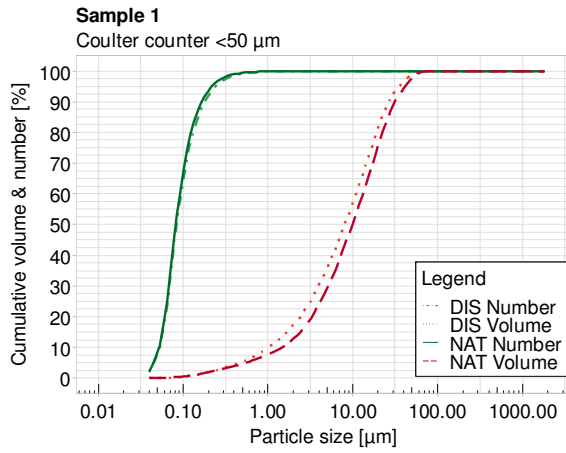
**Table 4.** Appendix A.1.1. Differential particle size distribution: values [%] for wet sieving for particles between 50 and 2000 µm and estimated particle diameter  $d_{10}$ ,  $d_{50}$  and  $d_{90}$  [µm] with and without a dispersant.

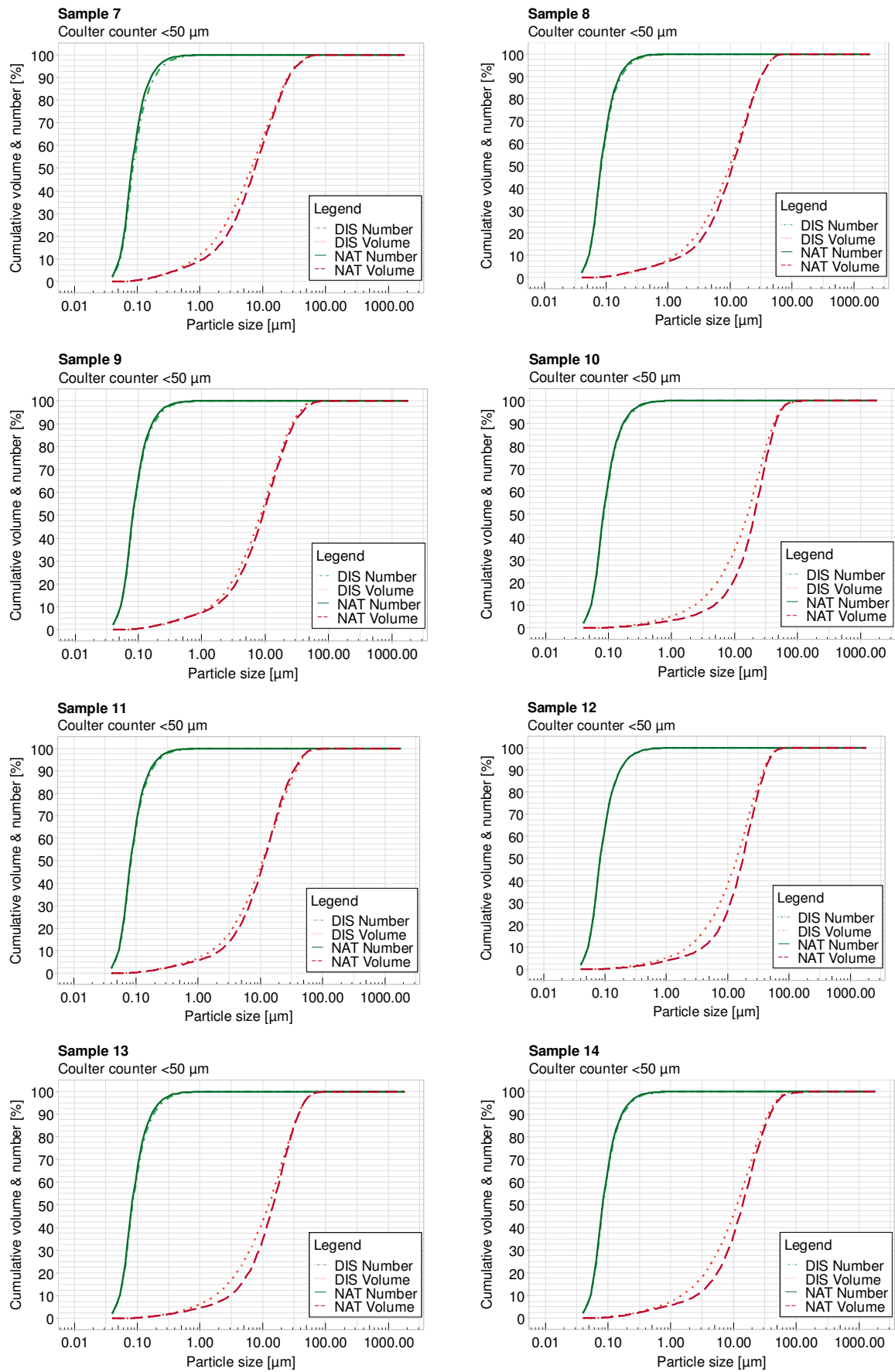
| Area type  | Gully pot | PSD fractions [%] |          |           |            |            |            |             |              | [µm]       |            |            |      |
|------------|-----------|-------------------|----------|-----------|------------|------------|------------|-------------|--------------|------------|------------|------------|------|
|            |           | <50 µm            | 50-75 µm | 75-100 µm | 100-150 µm | 150-250 µm | 250-500 µm | 500-1000 µm | 1000-2000 µm | $d_{10}^*$ | $d_{50}^*$ | $d_{90}^*$ |      |
| No traffic | 1         | NAT               | 72.14    | 6.57      | 4.00       | 4.66       | 5.08       | 4.47        | 2.17         | 0.91       | <50        | <50        | 200  |
|            |           | DIS               | 73.69    | 6.43      | 3.57       | 4.17       | 4.64       | 4.29        | 2.14         | 1.07       | <50        | <50        | 200  |
|            | 2         | NAT               | 59.72    | 8.06      | 4.27       | 4.98       | 5.45       | 7.58        | 5.69         | 4.27       | <50        | <50        | 500  |
|            |           | DIS               | 63.10    | 7.13      | 3.98       | 4.40       | 4.82       | 6.50        | 5.24         | 4.82       | <50        | <50        | 500  |
|            | 10        | NAT               | 15.61    | 2.71      | 2.32       | 3.87       | 8.52       | 19.35       | 23.87        | 23.74      | <50        | 450        | 1500 |
|            |           | DIS               | 14.11    | 2.31      | 2.17       | 3.93       | 8.28       | 19.27       | 24.29        | 25.64      | <50        | 500        | 1500 |
| 11         | NAT       | 1.05              | 0.73     | 1.38      | 3.73       | 12.08      | 32.44      | 33.41       | 15.17        | 175        | 500        | 1250       |      |
|            | DIS       | 0.00              | 0.87     | 1.39      | 4.08       | 12.59      | 33.42      | 32.99       | 14.67        | 175        | 500        | 1250       |      |
| 12         | NAT       | 38.16             | 5.83     | 5.48      | 6.36       | 9.54       | 13.60      | 12.37       | 8.66         | <50        | 100        | 950        |      |
|            | DIS       | 36.13             | 6.52     | 5.03      | 6.70       | 9.68       | 13.78      | 13.04       | 9.12         | <50        | 125        | 950        |      |
| Traffic    | 3         | NAT               | 31.06    | 7.74      | 5.43       | 7.34       | 10.45      | 12.86       | 13.27        | 11.86      | <50        | 150        | 1000 |
|            |           | DIS               | 29.38    | 7.68      | 5.83       | 8.09       | 10.64      | 13.10       | 13.72        | 11.57      | <50        | 150        | 1000 |
|            | 4         | NAT               | 19.90    | 3.84      | 3.03       | 4.95       | 8.89       | 19.29       | 20.10        | 20.00      | <50        | 350        | 1500 |
|            |           | DIS               | 15.35    | 4.09      | 3.49       | 5.68       | 10.47      | 20.54       | 20.44        | 19.94      | <50        | 350        | 1500 |
|            | 5         | NAT               | 10.56    | 3.02      | 2.70       | 4.76       | 9.29       | 20.48       | 26.59        | 22.62      | 50         | 500        | 1500 |
|            |           | DIS               | 7.96     | 3.20      | 2.85       | 5.10       | 9.43       | 22.23       | 27.08        | 22.15      | 65         | 500        | 1500 |
|            | 6         | NAT               | 13.07    | 2.99      | 2.33       | 3.55       | 6.82       | 16.53       | 26.14        | 28.57      | <50        | 600        | 1600 |
|            |           | DIS               | 12.21    | 3.29      | 2.58       | 4.11       | 7.28       | 17.25       | 26.17        | 27.11      | <50        | 550        | 1600 |
|            | 7         | NAT               | 66.12    | 8.24      | 4.68       | 4.98       | 4.37       | 4.07        | 3.66         | 3.87       | <50        | <50        | 350  |
|            |           | DIS               | 67.45    | 8.24      | 4.39       | 4.50       | 4.18       | 3.75        | 3.53         | 3.96       | <50        | <50        | 350  |
|            | 8         | NAT               | 53.16    | 8.01      | 4.25       | 4.61       | 4.13       | 5.83        | 9.47         | 10.56      | <50        | <50        | 1000 |
|            |           | DIS               | 46.58    | 7.22      | 4.30       | 4.56       | 4.68       | 6.96        | 12.15        | 13.54      | <50        | 60         | 1100 |
|            | 9         | NAT               | 54.24    | 4.16      | 2.24       | 2.72       | 2.88       | 4.16        | 8.16         | 21.44      | <50        | <50        | 1500 |
|            |           | DIS               | 50.99    | 3.78      | 2.30       | 2.63       | 2.96       | 4.61        | 8.88         | 23.85      | <50        | <50        | 1500 |
| 13         | NAT       | 50.80             | 4.47     | 3.83      | 4.47       | 8.63       | 14.38      | 8.63        | 4.79         | <50        | <50        | 650        |      |
|            | DIS       | 34.99             | 5.23     | 4.13      | 5.23       | 11.02      | 19.56      | 12.12       | 7.71         | <50        | 150        | 900        |      |
| 14         | NAT       | 17.23             | 3.35     | 2.44      | 3.20       | 4.57       | 9.76       | 20.43       | 39.02        | <50        | 700        | 1750       |      |
|            | DIS       | 16.49             | 3.38     | 2.54      | 3.17       | 4.44       | 9.94       | 20.93       | 39.11        | <50        | 700        | 1750       |      |

\* estimated values

### A.1.2. Coulter counter <50 $\mu\text{m}$

The cumulative particle size distribution, both volume- and number-based from using the Coulter counter for particles <50  $\mu\text{m}$  are shown in the diagrams below.





**Figure 13.** Appendix A.1.2. Cumulative particle size distribution [%] using a Coulter counter, both volume- and number-based for particles <50 μm, with and without a dispersant.



Table 5 shows size characteristics of each gully pot, both volume- and number-based.

**Table 5.** Appendix A.1.2. Particle size diameter  $d_{10}$ ,  $d_{50}$ ,  $d_{90}$ ,  $d_{mean}$ ,  $d_{mode}$ , S.D. [ $\mu\text{m}$ ] for Coulter counter, both volume- and number-based for particles  $<50 \mu\text{m}$ , with and without a dispersant.

| Area type  | Gully pot | Volume-based                  |                               |                               |                           |                           |                           | Number-based                  |                               |                               |                           |                           |                           |        |
|------------|-----------|-------------------------------|-------------------------------|-------------------------------|---------------------------|---------------------------|---------------------------|-------------------------------|-------------------------------|-------------------------------|---------------------------|---------------------------|---------------------------|--------|
|            |           | $d_{10}$<br>[ $\mu\text{m}$ ] | $d_{50}$<br>[ $\mu\text{m}$ ] | $d_{90}$<br>[ $\mu\text{m}$ ] | Mean<br>[ $\mu\text{m}$ ] | Mode<br>[ $\mu\text{m}$ ] | S.D.<br>[ $\mu\text{m}$ ] | $d_{10}$<br>[ $\mu\text{m}$ ] | $d_{50}$<br>[ $\mu\text{m}$ ] | $d_{90}$<br>[ $\mu\text{m}$ ] | Mean<br>[ $\mu\text{m}$ ] | Mode<br>[ $\mu\text{m}$ ] | S.D.<br>[ $\mu\text{m}$ ] |        |
| No traffic | 1         | NAT                           | 1.614                         | 10.860                        | 34.870                    | 15.180                    | 19.760                    | 14.200                        | 0.0573                        | 0.0897                        | 0.1820                    | 0.1130                    | 0.0733                    | 0.1180 |
|            |           | DIS                           | 1.120                         | 8.859                         | 29.380                    | 12.750                    | 18.000                    | 12.450                        | 0.0582                        | 0.0921                        | 0.1970                    | 0.1200                    | 0.0805                    | 0.1300 |
|            | 2         | NAT                           | 2.853                         | 15.070                        | 41.910                    | 19.290                    | 19.760                    | 16.510                        | 0.0575                        | 0.0900                        | 0.1840                    | 0.1140                    | 0.0733                    | 0.1240 |
|            |           | DIS                           | 2.181                         | 12.610                        | 37.460                    | 16.560                    | 19.760                    | 14.230                        | 0.0577                        | 0.0907                        | 0.1870                    | 0.1160                    | 0.0805                    | 0.1290 |
|            | 10        | NAT                           | 5.206                         | 22.950                        | 49.860                    | 26.020                    | 28.700                    | 18.700                        | 0.0579                        | 0.0911                        | 0.1900                    | 0.1160                    | 0.0733                    | 0.1360 |
|            |           | DIS                           | 2.529                         | 17.770                        | 46.890                    | 22.060                    | 23.810                    | 19.180                        | 0.0582                        | 0.0920                        | 0.1960                    | 0.1200                    | 0.0805                    | 0.1370 |
| 11         | NAT       | 2.426                         | 12.630                        | 37.910                        | 16.970                    | 16.400                    | 15.710                    | 0.0575                        | 0.0900                        | 0.1840                        | 0.1140                    | 0.0733                    | 0.1230                    |        |
|            | DIS       | 1.793                         | 11.980                        | 40.960                        | 16.930                    | 19.760                    | 15.680                    | 0.0581                        | 0.0917                        | 0.1940                        | 0.1180                    | 0.0805                    | 0.1310                    |        |
| 12         | NAT       | 4.382                         | 19.420                        | 45.120                        | 22.550                    | 23.810                    | 16.210                    | 0.0581                        | 0.0919                        | 0.1950                        | 0.1190                    | 0.0805                    | 0.1420                    |        |
|            | DIS       | 2.468                         | 15.450                        | 42.690                        | 19.540                    | 21.690                    | 16.800                    | 0.0581                        | 0.0917                        | 0.1940                        | 0.1190                    | 0.0805                    | 0.1400                    |        |
| Traffic    | 3         | NAT                           | 2.207                         | 12.070                        | 35.690                    | 16.230                    | 16.400                    | 14.480                        | 0.0573                        | 0.0896                        | 0.1820                    | 0.1130                    | 0.0733                    | 0.1240 |
|            |           | DIS                           | 1.716                         | 12.010                        | 36.000                    | 15.810                    | 19.760                    | 13.900                        | 0.0582                        | 0.0920                        | 0.1960                    | 0.1200                    | 0.0805                    | 0.1360 |
|            | 4         | NAT                           | 1.920                         | 11.350                        | 36.230                    | 15.890                    | 13.610                    | 14.970                        | 0.0575                        | 0.0901                        | 0.1830                    | 0.1130                    | 0.0733                    | 0.1170 |
|            |           | DIS                           | 1.587                         | 10.900                        | 37.560                    | 15.810                    | 19.760                    | 15.340                        | 0.0581                        | 0.0917                        | 0.1930                    | 0.1190                    | 0.0805                    | 0.1310 |
|            | 5         | NAT                           | 1.930                         | 12.790                        | 42.310                    | 17.920                    | 19.760                    | 16.540                        | 0.0575                        | 0.0900                        | 0.1830                    | 0.1130                    | 0.0733                    | 0.1170 |
|            |           | DIS                           | 1.606                         | 11.980                        | 40.090                    | 16.810                    | 21.690                    | 15.730                        | 0.0581                        | 0.0915                        | 0.1930                    | 0.1180                    | 0.0805                    | 0.1300 |
|            | 6         | NAT                           | 1.621                         | 11.020                        | 39.150                    | 16.220                    | 19.760                    | 15.720                        | 0.0573                        | 0.0896                        | 0.1810                    | 0.1120                    | 0.0733                    | 0.1140 |
|            |           | DIS                           | 1.159                         | 10.110                        | 38.400                    | 15.450                    | 19.760                    | 15.670                        | 0.0583                        | 0.0924                        | 0.1980                    | 0.1210                    | 0.0805                    | 0.1290 |
|            | 7         | NAT                           | 1.240                         | 8.264                         | 28.310                    | 12.340                    | 9.370                     | 12.570                        | 0.0574                        | 0.0899                        | 0.1830                    | 0.1130                    | 0.0733                    | 0.1160 |
|            |           | DIS                           | 0.911                         | 7.269                         | 27.310                    | 11.480                    | 10.290                    | 12.300                        | 0.0587                        | 0.0940                        | 0.2070                    | 0.1240                    | 0.0805                    | 0.1350 |
|            | 8         | NAT                           | 1.902                         | 12.140                        | 35.290                    | 15.910                    | 18.000                    | 13.830                        | 0.0575                        | 0.0899                        | 0.1820                    | 0.1120                    | 0.0733                    | 0.1120 |
|            |           | DIS                           | 1.437                         | 10.910                        | 35.290                    | 15.060                    | 19.760                    | 13.920                        | 0.0580                        | 0.0914                        | 0.1930                    | 0.1180                    | 0.0805                    | 0.1290 |
|            | 9         | NAT                           | 1.684                         | 10.150                        | 31.640                    | 14.170                    | 12.400                    | 13.580                        | 0.0574                        | 0.0898                        | 0.1820                    | 0.1130                    | 0.0733                    | 0.1160 |
|            |           | DIS                           | 1.442                         | 9.300                         | 29.450                    | 12.910                    | 16.400                    | 11.940                        | 0.0579                        | 0.0911                        | 0.1910                    | 0.1180                    | 0.0805                    | 0.1300 |
| 13         | NAT       | 3.276                         | 16.050                        | 41.080                        | 19.630                    | 21.690                    | 15.740                    | 0.0577                        | 0.0906                        | 0.1880                        | 0.1150                    | 0.0733                    | 0.1300                    |        |
|            | DIS       | 1.907                         | 13.750                        | 40.810                        | 18.120                    | 21.690                    | 16.460                    | 0.0583                        | 0.0924                        | 0.2000                        | 0.1220                    | 0.0805                    | 0.1420                    |        |
| 14         | NAT       | 2.780                         | 15.390                        | 42.470                        | 20.160                    | 19.760                    | 20.250                    | 0.0600                        | 0.0900                        | 0.1800                        | 0.1100                    | 0.0700                    | 0.1200                    |        |
|            | DIS       | 1.630                         | 12.550                        | 39.570                        | 17.230                    | 19.760                    | 16.310                    | 0.0600                        | 0.0900                        | 0.1900                        | 0.1200                    | 0.0800                    | 0.1300                    |        |

## A.2. Ignition loss

The ignition loss was determined for the whole samples. In addition, the ignition loss of each fraction was examined for some gully pots (see Table 6).

**Table 6.** Appendix A.2. Ignition loss of the whole samples [%] and of fractions [%].

| Area type  | Gully pot | Ignition loss [%] |                     |                      |                       |                       |                       |                        |                         |                     | Ignition loss of whole sample [%] |
|------------|-----------|-------------------|---------------------|----------------------|-----------------------|-----------------------|-----------------------|------------------------|-------------------------|---------------------|-----------------------------------|
|            |           | <50 $\mu\text{m}$ | 50-75 $\mu\text{m}$ | 75-100 $\mu\text{m}$ | 100-150 $\mu\text{m}$ | 150-250 $\mu\text{m}$ | 250-500 $\mu\text{m}$ | 500-1000 $\mu\text{m}$ | 1000-2000 $\mu\text{m}$ | >2000 $\mu\text{m}$ |                                   |
| No traffic | 1         | -                 | -                   | -                    | -                     | -                     | -                     | -                      | -                       | -                   | 5.93                              |
|            | 2         | -                 | -                   | -                    | -                     | -                     | -                     | -                      | -                       | -                   | 14.14                             |
|            | 10        | 15.99             | 12.90               | 11.86                | 7.23                  | 3.12                  | 2.44                  | 4.62                   | 2.61                    | 7.68                | 6.22                              |
|            | 11        | No particles      | 0.00                | 0.69                 | 0.22                  | 0.35                  | 0.29                  | 0.32                   | 0.54                    | 0.85                | 0.52                              |
|            | 12        | 14.74             | 12.84               | 15.92                | 14.87                 | 14.06                 | 12.72                 | 13.00                  | 9.94                    | 8.81                | 11.19                             |
| Traffic    | 3         | -                 | -                   | -                    | -                     | -                     | -                     | -                      | -                       | -                   | 1.16                              |
|            | 4         | -                 | -                   | -                    | -                     | -                     | -                     | -                      | -                       | -                   | 2.16                              |
|            | 5         | -                 | -                   | -                    | -                     | -                     | -                     | -                      | -                       | -                   | 2.12                              |
|            | 6         | 5.05              | 4.33                | 3.08                 | 2.80                  | 1.99                  | 1.53                  | 1.38                   | 1.17                    | -                   | 1.37                              |
|            | 7         | 3.41              | 5.38                | 4.51                 | 4.12                  | 3.18                  | 2.86                  | 2.17                   | 2.21                    | 3.71                | 5.63                              |
|            | 8         | 4.85              | 5.77                | 7.57                 | 7.99                  | 7.84                  | 5.79                  | 3.88                   | 4.82                    | 2.02                | 3.53                              |
|            | 9         | 5.22              | 14.12               | 11.82                | 13.24                 | 11.26                 | 10.53                 | 7.21                   | 3.31                    | 2.00                | 2.48                              |
|            | 13        | 14.93             | 14.38               | 13.74                | 10.92                 | 6.49                  | 4.63                  | 7.66                   | 14.02                   | 12.44               | 16.02                             |
| 14         | 3.64      | 4.86              | 3.96                | 3.08                 | 3.05                  | 2.17                  | 1.72                  | 1.30                   | 0.50                    | 1.28                |                                   |

## A.3. Specific gravity of fractions

Specific gravity of fractions has been measured for following samples (see Table 7).

**Table 7.** Appendix A.3. Specific gravity [ $\text{g}/\text{cm}^3$ ] of fractions.

| Area type  | Gully pot | Specific gravity of fractions [ $\text{g}/\text{cm}^3$ ] |                     |                      |                       |                       |                       |                        |                         |
|------------|-----------|--|---------------------|----------------------|-----------------------|-----------------------|-----------------------|------------------------|-------------------------|
|            |           | <50 $\mu\text{m}$  | 50-75 $\mu\text{m}$ | 75-100 $\mu\text{m}$ | 100-150 $\mu\text{m}$ | 150-250 $\mu\text{m}$ | 250-500 $\mu\text{m}$ | 500-1000 $\mu\text{m}$ | 1000-2000 $\mu\text{m}$ |
| No traffic | 10        | 2.54   | 2.74                | 3.02                 | 2.86                  | 2.86                  | 2.78                  | 2.80                   | 2.81                    |
|            | 11        | 3.02   | 2.95                | 2.90                 | 2.85                  | 2.80                  | 2.81                  | 2.86                   | 2.85                    |
|            | 12        | 2.68   | 2.63                | 2.59                 | 2.54                  | 2.85                  | 2.72                  | 2.63                   | 2.96                    |
| Traffic    | 7         | 2.52   | 2.66                | 2.73                 | 2.85                  | 2.75                  | 2.96                  | 2.90                   | 2.98                    |
|            | 8         | 2.72   | 2.74                | 2.96                 | 2.97                  | 2.91                  | 2.73                  | 2.74                   | 2.81                    |
|            | 9         | 2.26   | 2.60                | 4.66                 | 3.86                  | 4.16                  | 2.81                  | 2.67                   | 2.79                    |
|            | 13        | 2.68   | 3.03                | 3.17                 | 2.92                  | 2.68                  | 2.78                  | 2.86                   | 2.68                    |
|            | 14        | 2.87   | 3.39                | 4.41                 | 3.67                  | 3.58                  | 3.08                  | 3.15                   | 2.95                    |

## A.4. Heavy metals

Concentrations of heavy metals Cr, Cu, Fe, Mn, Ni, Pb, Sn and Zn for each fraction are presented in the table below.

**Table 8.** Appendix A.4. Heavy metals Cr, Cu, Fe, Mn, Ni, Pb, Sn and Zn [mg/kg] in fractions for each sample (minimum, maximum, median, mean, S.D. of 7 measurement runs per fraction).

| Area type  | Gully pot and metals | Parameter | Heavy metals in fractions [mg/kg] |                     |                      |                       |                       |                       |                        |                         |       |
|------------|----------------------|-----------|-----------------------------------|---------------------|----------------------|-----------------------|-----------------------|-----------------------|------------------------|-------------------------|-------|
|            |                      |           | <50 $\mu\text{m}$                 | 50-75 $\mu\text{m}$ | 75-100 $\mu\text{m}$ | 100-150 $\mu\text{m}$ | 150-250 $\mu\text{m}$ | 250-500 $\mu\text{m}$ | 500-1000 $\mu\text{m}$ | 1000-2000 $\mu\text{m}$ |       |
| No traffic | 10                   | Cr        | Min                               | 148.02              | 62.31                | 45.65                 | 35.94                 | 30.73                 | 0.00                   | 0.00                    | 0.00  |
|            |                      |           | Max                               | 228.66              | 152.65               | 138.28                | 102.87                | 68.96                 | 32.66                  | 60.72                   | 52.00 |
|            |                      |           | Med                               | 165.34              | 115.62               | 121.32                | 56.79                 | 37.63                 | 0.00                   | 0.00                    | 26.72 |
|            |                      |           | Mean                              | 177.80              | 118.37               | 110.73                | 66.99                 | 44.25                 | 12.60                  | 20.01                   | 23.97 |
|            |                      |           | S.D.                              | 28.12               | 30.10                | 30.15                 | 23.99                 | 15.19                 | 15.98                  | 26.32                   | 23.68 |
|            | Cu                   | Min       | 77.70                             | 46.25               | 73.71                | 31.12                 | 21.00                 | 0.00                  | 0.00                   | 0.00                    |       |
|            |                      | Max       | 122.97                            | 117.64              | 113.87               | 76.47                 | 45.65                 | 16.95                 | 27.98                  | 131.92                  |       |
|            |                      | Med       | 90.91                             | 85.54               | 97.34                | 55.82                 | 24.86                 | 0.00                  | 0.00                   | 22.60                   |       |
|            |                      | Mean      | 95.23                             | 85.12               | 97.91                | 57.14                 | 29.07                 | 6.53                  | 6.20                   | 36.21                   |       |
|            |                      | S.D.      | 18.54                             | 22.99               | 13.70                | 14.09                 | 9.25                  | 8.19                  | 11.19                  | 43.35                   |       |
|            | Fe                   | Min       | 30757.63                          | 23952.23            | 24956.12             | 21668.48              | 20890.81              | 16138.03              | 16729.93               | 20741.44                |       |
|            |                      | Max       | 41096.80                          | 34991.46            | 31189.38             | 24608.88              | 23608.28              | 21266.66              | 33778.88               | 44465.48                |       |
|            |                      | Med       | 31976.15                          | 29527.37            | 27583.77             | 22357.58              | 21294.14              | 18028.09              | 21948.08               | 39891.79                |       |
|            |                      | Mean      | 33918.37                          | 29482.66            | 27801.20             | 22702.83              | 21704.39              | 18659.40              | 23200.31               | 36655.13                |       |
|            |                      | S.D.      | 3709.88                           | 3291.12             | 1924.49              | 1015.89               | 1055.27               | 2195.42               | 7015.66                | 8017.21                 |       |
|            | Mn                   | Min       | 340.85                            | 296.09              | 251.07               | 310.28                | 414.18                | 307.69                | 263.77                 | 408.59                  |       |
|            |                      | Max       | 498.63                            | 413.63              | 418.84               | 405.66                | 746.79                | 673.37                | 615.43                 | 752.56                  |       |
|            |                      | Med       | 386.27                            | 365.51              | 303.67               | 359.24                | 482.22                | 436.65                | 388.72                 | 624.08                  |       |
|            |                      | Mean      | 400.83                            | 364.15              | 323.94               | 356.81                | 537.51                | 449.78                | 411.68                 | 602.81                  |       |
|            |                      | S.D.      | 64.15                             | 42.74               | 59.66                | 35.07                 | 135.40                | 126.03                | 142.28                 | 126.18                  |       |
|            | Ni                   | Min       | 0.00                              | 0.00                | 0.00                 | 0.00                  | 0.00                  | 0.00                  | 0.00                   | 35.69                   |       |
|            |                      | Max       | 0.00                              | 27.60               | 37.64                | 28.09                 | 43.87                 | 52.17                 | 54.84                  | 83.82                   |       |
|            |                      | Med       | 0.00                              | 0.00                | 0.00                 | 0.00                  | 34.10                 | 38.80                 | 39.11                  | 68.86                   |       |
|            |                      | Mean      | 0.00                              | 3.94                | 5.38                 | 4.01                  | 25.98                 | 36.28                 | 36.47                  | 63.57                   |       |
|            |                      | S.D.      | 0.00                              | 10.43               | 14.23                | 10.62                 | 20.71                 | 16.81                 | 17.36                  | 16.07                   |       |
|            | Pb                   | Min       | 34.58                             | 15.48               | 30.24                | 24.24                 | 14.47                 | 13.30                 | 10.66                  | 10.91                   |       |
|            |                      | Max       | 51.30                             | 55.80               | 45.91                | 82.83                 | 23.17                 | 18.80                 | 18.42                  | 23.26                   |       |
|            |                      | Med       | 41.23                             | 37.44               | 44.32                | 39.23                 | 20.69                 | 16.19                 | 14.15                  | 14.84                   |       |
|            |                      | Mean      | 41.91                             | 37.32               | 42.35                | 42.95                 | 19.88                 | 15.97                 | 14.44                  | 15.27                   |       |
|            |                      | S.D.      | 5.62                              | 12.00               | 5.56                 | 18.61                 | 3.38                  | 2.03                  | 2.43                   | 4.18                    |       |
|            | Sn                   | Min       | 0.00                              | 14.58               | 0.00                 | 15.21                 | 33.71                 | 32.69                 | 30.30                  | 28.28                   |       |
|            |                      | Max       | 28.82                             | 33.77               | 32.88                | 49.30                 | 47.54                 | 48.80                 | 60.84                  | 57.31                   |       |
|            |                      | Med       | 19.06                             | 22.95               | 0.00                 | 40.89                 | 37.90                 | 42.27                 | 38.67                  | 45.73                   |       |
|            |                      | Mean      | 14.37                             | 23.22               | 8.80                 | 39.08                 | 39.19                 | 42.59                 | 41.01                  | 45.99                   |       |
|            |                      | S.D.      | 13.80                             | 6.93                | 15.08                | 11.23                 | 5.25                  | 5.12                  | 9.97                   | 9.14                    |       |
|            | Zn                   | Min       | 436.47                            | 254.38              | 453.78               | 308.43                | 128.35                | 69.34                 | 74.11                  | 100.41                  |       |
| Max        |                      | 583.32    | 774.26                            | 605.40              | 378.53               | 221.39                | 218.16                | 135.54                | 127.39                 |                         |       |
| Med        |                      | 446.11    | 535.57                            | 593.50              | 319.96               | 135.36                | 109.90                | 117.38                | 116.16                 |                         |       |
| Mean       |                      | 472.46    | 527.52                            | 562.31              | 338.63               | 151.09                | 133.42                | 103.17                | 114.45                 |                         |       |
| S.D.       |                      | 52.20     | 153.09                            | 55.36               | 31.87                | 35.70                 | 54.04                 | 25.21                 | 8.88                   |                         |       |
| No traffic | 11                   | Cr        | Min                               | 249.50              | 48.06                | 30.62                 | 0.00                  | 0.00                  | 0.00                   | 0.00                    | 0.00  |
|            |                      |           | Max                               | 345.05              | 67.69                | 91.00                 | 43.19                 | 35.46                 | 0.00                   | 33.07                   | 38.78 |
|            |                      |           | Med                               | 311.87              | 55.07                | 48.61                 | 35.22                 | 0.00                  | 0.00                   | 0.00                    | 0.00  |

|            |       |    |      |          |          |          |          |          |          |          |          |       |
|------------|-------|----|------|----------|----------|----------|----------|----------|----------|----------|----------|-------|
| No traffic | 12    | Cu | Mean | 306.77   | 56.41    | 49.93    | 22.17    | 8.86     | 0.00     | 4.72     | 5.54     |       |
|            |       |    | S.D. | 33.35    | 8.39     | 20.03    | 20.88    | 15.35    | 0.00     | 12.50    | 14.66    |       |
|            |       | Cu | Min  | 60.72    | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 0.00  |
|            |       |    | Max  | 87.99    | 28.76    | 22.22    | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 28.98 |
|            |       |    | Med  | 71.72    | 18.62    | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 15.11 |
|            |       |    | Mean | 75.12    | 18.56    | 5.43     | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 13.19 |
|            |       |    | S.D. | 10.45    | 9.40     | 9.46     | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 13.23 |
|            |       | Fe | Min  | 36575.23 | 21162.05 | 20452.05 | 18014.37 | 16949.92 | 16256.90 | 18742.04 | 15571.67 |       |
|            |       |    | Max  | 48747.31 | 25404.00 | 22160.66 | 21738.74 | 23160.35 | 20306.58 | 26508.40 | 31531.83 |       |
|            |       |    | Med  | 41706.27 | 24784.90 | 21039.16 | 20513.86 | 18290.10 | 17405.77 | 19994.02 | 25877.06 |       |
|            |       |    | Mean | 42962.26 | 24185.19 | 21277.07 | 20408.42 | 18925.27 | 17808.02 | 21463.54 | 24536.06 |       |
|            |       |    | S.D. | 5416.69  | 1496.78  | 804.33   | 1166.39  | 2252.91  | 1514.65  | 3104.49  | 6592.87  |       |
|            |       | Mn | Min  | 475.48   | 427.04   | 471.53   | 540.00   | 463.92   | 330.08   | 424.93   | 292.83   |       |
|            |       |    | Max  | 756.76   | 576.12   | 717.20   | 723.92   | 815.38   | 683.87   | 1636.88  | 656.47   |       |
|            |       |    | Med  | 646.80   | 521.09   | 510.77   | 667.08   | 543.89   | 465.72   | 469.54   | 369.22   |       |
|            |       |    | Mean | 632.34   | 513.81   | 543.93   | 647.66   | 589.70   | 495.96   | 626.24   | 427.17   |       |
|            |       |    | S.D. | 98.42    | 50.03    | 83.20    | 68.64    | 123.63   | 116.01   | 446.14   | 132.16   |       |
|            |       | Ni | Min  | 0.00     | 0.00     | 0.00     | 31.39    | 0.00     | 0.00     | 40.35    | 29.47    |       |
|            |       |    | Max  | 56.85    | 43.54    | 58.21    | 57.71    | 60.24    | 66.74    | 75.44    | 71.20    |       |
|            |       |    | Med  | 30.81    | 34.76    | 36.98    | 44.19    | 44.41    | 52.79    | 62.26    | 58.17    |       |
|            |       |    | Mean | 26.12    | 26.17    | 24.62    | 43.18    | 39.87    | 46.08    | 62.32    | 56.67    |       |
|            |       |    | S.D. | 25.90    | 18.54    | 24.12    | 9.16     | 19.40    | 21.72    | 11.83    | 14.11    |       |
|            |       | Pb | Min  | 26.84    | 12.46    | 9.64     | 9.62     | 9.72     | 9.50     | 8.81     | 7.37     |       |
|            |       |    | Max  | 44.72    | 18.49    | 15.75    | 16.20    | 14.75    | 13.59    | 17.10    | 14.85    |       |
|            |       |    | Med  | 37.86    | 16.35    | 13.49    | 12.95    | 12.50    | 12.91    | 13.25    | 10.66    |       |
|            |       |    | Mean | 36.31    | 15.60    | 13.13    | 13.07    | 11.82    | 12.29    | 13.10    | 10.60    |       |
|            |       |    | S.D. | 6.69     | 2.45     | 1.93     | 2.34     | 1.85     | 1.43     | 2.94     | 2.33     |       |
|            |       | Sn | Min  | 23.91    | 44.02    | 43.10    | 33.28    | 28.10    | 27.49    | 27.35    | 44.79    |       |
|            |       |    | Max  | 50.75    | 55.71    | 57.71    | 57.76    | 43.49    | 53.62    | 48.38    | 58.50    |       |
|            |       |    | Med  | 47.81    | 48.01    | 48.75    | 38.77    | 37.81    | 39.86    | 37.08    | 50.28    |       |
|            |       |    | Mean | 41.17    | 49.86    | 50.18    | 42.75    | 38.04    | 39.68    | 37.66    | 50.04    |       |
|            |       |    | S.D. | 11.65    | 5.18     | 4.74     | 8.58     | 5.85     | 9.72     | 7.38     | 5.02     |       |
|            |       | Zn | Min  | 105.86   | 39.65    | 33.74    | 32.92    | 30.84    | 27.67    | 31.14    | 31.67    |       |
|            |       |    | Max  | 148.45   | 47.84    | 44.70    | 40.86    | 37.92    | 40.99    | 53.67    | 58.71    |       |
|            |       |    | Med  | 120.98   | 42.09    | 39.68    | 37.92    | 34.02    | 34.49    | 40.96    | 46.01    |       |
|            |       |    | Mean | 128.96   | 43.45    | 39.01    | 36.70    | 33.95    | 35.06    | 41.21    | 44.11    |       |
| S.D.       | 18.39 |    | 3.21 | 3.78     | 3.04     | 2.36     | 5.08     | 8.74     | 9.45     |          |          |       |
| No traffic | 12    | Cr | Min  | 108.10   | 63.32    | 57.96    | 57.75    | 32.65    | 44.02    | 38.41    | 0.00     |       |
|            |       |    | Max  | 381.28   | 126.40   | 139.15   | 130.64   | 89.90    | 90.52    | 121.59   | 90.17    |       |
|            |       |    | Med  | 173.92   | 87.86    | 98.11    | 87.82    | 62.72    | 78.58    | 61.99    | 73.34    |       |
|            |       |    | Mean | 208.11   | 87.97    | 97.62    | 84.27    | 65.61    | 67.10    | 65.55    | 66.52    |       |
|            |       |    | S.D. | 113.17   | 19.65    | 24.66    | 25.59    | 20.71    | 19.86    | 29.86    | 30.52    |       |
|            |       | Cu | Min  | 94.67    | 64.66    | 80.21    | 60.73    | 72.47    | 70.98    | 24.73    | 38.36    |       |
|            |       |    | Max  | 122.74   | 98.05    | 142.20   | 102.65   | 297.93   | 250.52   | 881.33   | 168.63   |       |
|            |       |    | Med  | 111.65   | 83.67    | 99.94    | 91.00    | 246.39   | 106.13   | 49.35    | 115.88   |       |
|            |       |    | Mean | 111.62   | 85.39    | 102.61   | 87.18    | 191.49   | 148.76   | 170.92   | 108.22   |       |
|            |       |    | S.D. | 9.31     | 11.57    | 19.55    | 17.41    | 97.95    | 74.22    | 313.93   | 55.55    |       |
|            |       | Fe | Min  | 38380.54 | 29986.87 | 28861.48 | 28024.79 | 25940.54 | 24781.78 | 23327.14 | 22067.20 |       |
|            |       |    | Max  | 43525.68 | 37338.23 | 37398.27 | 34090.63 | 30981.81 | 29779.13 | 29730.45 | 47416.33 |       |
|            |       |    | Med  | 41186.49 | 34200.34 | 36075.44 | 30812.92 | 27037.14 | 26918.63 | 29324.15 | 29035.60 |       |
|            |       |    | Mean | 41291.40 | 33839.97 | 34236.41 | 31312.85 | 27566.11 | 27283.25 | 28288.23 | 30852.18 |       |
|            |       |    | S.D. | 1893.60  | 2214.35  | 3332.13  | 2054.57  | 1684.31  | 1798.53  | 2336.57  | 7819.49  |       |
|            |       | Mn | Min  | 460.65   | 401.10   | 380.07   | 361.62   | 266.58   | 258.62   | 217.13   | 364.18   |       |
|            |       |    | Max  | 598.14   | 529.82   | 480.23   | 432.37   | 411.33   | 511.19   | 470.81   | 760.44   |       |
|            |       |    | Med  | 552.83   | 505.52   | 404.18   | 389.08   | 327.31   | 356.29   | 386.03   | 404.25   |       |
|            |       |    | Mean | 535.72   | 486.27   | 409.62   | 394.55   | 337.70   | 365.52   | 373.46   | 473.84   |       |
|            |       |    | S.D. | 53.60    | 48.24    | 33.95    | 27.89    | 49.26    | 103.08   | 84.56    | 145.69   |       |

|         |      |          |          |          |          |          |          |          |          |         |         |
|---------|------|----------|----------|----------|----------|----------|----------|----------|----------|---------|---------|
| Traffic | 7    | Ni       | Min      | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 0.00    | 0.00    |
|         |      |          | Max      | 0.00     | 49.06    | 46.09    | 28.22    | 0.00     | 0.00     | 32.89   | 49.83   |
|         |      |          | Med      | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 0.00    | 32.80   |
|         |      |          | Mean     | 0.00     | 7.01     | 6.58     | 4.03     | 0.00     | 0.00     | 9.30    | 24.01   |
|         |      |          | S.D.     | 0.00     | 18.54    | 17.42    | 10.67    | 0.00     | 0.00     | 15.89   | 23.01   |
|         |      | Pb       | Min      | 51.17    | 44.77    | 58.31    | 43.29    | 39.14    | 31.32    | 25.41   | 18.67   |
|         |      |          | Max      | 73.48    | 64.26    | 77.18    | 82.53    | 52.75    | 109.92   | 73.81   | 25.20   |
|         |      |          | Med      | 63.23    | 60.16    | 70.31    | 51.02    | 47.58    | 38.79    | 32.40   | 23.88   |
|         |      |          | Mean     | 64.08    | 55.27    | 68.73    | 54.72    | 46.21    | 48.73    | 40.66   | 22.63   |
|         |      |          | S.D.     | 7.91     | 8.85     | 6.42     | 13.33    | 4.52     | 27.36    | 18.24   | 2.46    |
|         |      | Sn       | Min      | 28.55    | 33.14    | 28.87    | 23.64    | 21.33    | 20.31    | 0.00    | 22.13   |
|         |      |          | Max      | 47.64    | 53.95    | 50.35    | 53.17    | 38.31    | 40.75    | 49.42   | 70.24   |
|         |      |          | Med      | 43.75    | 45.20    | 33.42    | 39.21    | 26.50    | 32.50    | 38.16   | 35.68   |
|         |      |          | Mean     | 41.47    | 44.95    | 35.81    | 36.82    | 27.71    | 31.60    | 34.31   | 40.88   |
|         |      |          | S.D.     | 6.32     | 6.71     | 7.30     | 9.87     | 6.61     | 7.17     | 16.12   | 16.27   |
|         |      | Zn       | Min      | 974.78   | 635.68   | 707.49   | 644.28   | 586.72   | 557.95   | 604.92  | 259.08  |
|         |      |          | Max      | 1115.97  | 933.72   | 1103.62  | 926.61   | 1038.87  | 870.25   | 1270.64 | 5455.45 |
|         |      |          | Med      | 1061.18  | 672.88   | 1079.99  | 675.78   | 726.89   | 715.70   | 736.17  | 447.14  |
|         |      |          | Mean     | 1062.08  | 705.34   | 955.49   | 729.62   | 750.74   | 733.41   | 810.28  | 1122.95 |
|         |      |          | S.D.     | 49.41    | 107.07   | 169.97   | 107.25   | 137.38   | 100.89   | 227.22  | 1912.74 |
| Cr      | Min  | 93.96    | 69.77    | 54.55    | 50.77    | 0.00     | 0.00     | 0.00     | 47.86    |         |         |
|         | Max  | 134.25   | 94.76    | 342.35   | 69.09    | 64.95    | 55.41    | 101.48   | 76.10    |         |         |
|         | Med  | 122.55   | 82.04    | 88.51    | 59.53    | 0.00     | 32.86    | 63.34    | 62.93    |         |         |
|         | Mean | 117.79   | 81.27    | 143.99   | 59.73    | 16.24    | 30.28    | 57.04    | 62.45    |         |         |
|         | S.D. | 14.70    | 8.63     | 105.08   | 8.83     | 32.48    | 22.99    | 42.99    | 12.57    |         |         |
| Cu      | Min  | 32.93    | 23.47    | 0.00     | 0.00     | 0.00     | 0.00     | 18.52    | 0.00     |         |         |
|         | Max  | 64.21    | 58.27    | 28.42    | 30.06    | 23.27    | 30.85    | 35.41    | 74.96    |         |         |
|         | Med  | 50.13    | 41.87    | 23.61    | 9.50     | 0.00     | 0.00     | 28.11    | 17.33    |         |         |
|         | Mean | 50.97    | 41.00    | 20.13    | 12.27    | 5.82     | 7.71     | 27.54    | 27.40    |         |         |
|         | S.D. | 11.44    | 12.37    | 9.87     | 14.86    | 11.64    | 15.43    | 9.03     | 35.67    |         |         |
| Fe      | Min  | 36793.61 | 31397.55 | 31512.89 | 29306.28 | 26284.42 | 28558.60 | 34039.54 | 44504.55 |         |         |
|         | Max  | 45578.96 | 33996.55 | 33318.63 | 34304.36 | 30550.45 | 33890.23 | 52582.06 | 57577.71 |         |         |
|         | Med  | 42846.56 | 33658.98 | 32683.29 | 30585.75 | 28316.62 | 31035.04 | 41243.29 | 52871.03 |         |         |
|         | Mean | 42239.15 | 33408.07 | 32603.64 | 31195.54 | 28367.03 | 31129.73 | 42277.04 | 51956.08 |         |         |
|         | S.D. | 2659.07  | 904.35   | 674.82   | 2161.81  | 1857.25  | 2263.07  | 8551.96  | 5876.95  |         |         |
| Mn      | Min  | 658.31   | 620.64   | 616.01   | 587.94   | 552.18   | 582.53   | 582.55   | 808.86   |         |         |
|         | Max  | 826.57   | 729.72   | 802.22   | 699.89   | 613.12   | 1202.10  | 954.65   | 1085.00  |         |         |
|         | Med  | 770.63   | 663.37   | 673.16   | 683.03   | 561.26   | 714.19   | 774.76   | 939.29   |         |         |
|         | Mean | 758.83   | 667.10   | 696.94   | 663.47   | 571.95   | 803.25   | 771.68   | 943.11   |         |         |
|         | S.D. | 54.17    | 34.86    | 78.05    | 51.03    | 28.54    | 275.89   | 155.55   | 122.13   |         |         |
| Ni      | Min  | 36.37    | 39.26    | 36.18    | 44.13    | 38.11    | 33.97    | 50.46    | 56.25    |         |         |
|         | Max  | 60.47    | 66.07    | 75.88    | 63.86    | 63.47    | 62.76    | 78.86    | 75.65    |         |         |
|         | Med  | 51.27    | 53.22    | 61.51    | 54.53    | 55.57    | 55.80    | 68.34    | 68.40    |         |         |
|         | Mean | 50.28    | 51.76    | 57.16    | 54.26    | 53.18    | 52.08    | 66.50    | 67.18    |         |         |
|         | S.D. | 8.90     | 10.39    | 16.20    | 8.14     | 11.54    | 12.54    | 14.57    | 8.59     |         |         |
| Pb      | Min  | 12.02    | 12.37    | 12.20    | 13.59    | 8.78     | 12.01    | 10.94    | 10.67    |         |         |
|         | Max  | 20.25    | 21.69    | 17.21    | 16.81    | 14.75    | 18.08    | 16.23    | 16.44    |         |         |
|         | Med  | 15.19    | 15.76    | 14.15    | 15.57    | 13.64    | 13.49    | 12.43    | 11.89    |         |         |
|         | Mean | 15.51    | 16.67    | 14.30    | 15.38    | 12.70    | 14.27    | 13.01    | 12.72    |         |         |
|         | S.D. | 2.55     | 3.49     | 1.49     | 1.40     | 2.75     | 2.72     | 2.49     | 2.60     |         |         |
| Sn      | Min  | 48.04    | 39.91    | 34.85    | 43.18    | 47.10    | 45.03    | 49.30    | 54.27    |         |         |
|         | Max  | 64.77    | 56.18    | 61.13    | 59.88    | 62.04    | 59.46    | 60.66    | 75.87    |         |         |
|         | Med  | 57.30    | 50.02    | 52.86    | 53.24    | 48.55    | 52.75    | 54.19    | 59.82    |         |         |
|         | Mean | 56.01    | 49.83    | 51.06    | 52.39    | 51.56    | 52.50    | 54.58    | 62.45    |         |         |
|         | S.D. | 6.04     | 5.69     | 8.82     | 7.22     | 7.06     | 6.76     | 4.81     | 9.42     |         |         |
| Zn      | Min  | 150.28   | 125.94   | 132.35   | 104.84   | 79.74    | 92.08    | 64.63    | 77.17    |         |         |
|         | Max  | 193.80   | 172.61   | 190.78   | 163.29   | 136.77   | 147.03   | 169.10   | 93.09    |         |         |

|         |      |        |        |          |          |          |          |          |          |          |          |
|---------|------|--------|--------|----------|----------|----------|----------|----------|----------|----------|----------|
|         |      | Med    | 182.64 | 144.13   | 135.99   | 148.59   | 99.89    | 101.38   | 120.79   | 84.23    |          |
|         |      | Mean   | 177.61 | 142.07   | 145.14   | 141.33   | 104.07   | 110.47   | 118.83   | 84.68    |          |
|         |      | S.D.   | 15.76  | 16.85    | 20.95    | 27.63    | 25.40    | 25.45    | 43.73    | 7.45     |          |
| Traffic | 8    | Cr     | Min    | 109.65   | 37.43    | 33.77    | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     |
|         |      |        | Max    | 247.18   | 66.20    | 104.72   | 54.54    | 59.07    | 105.65   | 71.28    | 112.64   |
|         |      |        | Med    | 165.38   | 44.74    | 67.93    | 0.00     | 36.83    | 36.52    | 40.10    | 81.00    |
|         |      |        | Mean   | 172.04   | 49.62    | 69.61    | 22.24    | 30.55    | 31.51    | 37.51    | 67.71    |
|         |      |        | S.D.   | 56.18    | 11.63    | 25.38    | 27.78    | 22.71    | 37.91    | 21.39    | 41.66    |
|         |      | Cu     | Min    | 18.03    | 0.00     | 18.98    | 0.00     | 16.44    | 0.00     | 0.00     | 0.00     |
|         |      |        | Max    | 42.58    | 31.58    | 50.28    | 105.81   | 34.01    | 25.52    | 23.26    | 56.60    |
|         |      |        | Med    | 27.78    | 23.81    | 28.55    | 17.55    | 18.52    | 20.95    | 19.18    | 21.81    |
|         |      |        | Mean   | 30.30    | 21.45    | 30.53    | 30.15    | 21.79    | 17.08    | 14.24    | 23.04    |
|         |      |        | S.D.   | 8.19     | 10.02    | 10.16    | 35.20    | 6.85     | 8.68     | 9.91     | 16.82    |
|         |      | Fe     | Min    | 37154.69 | 28527.80 | 26574.24 | 24084.46 | 21340.60 | 22814.45 | 35010.13 | 35148.57 |
|         |      |        | Max    | 47643.94 | 34423.57 | 30818.11 | 34337.50 | 28357.02 | 30939.55 | 45022.82 | 45908.45 |
|         |      |        | Med    | 37734.04 | 33371.93 | 28789.70 | 26163.33 | 23749.69 | 28251.79 | 42033.14 | 43586.58 |
|         |      |        | Mean   | 40263.25 | 32464.05 | 28397.55 | 27997.66 | 24333.11 | 27922.68 | 40724.36 | 42268.34 |
|         |      |        | S.D.   | 3984.11  | 2029.07  | 1431.49  | 3656.14  | 2224.99  | 2675.37  | 3463.08  | 3780.76  |
|         |      | Mn     | Min    | 505.39   | 373.77   | 326.41   | 332.99   | 246.59   | 351.23   | 633.42   | 625.91   |
|         |      |        | Max    | 799.41   | 627.40   | 449.48   | 593.80   | 489.64   | 656.40   | 759.26   | 766.74   |
|         |      |        | Med    | 540.19   | 578.18   | 416.02   | 410.69   | 361.53   | 499.88   | 731.97   | 694.19   |
|         |      |        | Mean   | 584.83   | 539.64   | 408.73   | 437.22   | 378.01   | 506.01   | 722.96   | 703.05   |
|         |      |        | S.D.   | 102.93   | 88.91    | 40.44    | 92.13    | 79.70    | 92.96    | 43.25    | 45.69    |
|         |      | Ni     | Min    | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 35.85    | 0.00     |
|         |      |        | Max    | 29.88    | 35.31    | 0.00     | 41.68    | 31.06    | 0.00     | 68.50    | 76.55    |
|         |      |        | Med    | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 60.42    | 54.19    |
|         |      |        | Mean   | 4.27     | 13.86    | 0.00     | 5.95     | 4.44     | 0.00     | 55.37    | 43.57    |
|         |      |        | S.D.   | 11.29    | 17.36    | 0.00     | 15.75    | 11.74    | 0.00     | 11.93    | 30.75    |
|         |      | Pb     | Min    | 0.00     | 6.02     | 5.69     | 0.00     | 0.00     | 0.00     | 7.87     | 0.00     |
|         |      |        | Max    | 16.32    | 11.95    | 11.96    | 17.93    | 11.90    | 10.89    | 14.30    | 17.29    |
|         |      |        | Med    | 9.18     | 9.90     | 6.26     | 7.05     | 6.09     | 6.87     | 11.36    | 9.50     |
| Mean    | 9.14 |        | 9.72   | 7.59     | 7.52     | 6.37     | 7.05     | 11.51    | 9.12     |          |          |
| S.D.    | 5.04 |        | 2.23   | 2.41     | 5.30     | 3.56     | 3.52     | 1.94     | 5.25     |          |          |
| Sn      | Min  | 17.29  | 38.28  | 0.00     | 0.00     | 0.00     | 33.06    | 57.22    | 38.82    |          |          |
|         | Max  | 62.94  | 59.33  | 45.25    | 48.63    | 77.01    | 56.30    | 74.78    | 68.72    |          |          |
|         | Med  | 35.01  | 50.26  | 31.58    | 37.10    | 27.74    | 40.95    | 60.54    | 58.81    |          |          |
|         | Mean | 39.27  | 49.44  | 29.67    | 31.45    | 32.62    | 42.64    | 62.68    | 58.45    |          |          |
|         | S.D. | 16.79  | 8.86   | 14.62    | 16.89    | 25.01    | 8.97     | 6.17     | 10.30    |          |          |
| Zn      | Min  | 125.21 | 94.39  | 93.56    | 100.32   | 106.17   | 99.62    | 65.85    | 55.44    |          |          |
|         | Max  | 164.58 | 158.54 | 164.32   | 185.95   | 164.12   | 145.04   | 106.30   | 125.71   |          |          |
|         | Med  | 134.91 | 103.65 | 149.17   | 113.02   | 136.94   | 123.53   | 72.34    | 66.72    |          |          |
|         | Mean | 137.14 | 114.18 | 143.46   | 136.21   | 134.30   | 122.74   | 77.83    | 75.32    |          |          |
|         | S.D. | 13.67  | 23.09  | 23.30    | 39.25    | 17.39    | 16.04    | 14.14    | 23.16    |          |          |
| Traffic | 9    | Cr     | Min    | 135.30   | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 54.19    | 0.00     |
|         |      |        | Max    | 178.44   | 52.72    | 0.00     | 64.34    | 105.71   | 61.79    | 92.60    | 478.98   |
|         |      |        | Med    | 143.14   | 0.00     | 0.00     | 0.00     | 0.00     | 44.27    | 62.70    | 36.92    |
|         |      |        | Mean   | 148.00   | 13.82    | 0.00     | 15.06    | 25.42    | 38.69    | 66.94    | 107.50   |
|         |      |        | S.D.   | 14.54    | 23.73    | 0.00     | 26.58    | 39.29    | 19.56    | 14.22    | 167.35   |
|         |      | Cu     | Min    | 21.15    | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     |
|         |      |        | Max    | 49.17    | 19.90    | 0.00     | 14.39    | 16.97    | 16.52    | 23.23    | 36.48    |
|         |      |        | Med    | 30.46    | 12.60    | 0.00     | 0.00     | 0.00     | 0.00     | 17.27    | 18.10    |
|         |      |        | Mean   | 31.32    | 9.43     | 0.00     | 2.06     | 4.34     | 2.36     | 14.29    | 16.41    |
|         |      |        | S.D.   | 9.11     | 9.17     | 0.00     | 5.44     | 7.49     | 6.24     | 10.06    | 13.20    |
|         |      | Fe     | Min    | 47918.05 | 27056.67 | 23227.64 | 24374.60 | 23368.02 | 25540.89 | 25908.28 | 40188.79 |
|         |      |        | Max    | 55678.27 | 35811.10 | 27836.37 | 26902.86 | 26800.34 | 30014.92 | 41768.95 | 64241.83 |
|         |      |        | Med    | 48967.59 | 34193.36 | 26566.00 | 25351.36 | 25410.10 | 27472.96 | 38684.61 | 48030.58 |
|         |      |        | Mean   | 50709.11 | 32317.13 | 25723.94 | 25452.24 | 25459.45 | 27511.15 | 35794.94 | 50175.21 |

|         |       |       |          |          |          |          |          |          |          |          |
|---------|-------|-------|----------|----------|----------|----------|----------|----------|----------|----------|
|         |       | S.D.  | 2924.28  | 3215.59  | 1751.32  | 853.77   | 1272.53  | 1321.91  | 6670.23  | 8549.11  |
|         |       | Min   | 682.51   | 346.53   | 286.97   | 312.60   | 340.17   | 382.40   | 376.33   | 697.19   |
|         |       | Max   | 905.59   | 464.45   | 404.36   | 484.25   | 457.33   | 748.48   | 837.00   | 1327.46  |
|         |       | Med   | 782.64   | 446.75   | 351.54   | 390.37   | 431.24   | 519.35   | 653.72   | 888.20   |
|         |       | Mean  | 774.43   | 432.46   | 345.70   | 404.04   | 413.87   | 519.70   | 638.18   | 945.53   |
|         |       | S.D.  | 71.53    | 42.60    | 43.64    | 60.42    | 39.24    | 118.65   | 160.48   | 214.92   |
|         |       | Min   | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     |
|         |       | Max   | 53.73    | 0.00     | 0.00     | 0.00     | 0.00     | 30.63    | 74.25    | 147.65   |
|         |       | Med   | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 65.08    | 53.48    |
|         |       | Mean  | 7.68     | 0.00     | 0.00     | 0.00     | 0.00     | 8.51     | 48.48    | 64.98    |
|         |       | S.D.  | 20.31    | 0.00     | 0.00     | 0.00     | 0.00     | 14.55    | 33.42    | 48.88    |
|         |       | Min   | 9.46     | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 7.11     |
|         |       | Max   | 14.19    | 10.49    | 6.68     | 6.27     | 9.42     | 11.16    | 11.36    | 13.89    |
|         |       | Med   | 12.55    | 0.00     | 0.00     | 5.78     | 5.74     | 8.28     | 6.73     | 9.44     |
|         |       | Mean  | 12.10    | 2.79     | 0.95     | 4.15     | 4.31     | 6.25     | 7.19     | 9.92     |
|         |       | S.D.  | 1.67     | 4.79     | 2.52     | 2.85     | 4.17     | 4.48     | 3.95     | 2.19     |
|         |       | Min   | 49.34    | 0.00     | 0.00     | 0.00     | 13.04    | 21.54    | 37.73    | 45.00    |
|         |       | Max   | 78.19    | 14.24    | 0.00     | 27.51    | 44.77    | 42.90    | 63.02    | 66.97    |
|         |       | Med   | 58.49    | 0.00     | 0.00     | 14.04    | 37.08    | 38.20    | 51.33    | 57.85    |
|         |       | Mean  | 60.04    | 2.03     | 0.00     | 11.86    | 35.03    | 35.87    | 51.11    | 56.12    |
|         |       | S.D.  | 9.36     | 5.38     | 0.00     | 11.77    | 11.46    | 7.25     | 9.23     | 7.36     |
|         |       | Min   | 108.60   | 74.64    | 66.35    | 60.13    | 53.15    | 61.58    | 49.95    | 49.05    |
|         |       | Max   | 132.86   | 106.31   | 88.34    | 79.08    | 85.99    | 101.09   | 82.84    | 120.40   |
|         |       | Med   | 121.81   | 100.26   | 81.22    | 70.61    | 66.78    | 86.51    | 75.00    | 63.24    |
|         |       | Mean  | 120.50   | 93.76    | 77.92    | 70.71    | 68.23    | 82.80    | 67.97    | 69.14    |
|         |       | S.D.  | 7.97     | 12.48    | 8.70     | 6.67     | 12.79    | 15.80    | 13.81    | 24.36    |
| Traffic | 13    | Min   | 139.50   | 141.46   | 86.67    | 69.86    | 0.00     | 0.00     | 0.00     | 0.00     |
|         |       | Max   | 214.43   | 193.70   | 125.28   | 100.61   | 51.80    | 0.00     | 44.35    | 74.67    |
|         |       | Med   | 173.88   | 157.93   | 102.06   | 89.61    | 32.69    | 0.00     | 0.00     | 59.17    |
|         |       | Mean  | 174.64   | 164.00   | 103.16   | 87.66    | 27.82    | 0.00     | 16.81    | 50.83    |
|         |       | S.D.  | 24.47    | 20.11    | 13.21    | 10.81    | 20.41    | 0.00     | 21.14    | 26.83    |
|         |       | Min   | 35.64    | 35.83    | 30.72    | 17.67    | 0.00     | 0.00     | 0.00     | 0.00     |
|         |       | Max   | 58.01    | 67.66    | 40.68    | 42.49    | 24.18    | 0.00     | 20.00    | 64.76    |
|         |       | Med   | 47.39    | 47.53    | 33.60    | 27.80    | 12.69    | 0.00     | 0.00     | 21.66    |
|         |       | Mean  | 47.08    | 51.75    | 34.26    | 29.82    | 11.46    | 0.00     | 4.94     | 21.25    |
|         |       | S.D.  | 7.20     | 11.31    | 3.39     | 10.12    | 11.35    | 0.00     | 8.57     | 24.58    |
|         |       | Min   | 31944.36 | 30069.73 | 23363.36 | 24730.90 | 18665.62 | 11839.47 | 15101.10 | 18447.87 |
|         |       | Max   | 37391.04 | 31508.22 | 28694.50 | 27717.25 | 22480.78 | 17897.61 | 18971.01 | 40940.03 |
|         |       | Med   | 36574.96 | 31020.57 | 26641.03 | 27279.62 | 20337.54 | 13564.57 | 15685.06 | 31269.50 |
|         |       | Mean  | 35707.23 | 30827.46 | 26865.11 | 26767.87 | 20350.28 | 14760.08 | 16521.35 | 32218.56 |
|         |       | S.D.  | 1989.23  | 482.26   | 1888.01  | 1168.69  | 1123.78  | 2369.48  | 1678.08  | 8224.30  |
|         |       | Min   | 351.55   | 323.07   | 237.46   | 310.14   | 269.71   | 234.14   | 229.69   | 344.40   |
|         |       | Max   | 522.03   | 394.46   | 369.04   | 428.60   | 399.69   | 523.29   | 307.63   | 773.17   |
|         |       | Med   | 492.02   | 344.86   | 320.30   | 358.83   | 310.89   | 253.70   | 266.78   | 522.13   |
|         |       | Mean  | 475.95   | 355.21   | 318.42   | 362.75   | 309.76   | 300.06   | 269.36   | 570.72   |
|         |       | S.D.  | 58.63    | 27.37    | 45.12    | 45.35    | 43.49    | 103.43   | 23.20    | 162.24   |
|         |       | Min   | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     |
|         |       | Max   | 40.72    | 37.45    | 36.10    | 27.97    | 0.00     | 25.81    | 40.68    | 63.98    |
|         |       | Med   | 30.89    | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 0.00     | 45.80    |
|         |       | Mean  | 24.73    | 9.66     | 9.07     | 7.82     | 0.00     | 3.69     | 10.70    | 40.02    |
|         |       | S.D.  | 17.46    | 16.63    | 15.70    | 13.36    | 0.00     | 9.76     | 18.37    | 22.18    |
|         |       | Min   | 17.44    | 19.17    | 19.33    | 21.10    | 18.31    | 15.12    | 10.73    | 7.70     |
|         |       | Max   | 32.68    | 25.19    | 25.11    | 43.36    | 26.87    | 21.56    | 19.01    | 21.60    |
|         |       | Med   | 30.50    | 24.25    | 21.84    | 29.94    | 22.99    | 18.70    | 15.67    | 10.10    |
| Mean    | 27.64 | 23.37 | 21.75    | 29.93    | 22.98    | 18.31    | 14.92    | 11.75    |          |          |
| S.D.    | 5.75  | 2.34  | 2.12     | 8.91     | 2.78     | 2.62     | 2.99     | 4.65     |          |          |

|         |      |          |          |          |          |          |          |          |          |        |        |
|---------|------|----------|----------|----------|----------|----------|----------|----------|----------|--------|--------|
| Traffic | 14   | Sn       | Min      | 40.47    | 14.81    | 21.64    | 27.65    | 29.18    | 30.03    | 32.31  | 32.55  |
|         |      |          | Max      | 56.13    | 43.77    | 43.88    | 46.18    | 48.77    | 38.59    | 61.27  | 73.15  |
|         |      |          | Med      | 51.77    | 34.03    | 38.53    | 34.99    | 38.93    | 33.87    | 46.51  | 61.42  |
|         |      |          | Mean     | 49.38    | 30.36    | 34.44    | 35.61    | 37.84    | 34.24    | 44.13  | 55.31  |
|         |      |          | S.D.     | 6.26     | 11.80    | 9.32     | 6.96     | 6.99     | 2.95     | 9.57   | 16.13  |
|         |      | Zn       | Min      | 257.40   | 307.78   | 249.95   | 265.44   | 152.45   | 80.83    | 86.51  | 80.07  |
|         |      |          | Max      | 298.03   | 371.62   | 296.34   | 294.54   | 199.56   | 134.17   | 129.57 | 188.22 |
|         |      |          | Med      | 289.03   | 345.41   | 278.23   | 283.94   | 177.52   | 109.77   | 119.13 | 127.90 |
|         |      |          | Mean     | 284.35   | 344.43   | 276.35   | 282.54   | 176.83   | 109.53   | 108.79 | 133.15 |
|         |      |          | S.D.     | 14.44    | 20.45    | 19.88    | 8.78     | 15.28    | 17.27    | 19.35  | 36.46  |
|         | Cr   | Min      | 197.28   | 125.64   | 96.10    | 100.88   | 75.45    | 79.69    | 72.43    | 0.00   |        |
|         |      | Max      | 244.80   | 176.50   | 134.86   | 136.08   | 116.62   | 126.93   | 192.19   | 154.74 |        |
|         |      | Med      | 212.30   | 146.13   | 106.05   | 109.57   | 88.61    | 113.43   | 82.05    | 111.34 |        |
|         |      | Mean     | 216.87   | 148.96   | 111.68   | 114.39   | 90.12    | 109.48   | 99.41    | 101.08 |        |
|         |      | S.D.     | 17.97    | 15.78    | 14.66    | 14.45    | 13.26    | 16.94    | 42.11    | 53.12  |        |
|         | Cu   | Min      | 35.84    | 24.38    | 16.12    | 0.00     | 0.00     | 0.00     | 0.00     | 0.00   |        |
|         |      | Max      | 82.94    | 64.95    | 51.73    | 24.94    | 24.96    | 21.11    | 28.14    | 205.12 |        |
|         |      | Med      | 63.91    | 42.19    | 26.35    | 19.78    | 19.72    | 0.00     | 0.00     | 0.00   |        |
|         |      | Mean     | 61.66    | 44.01    | 29.40    | 18.26    | 15.63    | 3.02     | 6.55     | 38.96  |        |
| S.D.    |      | 15.01    | 16.85    | 12.45    | 8.42     | 10.86    | 7.98     | 11.59    | 75.40    |        |        |
| Fe      | Min  | 44717.25 | 51992.73 | 43153.46 | 46293.97 | 48112.47 | 47590.11 | 46404.48 | 48749.88 |        |        |
|         | Max  | 67100.40 | 58260.72 | 50798.38 | 55710.40 | 55537.76 | 60060.78 | 63840.78 | 97604.13 |        |        |
|         | Med  | 65568.69 | 55307.99 | 47832.32 | 49802.39 | 51561.20 | 49498.20 | 51317.27 | 74911.39 |        |        |
|         | Mean | 60310.70 | 55498.68 | 47618.38 | 49946.34 | 50954.87 | 53166.40 | 52927.46 | 76570.85 |        |        |
|         | S.D. | 8361.30  | 2453.12  | 2301.09  | 2867.73  | 2871.95  | 6068.04  | 7190.06  | 19193.95 |        |        |
| Mn      | Min  | 684.22   | 927.55   | 683.28   | 851.33   | 889.41   | 803.55   | 936.87   | 856.09   |        |        |
|         | Max  | 1239.23  | 1065.77  | 935.09   | 987.45   | 1318.68  | 1129.79  | 1195.73  | 2287.38  |        |        |
|         | Med  | 1142.71  | 980.10   | 860.06   | 906.14   | 975.47   | 985.37   | 980.46   | 1323.76  |        |        |
|         | Mean | 1063.28  | 993.64   | 842.50   | 918.27   | 1045.07  | 988.47   | 1014.10  | 1559.37  |        |        |
|         | S.D. | 193.97   | 44.78    | 77.75    | 50.64    | 151.23   | 121.50   | 90.69    | 578.48   |        |        |
| Ni      | Min  | 0.00     | 70.38    | 57.99    | 58.70    | 56.58    | 68.14    | 69.62    | 79.52    |        |        |
|         | Max  | 93.01    | 95.37    | 78.97    | 86.55    | 89.14    | 120.68   | 112.27   | 166.94   |        |        |
|         | Med  | 78.92    | 80.43    | 60.93    | 79.95    | 79.86    | 93.97    | 75.30    | 126.35   |        |        |
|         | Mean | 63.65    | 80.53    | 64.65    | 75.76    | 77.44    | 95.36    | 86.94    | 125.30   |        |        |
|         | S.D. | 32.86    | 8.58     | 7.65     | 11.55    | 10.98    | 20.70    | 18.17    | 30.66    |        |        |
| Pb      | Min  | 9.43     | 11.82    | 10.18    | 9.22     | 9.53     | 10.72    | 8.75     | 6.16     |        |        |
|         | Max  | 21.38    | 16.67    | 15.75    | 13.69    | 18.89    | 14.05    | 14.52    | 16.33    |        |        |
|         | Med  | 17.21    | 13.07    | 12.35    | 11.95    | 15.13    | 13.15    | 10.00    | 11.15    |        |        |
|         | Mean | 16.84    | 14.15    | 12.60    | 11.91    | 14.21    | 12.64    | 10.95    | 11.67    |        |        |
|         | S.D. | 4.80     | 1.98     | 1.75     | 1.57     | 3.10     | 1.31     | 1.94     | 3.58     |        |        |
| Sn      | Min  | 70.10    | 50.85    | 60.75    | 50.28    | 47.39    | 41.63    | 39.19    | 45.64    |        |        |
|         | Max  | 92.26    | 81.95    | 79.07    | 70.38    | 70.95    | 70.38    | 57.86    | 65.43    |        |        |
|         | Med  | 76.73    | 67.19    | 74.76    | 63.29    | 56.41    | 50.76    | 50.77    | 60.93    |        |        |
|         | Mean | 78.53    | 69.14    | 71.62    | 62.72    | 58.24    | 52.19    | 47.64    | 58.11    |        |        |
|         | S.D. | 7.55     | 11.61    | 6.67     | 6.24     | 8.82     | 9.02     | 7.48     | 8.08     |        |        |
| Zn      | Min  | 95.71    | 72.67    | 76.63    | 62.18    | 75.60    | 47.67    | 49.15    | 44.99    |        |        |
|         | Max  | 146.67   | 145.79   | 109.94   | 133.10   | 96.97    | 64.91    | 58.04    | 76.50    |        |        |
|         | Med  | 136.96   | 107.96   | 88.41    | 81.82    | 83.77    | 57.26    | 53.95    | 71.06    |        |        |
|         | Mean | 130.26   | 112.86   | 88.36    | 85.81    | 84.74    | 56.10    | 54.15    | 62.31    |        |        |
|         | S.D. | 17.74    | 30.25    | 11.73    | 25.47    | 7.53     | 5.95     | 2.92     | 13.33    |        |        |

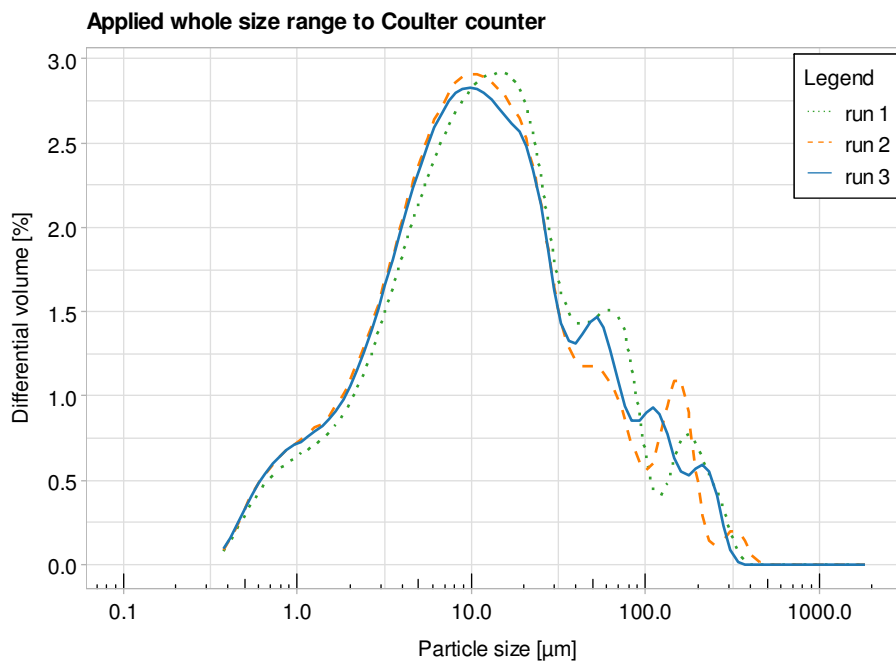


## Appendix B – Test procedure results

The following Appendices show the results of test procedures which were undertaken prior the actual analyses in order to find the most suitable analyzing method for RSGP sediment samples. The method described in chapter 2 was chosen concerning results of these tests.

### B.1. Applying the whole size range <2000 $\mu\text{m}$ in Coulter Counter

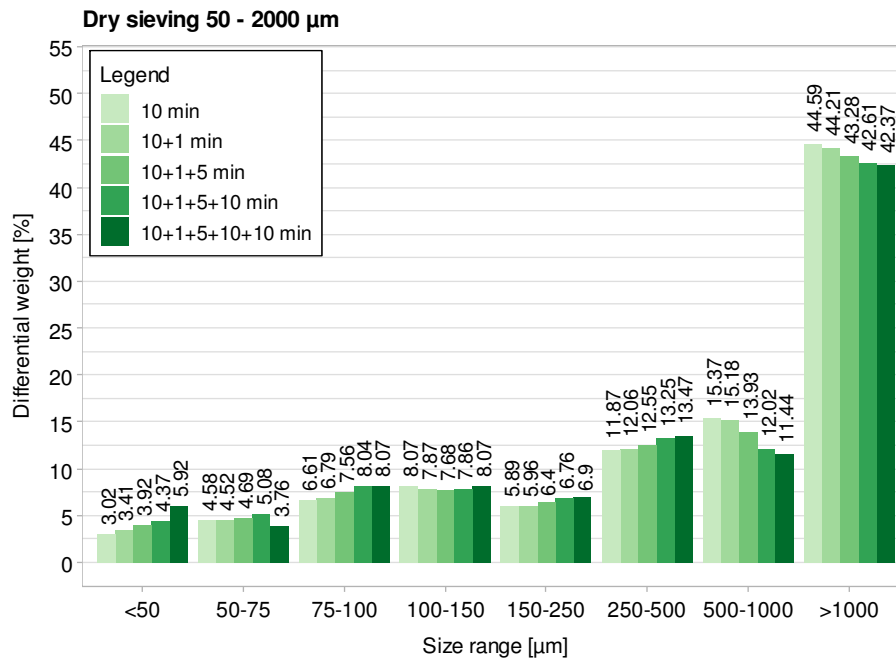
The following diagram shows the PSD from repeated runs of the same sample which was extracted by a spoon, diluted with distilled water and gently reversed before adding to the Coulter counter using a pipette with a 2000  $\mu\text{m}$  wide opening. It shows that the particles between 400  $\mu\text{m}$  and 2000  $\mu\text{m}$  probably settled faster and were not included in the measurement. In addition, it indicates an instable sample. Therefore, it is necessary to minimize the sample range before using a Coulter Counter.



**Figure 14.** Appendix B.1. Differential particle size distribution [%] using a Coulter counter for the whole size range <2000  $\mu\text{m}$ : 3 repeated runs of the same sample.

## B.2. Drying and dry sieving

Dry sieving resulted in no stable weights of the sieves after different times up to 36 min, different applied amplitudes and initial weights. One of the dry sieving test results is presented in the diagram below.



**Figure 15.** Appendix B.2. Differential fractions [%] from dry sieving <2000 µm after 10, 11, 16, 26 and 36 minutes for the fractions <50, 50-75, 75-100, 100-150, 150-250, 250-500, 500-1000 and >1000 µm.

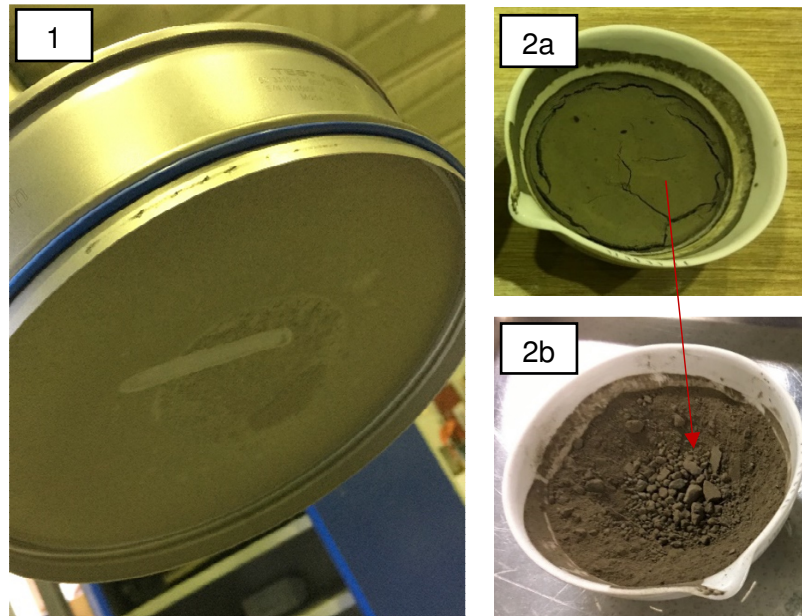
Table 9 shows the unstable fractions in [g]:

**Table 9.** Appendix B.2. Differential fractions [g] from dry sieving <2000 µm after 10, 11, 16, 26 and 36 minutes for the fractions <50, 50-75, 75-100, 100-150, 150-250, 250-500, 500-1000 and >1000 µm.

| Particle size range [µm] | Content [g] 10 min | Content [g] 10+1 min | Content [g] 10+1+5 min | Content [g] 10+1+5+10 min | Content [g] 10+1+5+10+10 min |
|--------------------------|--------------------|----------------------|------------------------|---------------------------|------------------------------|
| <50                      | 10.10              | 11.40                | 13.10                  | 14.60                     | 19.80                        |
| 50-75                    | 15.30              | 15.10                | 15.70                  | 17.00                     | 12.60                        |
| 75-100                   | 22.10              | 22.70                | 25.30                  | 26.90                     | 27.00                        |
| 100-150                  | 27.00              | 26.30                | 25.70                  | 26.30                     | 27.00                        |
| 150-250                  | 19.70              | 19.90                | 21.40                  | 22.60                     | 23.10                        |
| 250-500                  | 39.70              | 40.30                | 42.00                  | 44.30                     | 45.10                        |
| 500-1000                 | 51.40              | 50.70                | 46.60                  | 40.20                     | 38.30                        |
| 1000-2000                | 149.10             | 147.70               | 144.80                 | 142.50                    | 141.80                       |

The issue is assumed to be electrostatic loaded small particles which were clogging the bottom side of the sieves, beginning on the sieve with mesh size 250 µm downwards (Figure 16, Photo 1) and particles sticking together after drying process (Photo 2a) which could not be broken by hand properly without destroying particles prior to PSD analysis (Photo 2b). Even

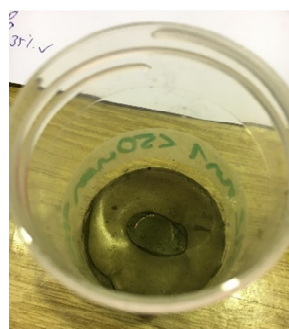
after glowing at 550 °C, the electrostatic loads under the sieves were still available. As a conclusion, dry sieving is not suitable for this kind of sample.



**Figure 16.** Appendix B.2. 1) Photo showing a clogged sieve on bottom side during dry sieving; 2a) Photo showing a sample after drying at 105 °C with a drying layer; 2b) Photo showing a sample after trying to break the drying layer (without breaking particles in its shape) with the result of still existing drying aggregates.

### B.3. Resuspension of dried particles

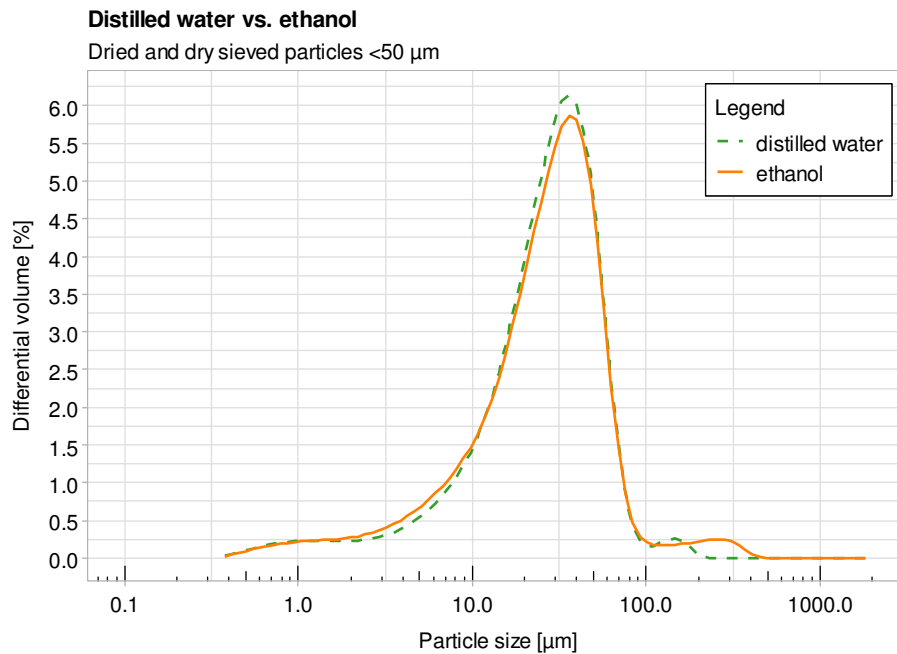
After drying and dry sieving procedure there was a hydrophobic reaction along with agglomerations in the small fraction <50 µm when trying to get the sample into water phase again (Figure 17). Therefore, there were tested different opportunities to solve this problem and test runs were performed by using the Coulter counter to see the effects.



**Figure 17.** Appendix B.3. Photo showing a hydrophobic reaction of dried and dry sieved particles <50 µm after adding distilled water.

### B.3.1. Distilled water vs. ethanol

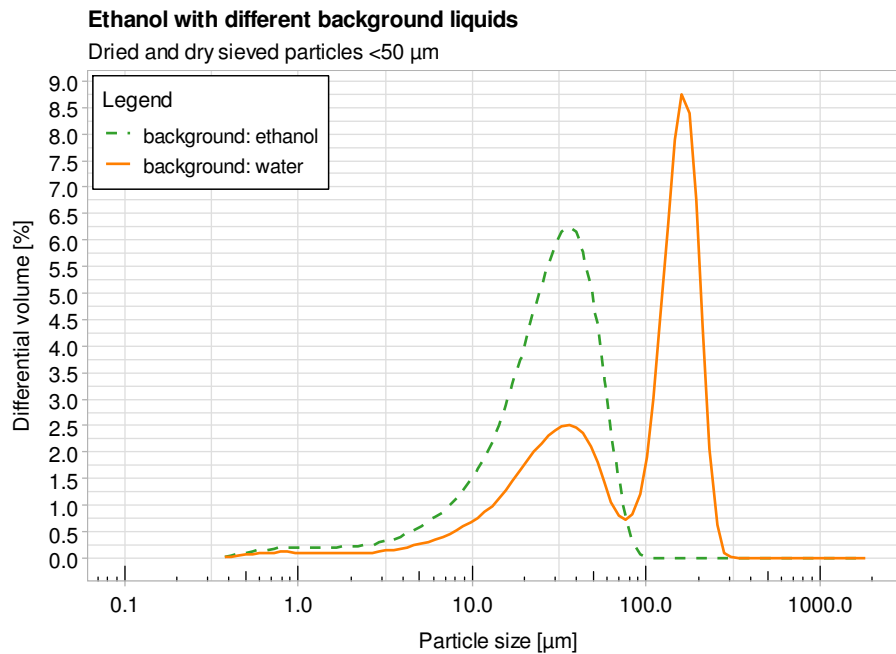
The following diagram shows the Coulter counter runs with dried and dry sieved particles <50  $\mu\text{m}$  with added distilled water and 70 % ethanol, respectively. There was no significant difference between these options. The peak remains in the range between 35 and 40  $\mu\text{m}$  (Figure 18). Hence, for further tests distilled water was used.



**Figure 18.** Appendix B.3.1. Differential particle size distribution [%] using a Coulter counter for dried and dry sieved particles <50  $\mu\text{m}$  + a) distilled water (green dashed line) and b) 70 % ethanol (orange solid line).

### B.3.2. Background liquid: Ethanol with water vs. merely ethanol

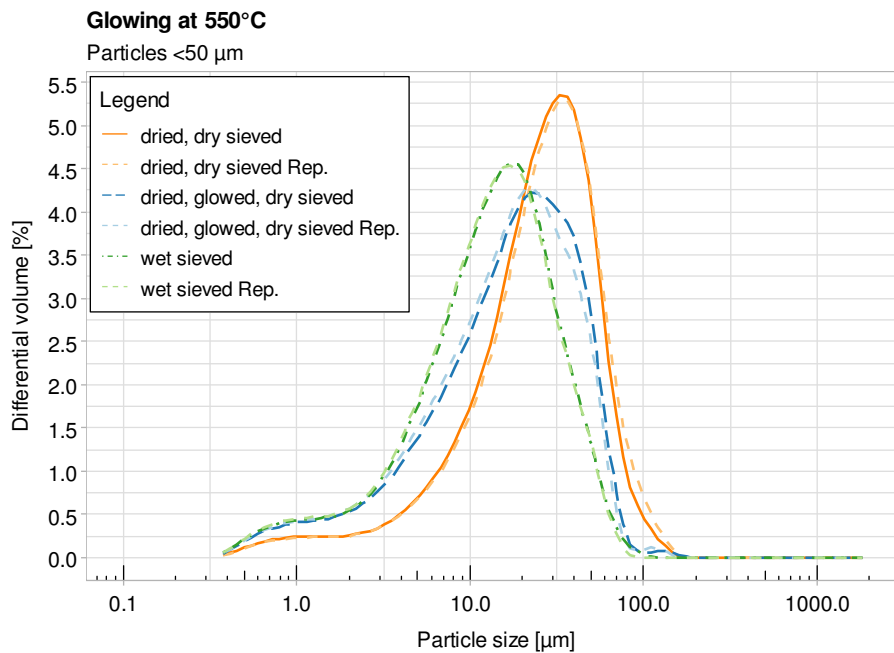
The following diagram shows the dried and dry sieved fraction <50  $\mu\text{m}$  suspended in ethanol. The green dashed curve represents run with ethanol as a background liquid. The orange solid curve is the result from the run with water mixed with ethanol as a background liquid. It demonstrates that there was a large change in PSD when changing concentration of a dispersant.



**Figure 19.** Appendix B.3.2. Differential particle size distribution [%] using a Coulter counter for dried and dry sieved particles <50  $\mu\text{m}$  diluted in 70 % ethanol with a) 70 % ethanol (green dashed line) and b) distilled water (orange solid line) as a background liquid.

### B.3.3. Glowing at 550 °C

Figure 20 shows the curves for: a) original wet sieved <50  $\mu\text{m}$  particles; b) dried and dry sieved <50  $\mu\text{m}$  particles + distilled water and c) dried, glowed and dry sieved <50  $\mu\text{m}$  particles + distilled water. Each run was repeated to demonstrate the stability of the sample. It displays the large effect of drying particles due to the shifted PSD towards big particle sizes (peak around 35  $\mu\text{m}$ ). In contrast wet sieved PSD has a peak at approximately 20  $\mu\text{m}$ . Even after glowing and dry sieving, the PSD is still shifted (peak approximately at 25  $\mu\text{m}$ ).

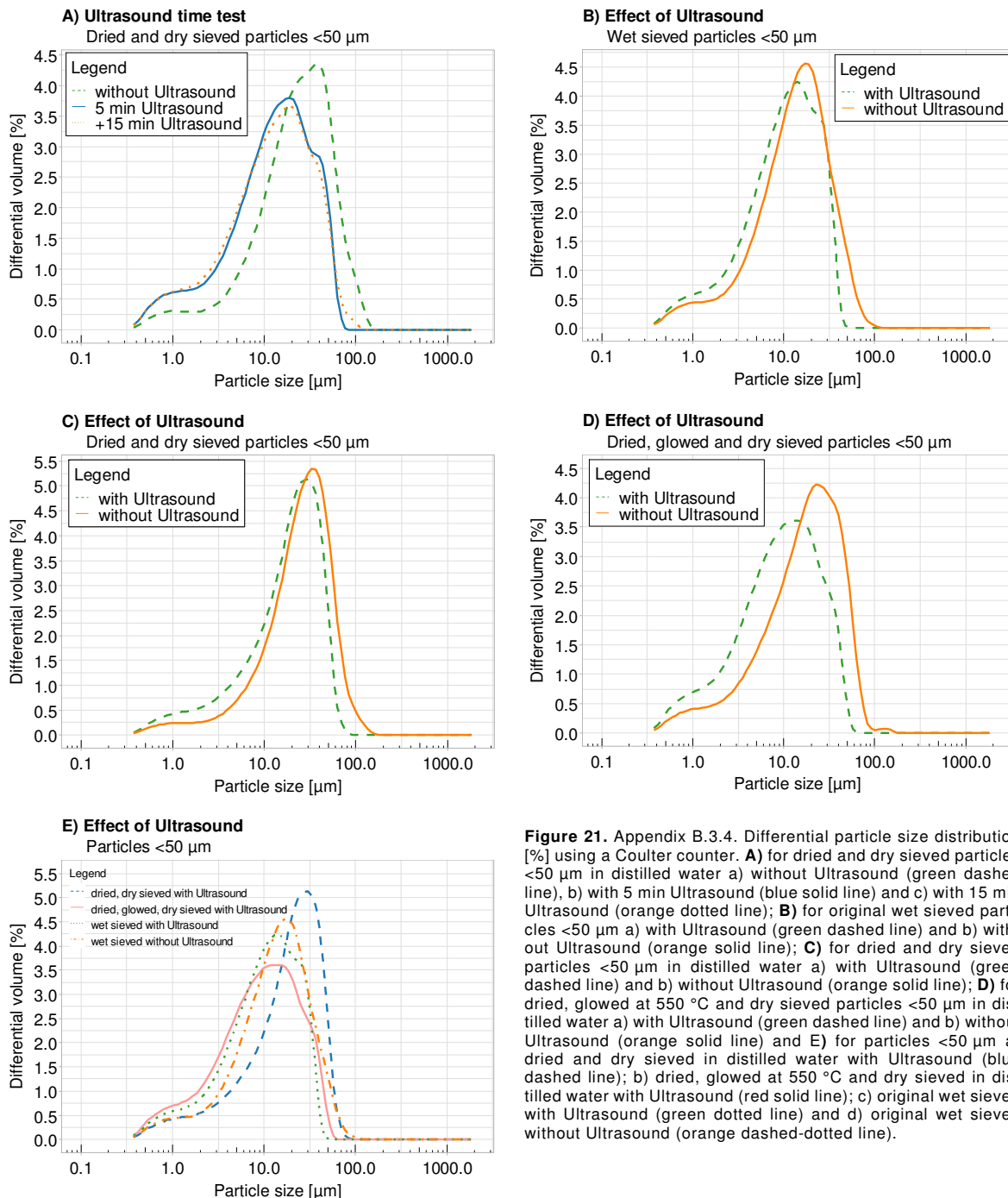


**Figure 20.** Appendix B.3.3. Differential particle size distribution [%] using a Coulter counter for particles <50  $\mu\text{m}$ : a) dried and dry sieved (orange solid line); b) dried, glowed at 550 °C and dry sieved (blue long-dashed line) and c) original wet sieved (green dot-dashed line); each with repeated runs (dashed lines).

### B.3.4. Ultrasound

Finally, an ultrasound as a mechanical dispersion was used trying to separate the particles after drying or glowing process. At first, an appropriate time of using the ultrasound was identified. It was tested 5 min and 15 min in addition. Figure 21, A) shows that 5 min was enough. This time was applied for the following tests.

The diagrams B), C) and D) show the effect of the ultrasound on the original wet sieved fraction <50  $\mu\text{m}$ , on a dried and dry sieved fraction <50  $\mu\text{m}$  suspended in distilled water and on a dried, glowing and dry sieved fraction <50  $\mu\text{m}$ .



**Figure 21.** Appendix B.3.4. Differential particle size distribution [%] using a Coulter counter. **A)** for dried and dry sieved particles <50  $\mu\text{m}$  in distilled water a) without Ultrasound (green dashed line), b) with 5 min Ultrasound (blue solid line) and c) with 15 min Ultrasound (orange dotted line); **B)** for original wet sieved particles <50  $\mu\text{m}$  a) with Ultrasound (green dashed line) and b) without Ultrasound (orange solid line); **C)** for dried and dry sieved particles <50  $\mu\text{m}$  in distilled water a) with Ultrasound (green dashed line) and b) without Ultrasound (orange solid line); **D)** for dried, glowing at 550  $^{\circ}\text{C}$  and dry sieved particles <50  $\mu\text{m}$  in distilled water a) with Ultrasound (green dashed line) and b) without Ultrasound (orange solid line) and **E)** for particles <50  $\mu\text{m}$  a) dried and dry sieved in distilled water with Ultrasound (blue dashed line); b) dried, glowing at 550  $^{\circ}\text{C}$  and dry sieved in distilled water with Ultrasound (red solid line); c) original wet sieved with Ultrasound (green dotted line) and d) original wet sieved without Ultrasound (orange dashed-dotted line).

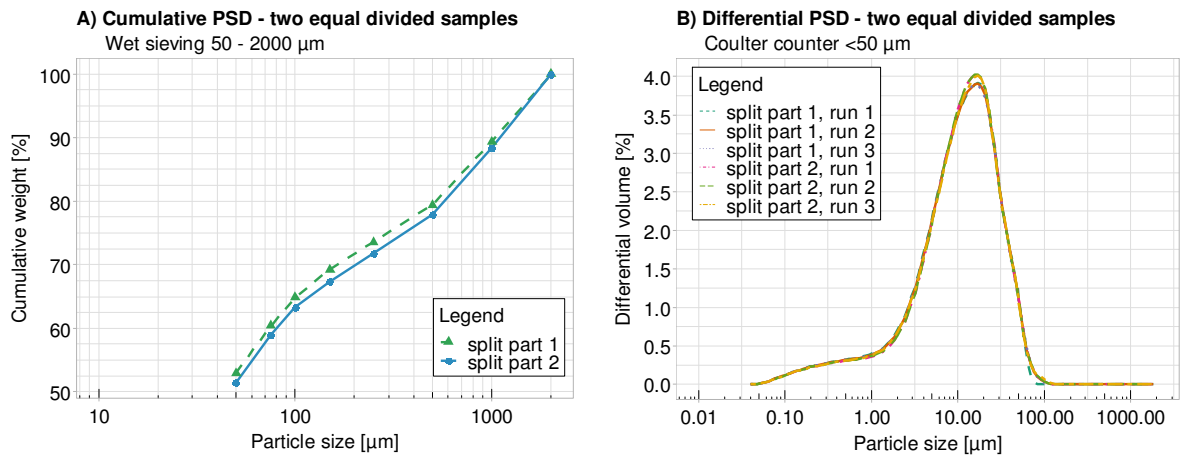
In summary, Figure 21, E) shows the PSD of particles  $<50\ \mu\text{m}$  for a) dried and dry sieved in distilled water with Ultrasound (blue dashed line); b) dried, glowd at  $550\ ^\circ\text{C}$  and dry sieved in distilled water with Ultrasound (red solid line); c) original wet sieved with Ultrasound (green dotted line) and d) original wet sieved without Ultrasound (orange dashed-dotted line). It means that even in nature, agglomerations are present. But it is questionable whether the ultrasound breaks down the particles to their original structure or whether it destroys the particles. An indicator for latter case would be the particle size distribution of the green dotted curve, which had almost no particles in the size of  $50\ \mu\text{m}$ . However, it is assumed to have larger particles than  $50\ \mu\text{m}$ , even with sieving with the mesh size  $50\ \mu\text{m}$  due to the non-uniform natural shape of particles. The dried and dry sieved fraction  $<50\ \mu\text{m}$  was affected by the drying procedure in a significant way, so that an ultrasound had no chance to get the particles in their original structure. This was a crucial criterion for exclusion of drying the sample prior the PSD. The glowd and dry sieved sample  $<50\ \mu\text{m}$  with ultrasound came close to the original sample with ultrasound. But anyway, due to the fact that glowing changes the nature of particles due to organic loss, using glowing as a dispersant was also excluded. These tests lead to the decision using wet sieving on not dried, original samples. Due to the knowledge that there could be some agglomerations even in not dried samples and the uncertainty of the actual impact using ultrasound, an alternative: Sodium pyrophosphate decahydrate was used as a dispersant for the valid experiments. It was no magnetic stirrer used because of the detected magnetic properties in the samples.

#### **B.4. Repeatability and representativeness of splitting and homogenizing**

Figure 22, A) and Table 10 show the results of wet sieving procedure with to equal gained samples by a riffle splitter without using a dispersant. It can be concluded that homogenizing by hand and splitting procedure using a riffle splitter was representative and repeatable.

The results of applying the smallest fraction  $<50\ \mu\text{m}$  of both equally split subsamples to the Coulter counter and repeating three times is shown in Figure 22, B). This confirms the representativeness and repeatability of the splitting procedure of the total sample and the extraction of a subsample  $<50\ \mu\text{m}$  by using a pipette.





**Figure 22.** Appendix B.4. Two equally divided parts of a sample by using a riffle splitter without a dispersant. **A)** Cumulative particle size distribution [%] for wet sieving for particles between 50 and 2000 µm; **B)** Differential particle size distribution [%] using a Coulter counter for particles <50 µm, each with two repeated runs.

**Table 10.** Appendix B.4. Differential particle size distribution fractions [%] for wet sieving for particles between 50 and 2000 µm without dispersant of two equal divided parts by using a riffle splitter.

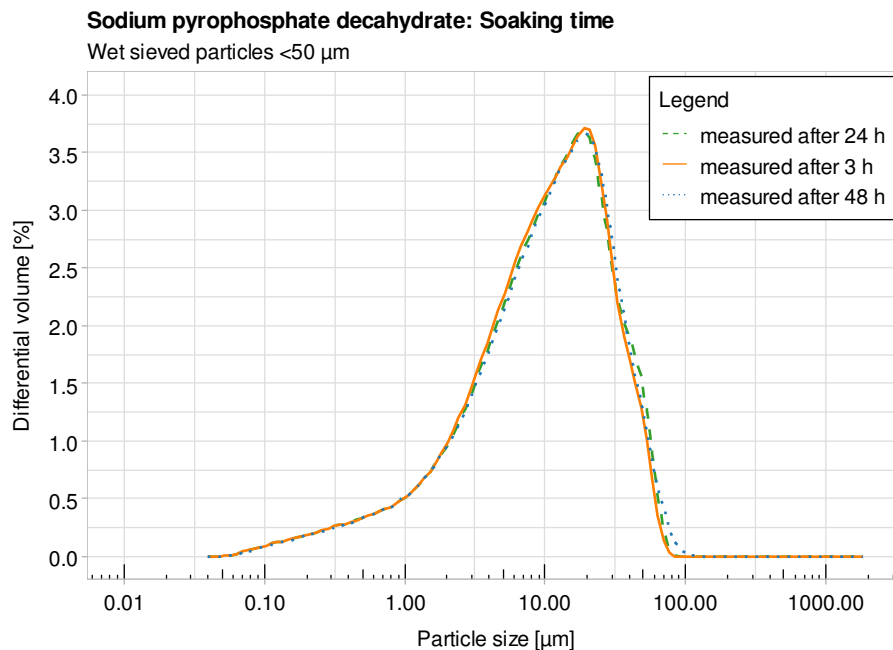
|              | PSD fractions [%] |          |           |            |            |            |             |              |
|--------------|-------------------|----------|-----------|------------|------------|------------|-------------|--------------|
|              | <50 µm            | 50-75 µm | 75-100 µm | 100-150 µm | 150-250 µm | 250-500 µm | 500-1000 µm | 1000-2000 µm |
| Split part 1 | 52.99             | 7.42     | 4.43      | 4.43       | 4.30       | 5.86       | 9.90        | 10.68        |
| Split part 2 | 51.42             | 7.55     | 4.27      | 4.13       | 4.42       | 6.13       | 10.40       | 11.68        |

## B.5. Effect of Sodium pyrophosphate decahydrate as a dispersant

This chapter contains the results of verifying procedures with the applied dispersant.

### B.5.1. Soaking time

Tests of soaking time has been performed on a wet sieved fraction <50  $\mu\text{m}$  dispersed with 3 g/l SPD. After adding the dispersant to the sample, the PSD was measured with the Coulter counter after 3 h, 24 h and 48 h. This test has been performed on 3 samples. For all samples a soaking time of 3 h was adequate. One sample is displayed below.

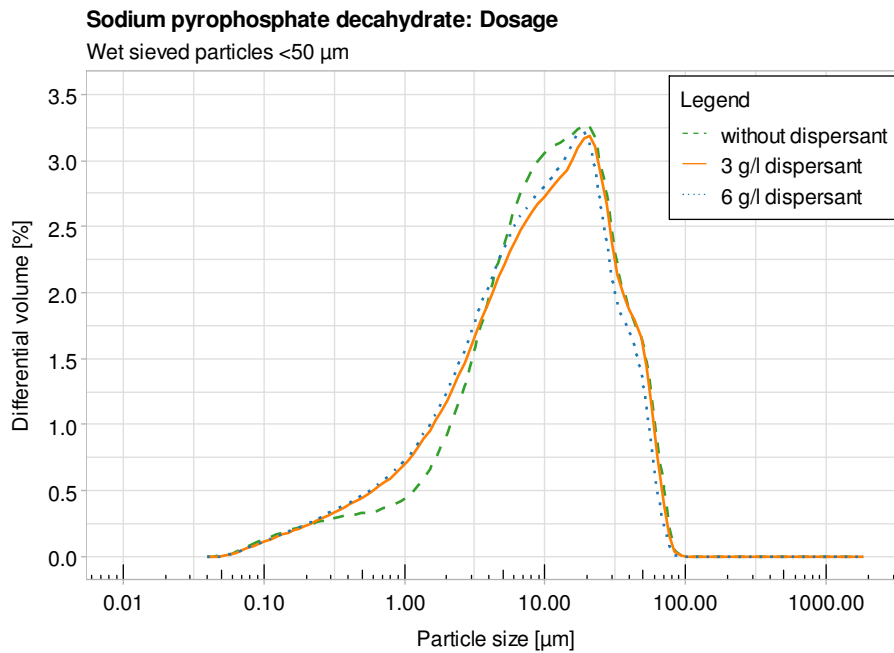


**Figure 23.** Appendix B.5.1. Differential particle size distribution [%] using a Coulter counter for wet sieved particles <50  $\mu\text{m}$  dispersed with 3 g/l of SPD a) measured after 3 h (orange solid line); b) measured after 24 h (green dashed line) and c) measured after 48 h (blue dotted line).

### B.5.2. Adequate dosage

The dosage of 3 g/l SPD adapted from Konert and Vandenberghe (1997) and DIN EN ISO 17892-4 (2017) was tested on its efficiency by applying a higher dosage of 6 g/l in the Coulter counter. In addition, zeta-potential was measured of the different dosage samples to evaluate the dispersion stability of the particles. A high zeta potential (mV), either positive or negative means stable particles, whereas zeta potential values near zero means a nonstable sample building agglomerations (Beckman Coulter, 2011 a). The test was performed with two samples. Unfortunately, just one sample could be completed due to technical issues of the device.

Following diagram shows the PSD of an original wet sieved sample a) without dispersant (green dashed line), b) with 3 g/l dispersant (orange solid line) and c) with 6 g/l dispersant (blue dotted line). Between 3 g/l and 6 g/l was a neglectable difference.



**Figure 24.** Appendix B.5.2. Differential particle size distribution [%] using a Coulter counter for wet sieved particles <50  $\mu\text{m}$  a) without dispersant (green dashed line); b) with 3 g/l dispersant (orange solid line) and c) with 6 g/l dispersant (blue dotted line).

The table below presents the measured zeta potentials [mV] for these samples.

**Table 11.** Appendix B.5.2. Measured zeta potential [mV] of Sample 6 a) without dispersant; b) with 3 g/l dispersant and c) with 6 g/l dispersant, measured 3 times and calculated the average values.

| Sample 6      | Zeta potential [mV] |                  |                  |
|---------------|---------------------|------------------|------------------|
|               | without dispersant  | 3 g/l dispersant | 6 g/l dispersant |
| Measurement 1 | -21.42              | -37.14           | -40.33           |
| Measurement 2 | -19.69              | -40.97           | -37.65           |
| Measurement 3 | -23.23              | -                | -                |
| Average       | -21.45              | -39.06           | -38.99           |

There was an increase, almost the double, in zeta potential between the undispersed sample and the sample with 3 g/l dispersant. It shows that there was a rising stability of the dispersion. But there is no difference between the two dosage 3 g/l and 6 g/l. That means that a dosage of 3 g/l is adequate to get the most potential stability of the sample. The difference in the stability of a sample was also confirmed using a microscope and monitor a dispersed and an undispersed sample. The sample with dispersant began to build agglomerations in the first approximately 30 seconds, whereas the dispersed sample with 3 g/l SPD was calmer and no movement could be observed.

