Evaluation of sensor techniques for detection of contaminants in greywater from showers

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Abstract

This thesis has been made in collaboration with the company Orbital Systems. The aim has been to optimize their product the Orbital shower, which is a circulating shower system.

The work evaluates different sensor techniques suitable for detection of contaminants in greywater. After a comprehensive literature study, three techniques; ultrasound, IR and impedance were further investigated and practically tested. Impedance values and IR absorbance were obtained from contaminants in still water, while the speed and frequency content of ultrasound was calculated using time of flight and FFT in both still and circulating water.

Test solutions used to evaluate the sensor techniques consisted of personal care products (shampoo, body wash and conditioner), human body contaminants and calcium. Varying concentrations of sodium chloride and cattle blood was used to simulate body fluids. Calcium was used to modify the hardness of water.

All three techniques were able to detect some contaminants and failed to detect others. The widest range of contaminants were detected when using a combination of IR and impedance. This combination did not only detect all test solutions but it could also to some extent quantify the contaminants. It is recommended that this combination should be investigated further. Technical parameters, advantages and limitations of each technique are discussed.

Lastly, an extensive recommendation was presented to Orbital Systems regarding how future research on sensor techniques should be conducted.

Sammanfattning

Detta examensarbete har utförts tillsammans med företaget Orbital Systems. Syftet har varit att förbättra detektionen i deras produkt, Orbital shower, som är ett recirkulerande duschsystem.

Under arbetet har flera sensortekniker, lämpliga för detektion av kontaminanter i gråvatten, undersökts. Efter en övergripande litteraturstudie har tre olika tekniker; ultraljud, IR och impedans, testats vidare praktiskt. Resultat från tester med IR och impedans har undersökts i stilla vatten. Hastighet och frekvensinnehåll på ultraljud har beräknats med time of flight och FFT i både stilla och cirkulerande vatten.

Testlösningar som använts för att utvärdera sensorteknikerna har inkluderat kroppsvårdsprodukter (schampo, duschtvål och balsam), kontaminanter från kroppen och kalcium. Varierande koncentrationer av natriumklorid och oxblod hra använts för att simulera kroppsvätskor. Kalcium användes för att ändra vattnets hårdhet.

Samtliga tekniker lyckades detektera vissa kontaminanter och misslyckades detektera andra. Det bredaste spektrumet av kontaminanter blev detekterat genom en kombination av IR och impedans. Denna kombination kunde inte bara detektera testlösningar utan kunde i viss mån även kvantifiera dem. Denna kombination av tekniker bör undersökas vidare. Tekniska parametrar, fördelar och begränsningar för varje enskild teknik diskuteras i rapporten.

Slutligen presenterades en utförlig rekommendation av vilka sensortekniker som fortsatt borde undersökas av Orbital Systems.

Acknowledgements

Popular abstract

Most innovative technology today contain at least one sensor of some sort. Sensors can measure movement, temperature or position to name a few. Limited research has been found on sensors regarding instantaneous water quality detection. This work is somewhat unique in this field.

Ever since the first water system was constructed in ancient Rome over 2000 years ago, water has been used in the same way. Water is taken from a big reservoir, used for one single purpose and then directly disposed. In ancient Rome, aqueducts led water from large lakes. Nowadays, pipes lead water from water treatment plants. Access to water is taken for granted in many parts of the world, but this is about to change. The outdated system where water is disposed immediately after use together with increasing population and climate change results in water scarcity in many parts of the world. One of the most affected countries, South Africa, has been forced to hold numerous nationwide campaigns acknowledging water use and trying to make the population waste less water.

Orbital Systems is an up and coming water saving company based in Malmö. Their aim is to change the way society uses water and help create a sustainable planet. Almost 30% of the total water consumed by a person each day is used as shower water. This is the first problem that Orbital Systems has tackled with their creation the recirculating Orbital shower. The shower analyses the water before it reaches the shower drain and if deemed safe to reuse, gets drizzled back down on the user.

The brain of the product is a systems that 20 times every second measures the water and decides whether or not to reuse the water. This work has researched and evaluated different sensor techniques in order to improve the sensitivity of detection and thus further optimize the use of water during a shower session. Practical tests, using different possible shower contaminants, were carried out for the three most promising techniques. These three were ultrasound, IR and impedance. Ultrasound is commonly associated with imaging fetuses, IR is frequently used in heat cameras or motion sensors and impedance sensors are commonly found in several industry applications.

Each technique had their advantages and limitations and it was easy to conclude that none of the three could detect all tested contaminates. A combination of techniques is required for optimal detection and a sensor system using IR and impedance seems like the most promising combination.

Contents

1 Introduction

1.1 Background

Out of all the water available on our planet, a staggering small amount is available and deemed safe enough to use. Only 2.53% has low enough salinity to be useable and out of this small percentage only a fraction is accessible. The rest is either trapped as ice in glaciers or deep underground. This means that only 0.01% of all water on earth is usable and accessible for humans. Drinking water is hence a scarcity in many parts of the world and not much indicate that it will change anytime soon. [1]

Orbital Systems is an innovative water saving company based in Malmö. Their first product, the Orbital shower, is the world's first recirculating shower system. The product is sprung from a space project question from NASA: "how can we enable a lifestyle on Mars similar to that on earth?". The company is now eight years old and has around 40 employees with a big area of expertise. The uniqueness of the shower is that it is able to reuse up to 90% of the water and save 80% of the energy used during a shower session. Prior to the water reaching the shower drain, it is analysed by a set of sensors. If the water is deemed to be uncontaminated, it is sent back up into the shower. The water then passes through two types of filters before it is reheated and can drizzle back down on the user. *Figure 1* show a schematic illustration of the Orbital shower. [2]

Each shower sessions begins with a start-up cycle where the water is heated to a starting temperature of $40°C$. During this cycle, the sensors are calibrated according to water that is available at that location at that particular time. Water parameters differ depending on location and time. It is thus vital for the sensors to be calibrated to the water being used for that particular shower session. The response from the sensors is the deciding factor whether the water can be reused or not. The sensors can be exposed to a large range of possible contaminants. It can be chemicals from personal care products, body fluids from the user such as sweat and urine or it can be dirt, hair and other larger particles. The sensors must not only be able to detect these contaminants, but must also quickly be able to detect when the water is uncontaminated in order to start reusing the water again.
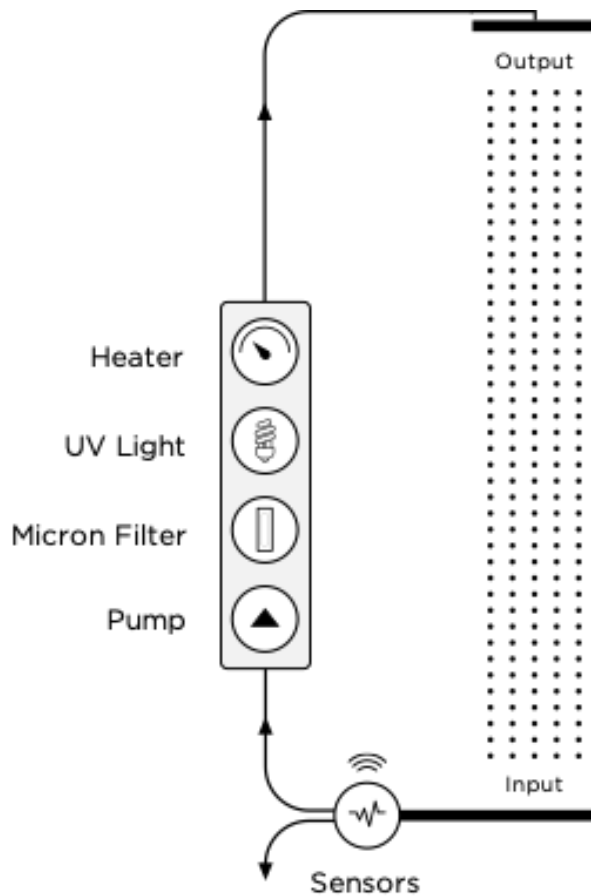
Figure 1: *Schematic of the recirculating Orbital shower.*

Orbital Systems is still a young company and they are aware that all technical possibilities are yet to be explored. The sensor system currently in place works sufficiently but the company believes that it can be further improved. Three versions of the shower have already been created and further versions are under development. An updated sensor system can easily be integrated in these new versions and can benefit the user and the environment in the near future.

1.2   Aim

The main objective is to, at the end of this thesis, present a recommendation to Orbital Systems regarding what sensor techniques that could improve the detection of contaminants. The authors have during the duration of this work obtained detailed knowledge about the sensor system currently in place in the product. This information will partly be used in the recommendation. Due to the sensitivity of this information, the extensive recommendation will only be given to the company. The conclusions in this report will hence be in general terms and not consider any information regarding the sensor system currently in place. The recommendation will be supported by both literature and tests. The extent of testing reasonable to achieve is difficult to assess beforehand. The strengths and weaknesses of the sensor techniques will be presented and the recommendation will most likely be a combination of different sensors, rather than one sole sensor that can cover the wide range of possible contaminants.

There is a limit to how small concentrations the sensors can detect, and hence the product has filters that cleans the water. The durability of the filters are dependent on how much contamination they are exposed to, which means that more accurate sensors will improve the lifetimes of the filters. An improved detection will also make the product waste less clean water as it not only will be better at detecting when the water is contaminated but also when it is clean enough again to be reused.

One of the biggest challenges with this work is that the sensor will always be exposed to contaminants that are dissolved in water. The volume of the contaminant will more often than not be small compared to that of the water. Hence, the sensor has to be able to detect fairly small concentrations. Additionally, many of the contaminants already contain water, which complicates detection. The following possible contaminants have been in focus in this work:

- Shampoo

- Body wash

- Conditioner

- Sodium chloride

- Blood

- Calcium

Both urine and sweat contain different levels of salt. These biological rest products often occur in wastewater from showers and are thus of great importance to detect. Another biological waste product that can occur is blood. Although, it does not occur as often as sweat and urine it is still important to detect. Calcium was tested since it is naturally found in tap water at varying concentrations. Calcium can create deposits in all areas where water is present. These deposits can cause energy losses and additional power consumption [3]. Orbital Systems have a system in place to prevent calcium deposits, but excess calcium is not optimal. It is therefore interesting to be able to monitor the levels of calcium in the water and it has thus been included in this work as a possible contaminant. Further information regarding the different contaminants can be found in *Water content*.

The potential sensors will be exposed to the above listed contaminants in order to evaluate what sensor is best fitted to be integrated in a product such as the Orbital shower.

1.3   Approach and Limitations

A sensor in its most basic form is a device that changes its electrical output according to the input to the device, which is the parameter being measured. An advantage of this work is that the sensors considered only have to detect a contaminant rather than to quantify it. Quantification demands much higher accuracy of the sensors than detection. This makes it possible to consider a wide range of different kind of sensors.

The process of finding a new sensor, applying it to a product and to implementing it is bigger than the scope of this work. Focus has been on finding interesting sensor techniques and to test them in order to give a recommendation. This has led to the authors of this work having a strict engineering focus, rather than a business focus or a final solution in mind. Factors such as integrating a possible sensor with existing sensors, use of software or commercial factors are not considered.

Factors that have been considered are presented in the list below in order of importance. The first factor, time, has been the most crucial one. The detection cannot take too long, as the decision whether the water should be recycled or not has to be close to instantaneous. The distance between the sensor and the inlet for the recycling circuit is around 15 *cm* and the decision has to be made during the time the water travels this distance, which is around one second. Currently, measurements are taken 20 times every second. All the sensor techniques considered in this report are capable of making a detection in such a small period of time that they are possible to be considered

as future sensors in the product. The second factor considered is accuracy. The concentrations tested are fairly small and an implemented sensor in the product can be exposed to even smaller concentration. It is important that the sensor gives as few false positives as possible. To have contaminated water wash over the user is worse then accidentally waste some clean water, i.e. a false negative response from the sensor. As previously mentioned, the product has two filters that takes care of potential false positives from the sensor. Many incorrect detections will worsen the filter and thus the accuracy of the sensor is important. Another factor that has been examined is the durability of the sensor. The life cycle should not be shorter than that of the product. One possible solution could be to make the user change sensors themselves, but this is not optimal. Furthermore, the sensor should be able to be made fairly small, such that it can be integrated in the product. An actual size limit has never been stated, but rather something that is theoretically possible to be made small enough to be included in a product. For instance, all types of spectroscopy are done with machines that would be bigger than the product itself and hence deemed as to large. The last factor that has been considered is the cost. No thoughts have been given on what technique is the most economically beneficial to include in the product or what solution is the best economically to scale up. The only economical aspect that has been viewed is that the components bought to be used in testing should be reasonable with the research budget that Orbital System has. Factors considered:

- Detection time

- Accuracy

- Durability

- Size

- Price

Given the requirements described above, an extensive literature study was performed. Firstly, the contents of water were reviewed, then possible contaminants from the human body and from personal care products were analyzed. From this, a vast amount of different sensor techniques were researched. Some internal knowledge regarding the current sensor system in place was also required and used in the evaluation of techniques. This knowledge however is confidential and not presented. The techniques were narrowed down by placing them in graph with *Theoretical potential* on one axis and *Practical feasibility* on the other, see *figure 2*. The placement in the graph was based on literature and discussions with supervisors. The three
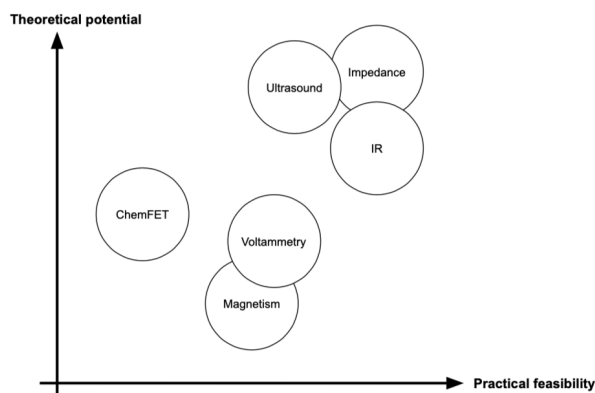
Figure 2: *Theoretical evaluation of sensor techniques*

techniques at the top right of the graph were the ones deemed best suitable and these were evaluated further.

The three honourable mentions that did not make the final cut were voltammetry, ChemFET and magnetism. These three techniques seemed probable at first but in the end they failed on one or more of the criteria. Voltammetry and especially cyclic voltammetry are frequently used in electrochemistry. It is a technique that is mentioned often in papers, but in the end the analysis is too slow to fit our requirements. The duration of the analysis is a matter of minutes rather than milliseconds. Magnetism is used at hospitals in MRIs. The detection of water using magnetic fields is theoretically possible but it is unlikely to be able to detect the small quantities of contaminants that this work tries to deal with. Lastly, ChemFET was a technique discarded due to its short life span. The membrane on a ChemFET is also highly specific towards one particular contaminant, which is not optimal as the sensors in the product needs to be able to handle a fairly big range of possible contaminants. Further, the membrane needs to be replaced in a matter of days, which does not fulfill the durability requirement.

The most promising techniques were the following:

- Ultrasound

- IR

- Impedance

These three sensor techniques are researched in depth, tested and evaluated in this report. The theory and reasoning behind the choice of these three techniques will first be explained. The techniques will then be tested, discussed, and finally be given a total performance score. A summarized illustration of the described project approach can be seen in *figure 3*.
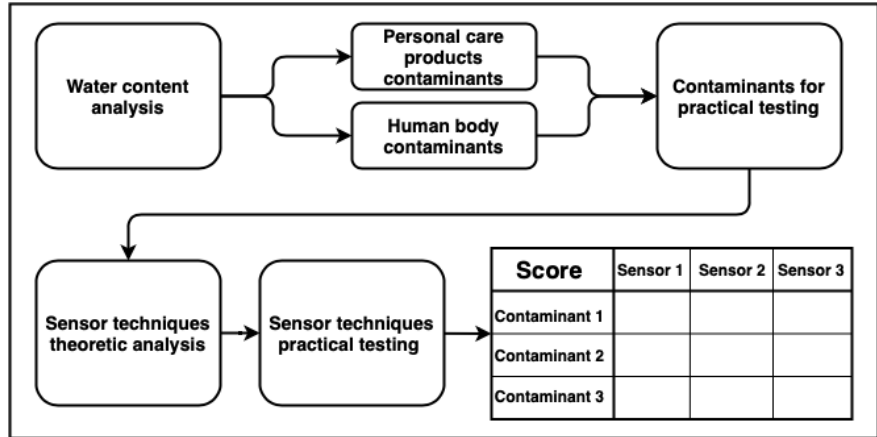
Figure 3: *Project approach.*

2   Theory

2.1   Water content
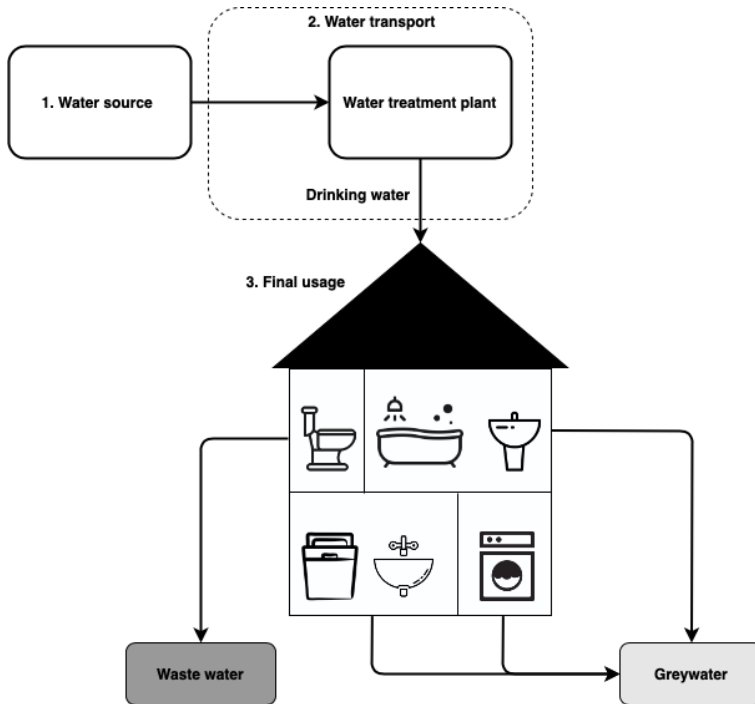
Figure 4: *Example of greywater sources in a household.*

Greywater is the water generated from all sources in a building except toilets. Examples from a typical household are showers, baths, sinks or washing machines, see *figure 4*.

The quality of the water varies depending on its characteristics, which can be divided into three major categories: physical, chemical and biological. Temperature, color and solid substances such as hair, food and fibers are examples of physical parameters. Ions and substances derived from personal care products belong to the chemical category while the biological category covers microorganism and biofilm. The content in the different categories are affected firstly by the source of the water, secondly by the transport and lastly by the final usage. [4]

Water source

Tap water used in Swedish households originate either from surface water (lakes, streams and sea) or from groundwater. The groundwater is obtained from large depths underground and has therefore slower circulation rate than surface water, which generally results in a larger ion concentration. This concentration varies with bedrock and climate and is therefore different across the country.

The level of hardness in the water is measured in degrees of general hardness, °*dH* where 1 °*dH* is defined as 10 *mg* of CaO per litre water. *Table 1* shows how The Geological Survey of Sweden defines water hardness level depending on the °*dH* of the water. Tap water can differ greatly within a in a city depending on the source. In Lund the water hardness differs from 3.4 °*dH* to 14 °*dH*, where the low values are water originating from lakes while the high values are from groundwater [5]. Copenhagen is another town were the hardness of the water is frequently discussed, the water in that area varies between 18 °*dH* and 24 °*dH*. [6]

°*dH*	*Water hardness level*
< 2.1	*Very soft water*
2.1 - 4.9	*Soft water*
4.9 - 9.8	*Medium hard water*
9.8 - 21	*Hard water*
> 21	*Very hard water*

Table 1: *Hardness of water according to The Geological Survey of Sweden.[5]*

Other ions that influence the quality of the water are the salts chloride Cl^- and sulphate SO_4^{2-}. These are of interest as they affect the conductivity of the water. Sodium Na^+ is brought to the groundwater either from areas near the sea or from the bedrock that earlier was covered with saltwater, including the north coastline of Sweden. Similarly to sodium, areas that earlier were covered by saltwater have an increased level of chloride and sulphate. [7]

Water transport

The journey from source to tap can be long and the water is influenced during the transport, in particular regarding biological degradation of chemical compounds [4]. Precipitations from copper and zinc are common from faucets and piping made from brass [7, 8]. A low pH value in the water increases the risk of excessive levels of both lead and copper while the same level of degradation for zinc seem to be for a pH of 8 [9, 10]. Degradation of zinc is also supposed to be accelerated by use of disinfecting chlorine tablets due to their acidity. [4]

From a biological perspective, water is affected not just by transport, but also during storage [11]. However this is beyond the scope of this report and hence not discussed further.

Final usage
The water quality is affected by the source and transport before it enters a household. Up until this point, water quality is more or less equal for all sources in a building. From here the water is affected depending on usage. It can be used as dishwater, water for washing clothes, shower water and several other activities. A frequently referenced study [4] is a meta study which includes a comparison of greywater originating from kitchen sinks, bathrooms and laundries. Water obtained from washing machines contain the largest concentrations of detergents and sodium, and together with kitchens sinks, the higher values of suspended solids. Except one contributing study that showed high values of zinc in shower water, one can assume from this study that the cleanest water seems to be originating from the shower. [4]

The shower water is affected by both the user and the products used during a shower. Obviously, some of the substances ending up on the shower floor might neither come from personal care products nor from the body. It can originate from outdoor activities or food misfortune in the kitchen. These speculative contaminants are left out of this report. The examination of contaminants is limited to include normal shower usage, which is in line with the product guarantee provided by Orbital Systems.

The main reason to take a shower is to get clean and remove unwanted substances from the body. Substances originating from the body can include sweat, blood, urine or saliva. The main constituent of these body fluids is water. Sweat, blood plasma and urine contain around 95% water, while saliva can reach up to 99% [12, 13, 14, 15]. Apart from water, sweat consists mostly of sodium chloride together with other electrolytes, such as potassium, calcium and magnesium [16]. The concentration of electrolytes in sweat and blood plasma is near to equal to each other [17]. Blood additionally contains small concentrations of proteins and iron, which contribute to a difference in color [12]. Urine consists of a similar electrolyte mixture together with urea, creatinine and nitrogen [18, 19]. What is noteworthy, particularly with urine and sweat, is that the concentration of electrolytes differs between individuals and from day to day.

Commonly between the body fluids mentioned above is that the total amount leaving the body during a shower often is small, and the concentration of the constituents are very low. Other body fluids that are not considered in this report, due to possibly even lower concentrations of detectable substances are tears, semen, snot and gastric acid. Substances aside fluids leaving the

body during a shower could involve hair, nails, vomit, faeces or dead skin. These larger substances are neither considered in this work. Particles of this size are not a risk to the product. The majority is filtered out at an early stage and thus does not affect the life span of the product.

Several different products can either be used or washed off during a shower. A selection of such products include shampoo, conditioner, body wash, soap, styling gel, hair wax, perming products, coloring products and many more. This section will describe a more detailed investigation of three commonly used products. These three were partly chosen due their frequent occurrence in shower sessions and partly due to a request by the company.

- Shampoo

- Body wash

- Conditioner

Shampoo can come in many different shapes and colors, often in the form of a viscous liquid. Different brands provides a wide variety of thickness, color, fragrance and other special additives. Shampoo represent the largest segment of hair care products [20]. Despite their apparent differences, what almost all shampoos contain in order to work as a cleansing product are surfactants [21]. 10-20% of the content in shampoo is typically represented by surfactants. Supplementary ingredients added, e.g to change pH, fragrance, viscosity or preservation usually add up to maximum a few percentages each of the total volume [20]. Therefore, together with water, a mix of surfactants form the main ingredients in modern shampoo formulas. *Table 2* shows some of the most popular used surfactants.

Ingredient	Surfactant	Function
Cocamidopropyl Betaine	*Amphoteric*	*Lathering*
Sodium Lauryl Sulfate	*Anionic*	*Lathering and cleansing*
Sodium Laureth Sulfate	*Anionic*	*Lathering and cleansing*
Ammonium Lauryl Sulfate	*Anionic*	*Deep cleansing*

***Table 2:** Commonly used surfactants in liquid shampoo.*

Surfactants in shampoo can be divided into four groups depending on the charge of the molecule, anionic, cationic, amphoteric and nonionic. Negatively charged surfactants, anionic, are primarily used in shampoos as cleanser. Sodium Lauryl Sulfate, SLS, or Sodium Lauryl ether Sulfate, SLES, are two of the most popular to use [21, 22]. Cocamidopropyl Betaine, CAB, is another widely used surfactant in shampoo [20, 21]. CAB is an amphoteric or

zwitterionic surfactant which means that it has both a negative and a positive charged polar group.

The formula of shower gel or body wash is similar to that of shampoo. The main constituent is water and surfactants. It also often contains some sort of skin benefit agent such as glycerin. Color and fragrance can vary between brands [23]. In order to get a clear picture of what commonly used body washes contain, a minor review was performed. The table of content for seven test products, found in regular grocery stores, was analysed and presented, see *table 3*. The surfactants CBA and SLES are commonly used. Both surfactants were present as one of the five prime ingredients in six out of the seven test products. Other common ingredients found in body wash are sodium chloride and glycerin, which were found in four respectively three products.

Product/Ingredient	1	2	3	4	5
Neutral shower gel	SLES	CAB	Glycerin	Sodium chloride	Sodium cocoyl glutamate
Nivea shower cream	SLES	CAB	PEG-7 Glyceryl cocoate	Glycerin	Glyceryl glucoside
Barnängen Dusch creme	SLES	Sodium Chloride	Disodium cocamphodi-acetate	CAB	Yogurt
Dove deeply nourishing shower creme	CAB	Sodium hy-droxypropyl starch phosphate	Lauric acid	Sodium lauryl glycinate	Sodium lauryl isethion-ate
Rexona shower gel aloe fresh	SLES	Aloe barbadensis leaf juice	Benzophenone-4	Citric acid	Cocamide MEA
Family fresh shower cream	SLES	Sodium chloride	CAB	PEG-4 Rape-seedamide	Lauryl glucoside
Ldb shower cream	SLES	CAB	Sodium chloride	Glycerin	Sodium lauroyl glutamate

Table 3: *Seven commonly used body washes. The number indicates the order which the ingredient has in the table of content for each product.*

Shampoo and body wash are both used to wash of dirt, the purpose of conditioner however is exclusively to deliver conditioning agents onto the hair. Therefore, the ingredients in conditioner differ compared to that of shampoo or body wash [20]. There are three main constituents in hair conditioner apart from deionized water: cationic surfactants, fatty alcohols and conditioning agents. *Table 4* show the distribution of the key ingredients in conditioner.

Ingredient	Function	Example	Amount of total volume
Water	Solvent	Deionized water	Up to 90%
Cationic surfactant	Surfactant, conditioning agent	Behetrimonium chloride, steartrimonium chloride, stearyl amidopropyldimethylamine	0.5 - 3%
Fatty alcohol	Cosurfactant, conditioning agent	Cetyl alcohol, stearyl alcohol, behenyl alcohol	0.5 - 10%
Silicone	Conditioning agent	Dimethicone, dimethiconol, amodimethicone	0 - 10%
Oil	Conditioning agent	Cetyl esters, mineral oil, oleyl alcohol, cetyl ethylhexanoate, squalane	0 - 5%
Polyol	Solvent, humectant	Glycerin, propylene glycol, dipropylene glycol, butylene alcohol	0 - 5%
Polymer	Rheology modifier	Hydroxyethyl cellulose, PEGs	0 - 2%
Organic acid	pH adjuster	Citric acid, lactic acid, glutamic acid	0 - 1%
Scalp care active	Scalp care	Zinc pyrithione, octopirox, dipotassium glycyrrhizate	0 - 1%
Preservative	Preservation	Methylparaben, phenoxyethanol, methylisothiazolinone	Less than 1%
Perfume	Fragrance	-	0.1 - 1%
Color	Appearance modifier	-	0 - 0.5%

Table 4: *Key ingredients in general conditioner formula. [20]*

2.2   Analysis background

2.2.1   Ultrasound

The human ear can detect sound waves of frequencies up to around 20 *kHz*. Sound waves with frequencies above this are classified as ultrasound. When sound hits an object or encounters a new medium, some of the waves will be reflected back and some will continue. The reflected waves can be analysed and give information about the object or medium that caused the reflection. This technique is most commonly associated with sonograms where a fetus inside a womb is examined. Another of the more known applications is sonar, where distance underwater is measured or used by fishermen to detect school of fish. The frequency of the ultrasound is of big importance as higher frequencies are more concentrated resulting in a high resolution in a medical image but lower depth penetration. By using lower frequencies, the sound waves will be able to be attenuated less and hence penetrate deeper. [24]

There are numerous advantages of using ultrasound sensors. The ones most relevant for this work is the high precision when it comes to speed measurements, the fast sensor response and that ultrasound sensors are robust and can be used for a long time. Two drawbacks are that the measurements are highly dependant on temperature and also sensitive to air bubbles. [25]

There is a big width of parameters that one can measure with ultrasound. One is the attenuation of the sound in the medium. A second is the acoustic impedance, which is a measurement of what happens with the sound at the interface between two different materials or mediums. These two parameters are not suitable for this thesis, rather two other different parameters have been paid more attention to. The first being the speed of the sound in the medium, usually denoted as c. The speed varies much depending on the medium it travels in, as well as the temperature of the medium. The speed in a fluid system is described by the following equation:

$$c^2 = 1/\rho\beta$$

where ρ is the density of the medium and β is compressibility. Compressibility is the measurement of the change in volume in response to external pressure. Both these two parameters are highly substance specific and it has been shown that the compressibility of liquids changes strongly with little variation in ion concentration or kind of ions [26]. This is further backed up by a study carried out by Chen-Tung Chen et al. [27] who studied the speed of sound in salt solutions. They found that a change of 0.03 *M* of NaCl resulted in a change in relative speed (having the speed of sound in pure water subtracted from the measured speed) from 0.58 *m/s* to 2.45 *m/s*. The speed of

sound is measured as time of flight, ToF, between a transmitter and receiver over a fixed distance.

The other parameter investigated in this thesis is the change in frequency of the ultrasound. This is today most commonly used in doppler shift measurements, where for example blood flow in the body can be analyzed. Through discussions with supervisors and other experts, it was hypothesised that the medium that the signal passes through could change the frequency of the signal. By analyzing the frequency content of the signal with the use of Fourier Transform one can see whether the presence of contaminants changes the frequency of the signal. A study [28] used the Fast Fourier Transform, FFT, in order to analyze water contaminated with kaolin, a form of clay. Their results were not totally conclusive, but they are promising enough for the method to be considered in this work.

A potential source of error that needs to be minimized is turbulence of the flow. Turbulence is the opposite to laminar flow where there can be local currents perpendicular to the direction of flow or swirls in the fluid. Laminar flow is when the fluid flows smoothly in paths with very little or even no mixing. Another study [29] showed that the propagation time of the sound wave depended on what path it took inside a pipe when turbulent flow was present. The effect of the turbulence was not quantified but it could be determined that a problem existed. The type of flow is decided by the viscosity of the fluid, its density and the dimensions of the pipe that the fluid flows in. The pipes used in the testing were small and together with the relatively low flow of the liquid, laminar flow should be obtained. However, random fluctuations can occur and this can cause a change in speed measurements [30]. Furthermore, the study [29] states that *"electronic noise can be neglected in comparison to turbulence induced noise"*. Noise is never optimal to have, but the fact that one can ignore noise from electronics makes the experimental setup easier to create.

Two different setups are possible. One where the sensor acts as both a receiver and a transmitter, *A*, and one where there is a separate transmitter and a separate receiver, *B*, commonly called through-transmission technique, see *figure 5*. In the case of one unit acting as both transmitter and receiver, the sound wave will bounce of a surface and then return. Hence, the distance measured will be twice that of the case when there is two separate units as transmitter/receiver. The time taken for the sound to reach the receiver will be measured, and by knowing the distance travelled, the speed of the sound can be found. Literature suggests that the speed can be determined with an accuracy better than 0.1 *m/s* [26].
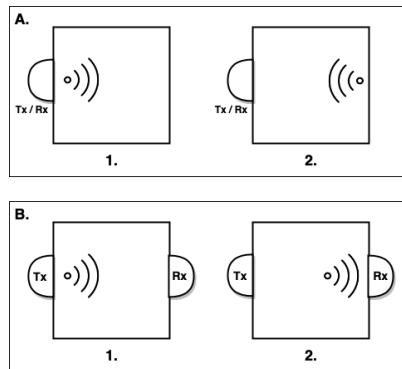
Figure 5: *Example of two possible setups. The upper figure (A) show a sensor acting as both transmitter and receiver. The lower figure (B) show two separate sensors, one transmitter and one receiver.*

The previous mentioned drawbacks of ultrasound measurements can be handled. A separate sensor can measure the temperature of the water which can then be used to adjust the other sensors to achieve accurate detection. Many sensors are dependant on temperature and to measure and calibrate for temperature is very common. Another drawback is the fact that the acoustic properties of the medium must be know in order to achieve accurate concentration measurements. This is not of concern in this thesis as the focus is on detection rather than quantification. The biggest source of error for this technique are potential air bubbles, due to the big difference in speed of sound in air compared to water, see *table 5*.

Medium [20°C]	Speed of sound [m/s]
Air	343
Water	1482
Salt water (3,5% salinity)	1521
Blood	1570

Table 5: *Speed of sound in various medium.*

If the sound wave interacts with any air bubbles, it would result in inaccurate reading due to the big difference in the speed of sound between the two media. Apart from the difference in speed, air bubbles can scatter the sound wave, resulting it not being detected by the receiver [31]. It is thus important to try and avoid air bubbles as much as possible. Air bubbles can interfere with other types of sensors as well, meaning that avoiding them is beneficial for all sensors being tested.

In summary, the opportunities and risks of using ultrasound from literature.

Opportunities:

- High accuracy for speed measurements

- Small change in salt concentration will significantly change the speed of sound

- Long lifetime of sensor

- The analyte will change the frequency content of the signal

Risks:

- Temperature dependant

- Interference by air bubbles

- Interference due to turbulence of water

2.2.2   IR

Visible light is a common mean of detection in sensors used for water analysis. Light shines through a sample and a photodiode on the opposite side measures the amount of light received. The technique measures turbidity, which is a measure of the transparency of the fluid, commonly used to determine the quality of drinking water. Depending on how much the water has been contaminated, different amount of light will be scattered and not make it to the photodiode. There are nevertheless many contaminants that can be missed by a turbidity sensor, as they do not alter the transparency of water. Instead of using visible light, another option would be to use light of higher wavelength, especially in the infrared spectrum, between 700 *nm* and 1 *mm*. Within this set of wavelengths lie the particular wavelengths that make inter- and intramolecular bonds move, known as vibration of molecules. Molecules can vibrate in different ways depending on the wavelength of light they are exposed to. That particular wavelength is then absorbed. It is thus possible to detect certain molecules in a mixed solution by using IR at a wavelength such to cause the molecules to absorb the light and detect this with a photodiode.

A Fourier transform infrared spectrophotometer, FTIR for short, can be used to generate the absorption spectra for an analyte over the entire IR spectra. The instrument produces a beam of IR radiation which passes through an interferometer. The interferometer creates constructive and destructive interference of the IR beam by recombining beams of different path lengths with each other. This makes it possible to emit IR waves of a particular wavelength towards the analyte. A detector then measures how much of the waves that has been absorbed by the analyte over time. This process is done for a wide set of wavelengths and lastly a spectrum is obtained by the use of the Fourier transform. By looking at the peaks in a spectrum one can either tell what molecule was analyzed, if the characteristic peaks already are known, or find the characteristic peaks for a known molecule. [32]

The optimal situation would be to conduct a FTIR analysis of various test solutions and compare their IR spectra. From this, one could then single out the wavelengths of particular interest and use LEDs that emit those wavelengths and pair them with a photodiode that can detect the same set of wavelengths. Two main problems arose with this approach. The first being that FTIR analysis was fairly complicated to do and the spectrophotometer available at LTH was not functionable during the entire length of this work. The approach was therefore changed to instead use spectra from papers and databases and analyze these. The following is a spectrum from a paper analyzing surfactants in personal care products, see *figure 6*.
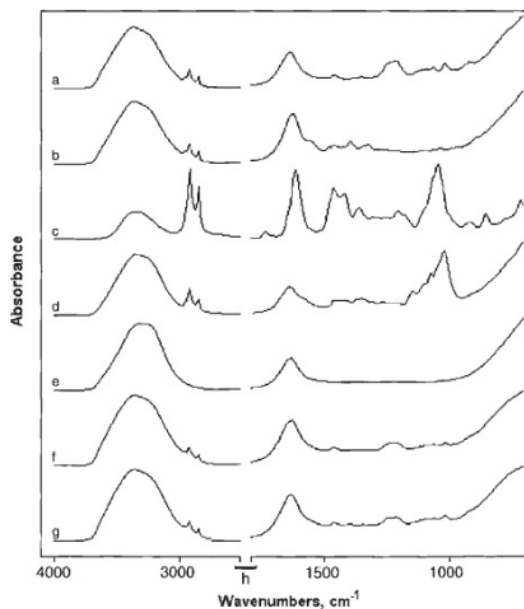
Figure 6: *Eight different absorption spectra for (a) sodium lauryl sulfate, (b) cocoamidopropyl betaine, (c) cocodiethanolamide, (d) alkylpolyglucoside, (e) water, (f) liquid soap and (g) shampoo. [33]*

When looking at the spectra above, one can see that around 1100 cm^{-1} there are some peaks that only appear for molecules used in personal care products, namely cocodiethanolamide and alkylpolyglucoside. Sodium lauryl sulfate that is a frequent constituent in both schampo and body wash has a peak at around 1200 cm^{-1}. All of these mentioned peaks are present at the same time as an absence of peaks can be seen in water. This is a vital factor as it makes it possible to detect the molecules in a mixture of water. Wavenumber and wavelength are two terms mentioned in literature that describe the same characteristic. The wavenumber is simply the inverse of the wavelength. A wave number of 1100 cm^{-1} is equal to a wavelength of around 9100 nm. This was where the second problem arose, it was difficult to find LEDs for wavelengths over 2500 nm. Some suppliers have one or two LEDs above 2500 nm but these did not match the desired wavelengths for this work. Different materials are used to create semiconductors that causes light to be emitted in a LED. *Table 6* show a selection of semiconductor materials with increasing maximum wavelength.

Semiconductor material	Bandgap (eV)	Max wavelength (nm)
Ge	0.660	1879
PdS	0.410	3024
InAs	0.360	3444
PbTe	0.310	4000
InSb	0.170	7294

Table 6: *Selection of materials used to create semiconductors. The third column shows the corresponding wavelength of the emitted light caused by each semiconductor.[34]*

The table only shows the semiconductors that produce the highest wavelength, semiconductors that produce lower wavelengths are omitted. It can be seen that there are some semiconductors that theoretically can produce LEDs of higher wavelengths than 2500 *nm*. However, it was understood that these semiconductors mostly are used in research and are not available for commercial purchase. Furthermore, the bandgap, which is the factor that decides the energy of the light, is small which means that the light will be difficult to detect. A detector for low energy light usually has to be cooled to very low temperatures. [34]

It is much easier to find LEDs in the area just above visible light, between 780 and 2500 *nm*. This area is called near infrared, NIR, and it also contains some interesting peaks. For instance, a study [35] found that it is possible to detect an overtone of OH groups at around 1420 *nm*. OH groups are a common side group in molecules, and the group exists in some of the substances found in personal care products. This is an interesting finding as it might make it possible to detect molecules without using wavelengths above the NIR area. The study however mentions that the peak is partly due to OH groups from molecules but also due to the water molecules that the solutions are mixed in. Another study [36] tested a number of samples containing molecules found in personal care products. The results indicate an interesting peak around 1140-1250 *nm* caused by the overtones of vibrational CH bonds present in the long alkyl chains of surfactants. As mentioned, literature points to the use of wavelengths above the NIR range for optimal detection of surfactants. However, detecting dissolved salts in water seems to be more promising. A study carried out by Reisha D Peters et al. [37] used wavelengths from the lower NIR region and showed positive results in the evaluation of NaCl and KCl in water of concentrations similar to the ones in this project. In another article studying the herring marinating process, a difference in concentration of NaCl could be measured using IR of wavelengths 1170-1290 *nm*. [38]

In summary, the opportunities and risks of using IR.

Opportunities:

- Certain wavelengths can detect specific molecules

- Overtones of OH and CH groups are possible to detect using wavelengths in the NIR spectra

- Numerous studies have successfully detected NaCl using NIR

Risks:

- LEDs above 2500 *nm* are difficult to obtain

- Chemistry of water might dominate

2.2.3   Impedance

Impedance (Z) is a value of the opposition to electric current in a medium or analyte. Impedance is a complex value that measure two different entities. The first real entity is the resistance (R) and the second imaginary entity is the reactance (X).

$$Z = R + jX$$

If a direct current, DC, is applied across a medium, the impedance will be equal to the resistance, which is not frequency dependant. This can be seen as impedance with zero reactance. When alternating current, AC, is used, the reactance becomes non-zero. A medium or analyte can show two types of reactance, inductance (L) or capacitance (C). Inductance is the medium's ability to oppose a change in current. This is due to the fact that flowing current creates a magnetic field around it. When alternating the direction of the current, when using AC, the direction of the magnetic field will change. This change in direction induces another electrical current in opposite direction of the change in current. Thus an inductive element will help oppose a change in current. The other type of reactance, capacitance, opposes a change in voltage. A capacitive element can be visualized as two conductors (material that leads current well) separated by and insulator (material that leads current poorly). An electric charge will be built up on one side of the capacitive element making it store charge. Should there be a drop in voltage in a circuit, a capacitive element can use some of its stored charge, hence a change in voltage will be opposed. Both the resistance and the reactance have the unit ohm (Ω). Both types of reactance are dependant on frequency, inductance increases with frequency while capacitance decreases:

$$X_L = \omega L$$

$$X_C = 1/\omega L$$

A second parameter of interest is phase (ϕ). The phase is the relationship between the resistance and the reactance. The phase angle shows how much shift there is between voltage and current. A perfect capacitive element will have a phase angle of -90° meaning that voltage is 90° after the current. A perfect inductive element will on the other hand have a phase angle of 90° meaning that the current is 90° after the voltage. [39]

Impedance spectroscopy is a method where the impedance is measured over many different frequencies. The impedance will change with varying frequency due to the capacitive or inductive behaviour of the solution. Solutions will show different impedance using the same frequency depending on

their chemistry. This is due to the fact that different chemicals lead electricity better or worse.

An impedance analyzer can perform an impedance spectroscopy. The analyzer keeps a constant current and voltage and varies the frequency. It can generate values for the real entity, resistance, and for the imaginary entity, reactance, for each frequency. It is also possible to measure the absolute value (ie. both resistance and reactance) and the phase angle.

$$|Z| = \sqrt{R^2 + X^2}$$

$$\tan(\phi) = X/R$$

The impedance analyzer is far more complex and big to be integrated into a product such as the Orbital shower. Instead one can construct impedance sensors that measure at one particular frequency. The values from the analyzer at different frequencies can then be compared and one can determine what frequency or frequencies one should measure on to best detect the different contaminants. This will be on the frequencies where the contaminants show the biggest difference from tap water.

In summary, the opportunities and risks of using impedance.

Opportunities:

- Can measure both absolute impedance and phase value

- Can analyze a wide range of different frequencies

- Impedance will change with chemistry of analyte

Risks:

- Not comparable to sensor that can be integrated in product

3   Method

3.1   Test solutions

Six possible contaminates have been used for test solutions. Some of the solutions have been evaluated in all of the measurements and a few have been used only for single tests.

The different contaminants have been used to create test solutions with varying concentration. The full list of test solutions can be seen in *table 7*.

Test solution	Concentration A	Concentration B	Concentration C
Shampoo	2 g/l	4 g/l	8 g/l
Body wash	2 g/l	4 g/l	8 g/l
Conditioner	2 g/l	4 g/l	8 g/l
NaCl	4 g/l	8 g/l	16 g/l
Calcium	32 mg/l	64 mg/l	128 mg/l
Blood	2.5 vol%	5 vol%	10 vol%

Table 7: *Test solutions and concentrations used during the entire project.*

Prior to the preparation of the solutions, a tank was filled with a sufficient amount of tap water. This was made in order to avoid the temperature variation that could occur when taking water from the tap multiple times. The container was kept in room temperature and all the solutions were mixed using water from this container. The test solutions were composed by first preparing the highest concentration. This was performed by weighing the product, using an Anvil brewing equipment scale, and mixing it with 500 *ml* water. A sample of this concentration was then saved. The solution of the highest concentration was then repeatedly diluted in order to obtain the test solutions of lower concentration, see *figure 7*. This method was used since the relationship between the solutions are more interesting than the absolute concentrations. This is a consequence of the project focusing on detection rather than quantification. All of the test solutions were kept in room temperature. The solutions used in the impedance measurements were mixed with deionized water except from calcium, which was instead mixed with tap water. This was done in order to quantify the impact of additional calcium to tap water, which already contains levels of calcium. The solutions used to test the techniques ultrasound and IR were mixed with tap water.
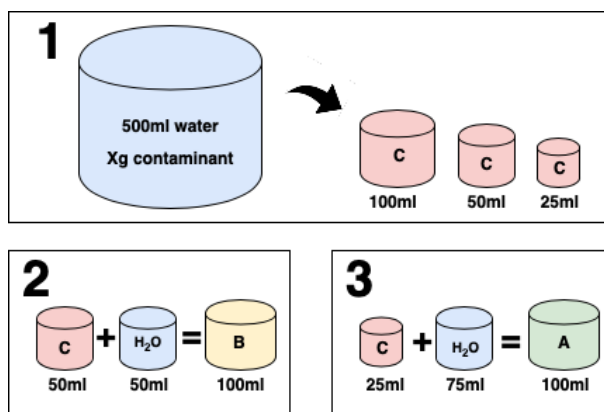
Figure 7: *Illustrates an example of the preparation process of the test solutions.*

The following parameters have been considered in the choice of personal care products.

- The personal care product should be found in regular Swedish grocery stores.

- The personal care product should not have any extraordinary purpose or contain any rare additional ingredient.

- For shampoo and body wash, the personal care product should contain the surfactants Sodium Laureth Sulfate and Cocamidopropyl Betaine described in *Theory*.

The shampoo used, Barnängen Normal shampoo, was confirmed by its producer to contain between 5-10% Sodium Laureth sulfate and between 1-5% Cocamidopropyl Betaine. The body wash used, Neutral shower gel, also contains the surfactants, but in unknown amounts. Three different concentrations was used for the test solutions containing personal care products. Four grams per liter has earlier been used for testing at Orbital Systems. Therefore, this concentration was used, together with a doubling and a halving in order to evaluate the impact of the concentration.

Sodium chloride was used to simulate urine and sweat. In a study [40] 8 *g/l* of NaCl was used in an artificial urine recipe. Blood, used for cooking obtained from cattle purchased in a regular grocery store, was used to simulate blood. See *Appendix* for table of contents of all products used.

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3.2   Ultrasound

The following materials were used for testing: Two ultrasonic sensors with output frequency of 2 *MHz*, two with frequency of 0.5 *MHz* and one *Panametrics Pulser/Receiver model 5072PR* (hereafter referred to as the signal generator). The signal generator is capable of changing the energy, damping and amplification of the signal. As previously mentioned in *Theory*, two setups are possible. One where a sensor acts as both a transmitter and a receiver and one where one sensor acts as a transmitter and another one acts as a receiver.

3.2.1   Initial testing

The optimal set up of the equipment, frequency and settings on the signal generator had to be found prior to real tests. A large water container was filled with tap water and the different types of sensors were tested in still water on different kinds of objects, such as the wall of the container, a drinking glass, the inside of a metal tube and the inside of a plastic tube. A digital oscilloscope, *Siglent SDS1102CML*, was used to view the output signal and the echoes received. A 12 V DC-pump was used to create airflow of 90 *ml/min* in order to create disturbing air bubbles in front of the sensors. A significant amount of NaCl was added to the container in order to perform a proof of concept. The setups, using one or two sensors, were tested on circulating tap water using a closed system powered by a *Topsflo TL-B10-B24-1208 DC pump* capable of creating a flow up to 12 *l/min*. Two different closed systems with circulating water were created in order to test both of the setups.

3.2.2   Still water

The setup containing two different sensors, one acting as a transmitter (Tx) and one acting as a receiver (Rx) was used. The sensors were glued onto two circular openings in a plastic tube, see *figure 8*. The tube was filled with varying solutions for the different tests. The water level was significantly higher than the height of the sensors in order to avoid having foam in front of them. The tube was thoroughly washed between each new contaminant. In order to achieve as accurate readings as possible, each solution was tested with increasing concentration as to avoid higher concentration solutions affecting the readings of lower concentrations.
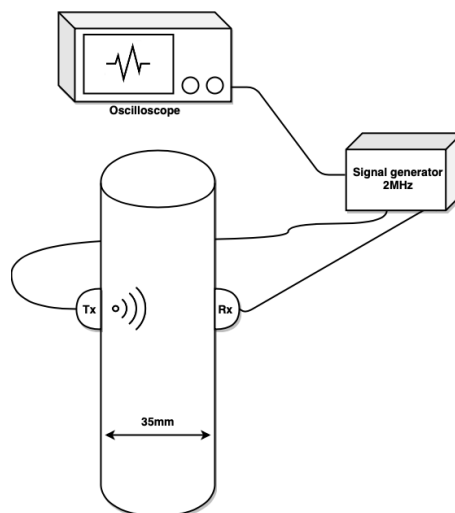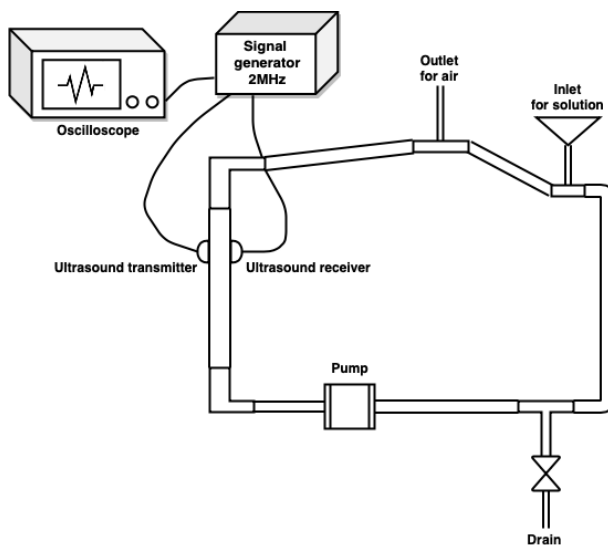
Figure 8: *Setup used for tests in still water.*

All the possible contaminants with their concentrations A, B and C mentioned in *table 7* were tested with ten performed trials for each. A three minute wait was held between the time that the contaminant was poured into the tube and the time of the first measurement. This was done in order to make sure that no foam affected the measurements and that a stable signal was obtained.

3.2.3   Circulating water

The tube containing the two sensors, one transmitter and one receiver, was integrated into a system with circulating solution. The system is shown in *figure 9*.

The circulating system was built in the lab at Orbital Systems using tubes and components available there. The set up with the oscilloscope and the signal generator was identical as in the tests with still water in *figure 7*. The system contained around 1.2 *l* of test solution and it was completely filled for each test. The previously mentioned Topsflo pump was used to create a constant flow rate of 0.1 *m/s*. The flow was measured by a *Kytola HV-4EC flowmeter*. In order to avoid air bubbles affecting the results, an outlet of air was created at the top of the system. The majority of the tubes in the setup were transparent which made it possible to wait until no visible air bubbles were present in the system before taking any measurements.

Figure 9: *Setup used for tests in circulating water.*

All concentrations of the following contaminants were tested with 15 trials each:

- Shampoo

- Conditioner

- NaCl

The contaminants were mixed and tested in an identical manner as with still water.

3.2.4   Data analysis

The output signal from the signal generator had 250 Ω damping and the signal was amplified with 10 *dB*. No types of filters were used. The signal was displayed on the oscilloscope described above and stored on a USB drive. The results were then analysed using MATLAB. Two different analyses were performed on the output signal, Time of flight, ToF, and analysis of the frequency content.

The ToF was simply calculated as the difference between the reference pulse and the first received signal, see *figure 9*. The transmitted reference pulse

started with a negative half period unlike the received pulse whose appearance varies. Different approaches regarding threshold were therefore used to select the start of the received signal. Frequency analysis of the received pulse was performed using Fast Fourier Transform, FFT. A Hanning window together with zero padding were applied prior to FFT-transformation in order to improve the quality of the signal in frequency domain. The FFT was then analysed as seen in *figure 9* but also normalized with the total signal in time domain. This normalized value is referred to as "FFT peak to total signal ratio". This was performed due to varying amplitude of the received signal. The "FFT peak to total signal ratio" was computed as the FFT peak value divided by the total signal amplitude which makes it dimensionless and gain independent.
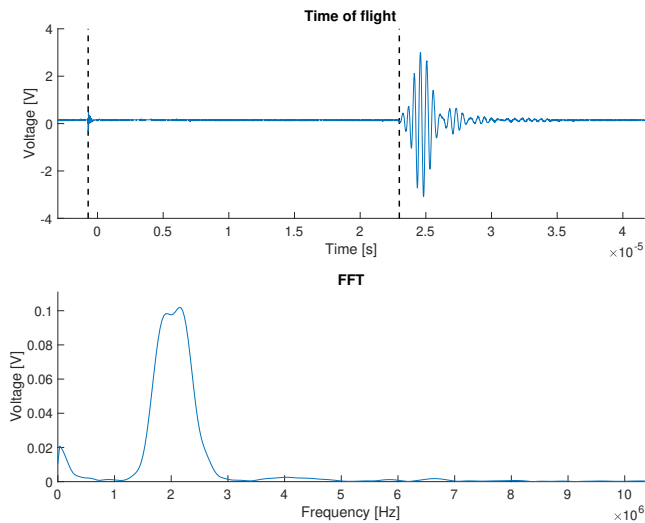
Figure 10: *The upper subfigure show the received ultrasound signal in time domain. The dashed lines indicate measured time between transmitted pulse to received signal. The lower subfigure show the same signal in frequency domain. The large peak at 2 MHz is due to the frequency transmitted by the ultrasound.*

3.3 IR

Three IR LEDs of peak wavelength 1200, 1300 and 1450 *nm* were ordered together with a photodiode capable of detecting IR light at mentioned wavelengths. Their individual spectra can be found in the *Appendix*.
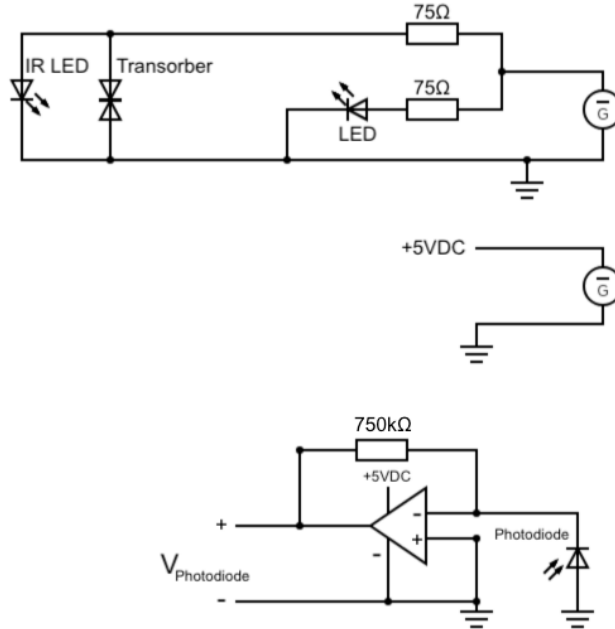

Figure 11: *Schematic drawing of the electrical circuit used to power the LEDs and to measure the output of the photodiode.*

Figure 10 illustrates the electrical circuit used in the IR measurements. The first thing one should note is the use of two LEDs in the circuit, one normal LED and one IR LED. The IR LED could be switched to either of the three mentioned in the beginning of this section. The normal LED was simply used as visual feed back to indicate that the IR LED was active. A second thing to notice is that the resistance in the lower part of the figure, 750 *k*Ω, was used to amplify the signal. The value of this resistor was decided through trial and error. A *UNI-T UT213B Digital Clamp Multimeter* was used to measure the potential difference across the photodiode. The circuit board was applied with 4.9 *V* and 300 *mA* by an *TTI CPX400DP power supply*. The same power supply applied the two LEDs on the circuit board with 7.6 *V* and 120 *mA*.

In order to make sure that the LED and the photodiode were at a constant distance from each other and completely opposite of each other, a small housing was produced. The housing was 3D printed and could thus be tailored for the LEDs and photodiode in question. The housing had the shape of a cube with sides of 2 *cm* containing four circular openings. Two circular openings opposite of each other had the same diameter as the plastic transparent tube that was filled with test solution. The plastic tube had a diameter of 9 *mm* and a thickness of 2 *mm*. The other two openings had the precise dimensions to fit the diode or any of the three LEDs. The photodiode and LEDs were attached so that the end of the sensors were in direct contact with the plastic tube. The test setup is illustrated in *figure 11*.
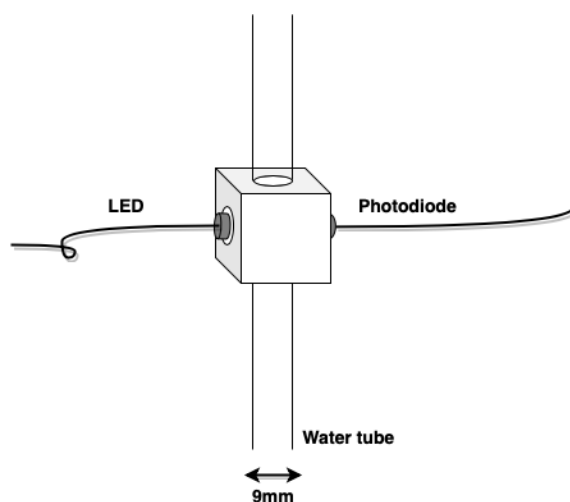
Figure 12: *The printed housing used to keep the LED and photodiode in a constant position during measurements. The housing was attached to the plastic tube containing the test solution.*

The contaminants were tested with increasing concentration as to avoid higher concentrations solutions affecting the results of lower concentration. All contaminants were tested with all the different concentrations as presented in *table 7*. Three iterations were made for each concentration. Measurements were taken by manually reading of the multimeter. Instead of making one instantaneous measurement, one was taken every tenth second over a period of one minute for each trial. This was due to the fact that the LEDs produced a significant amount of heat when current was applied across them, causing the photodiode to register decreasing voltage over time. See example in *figure 22*.

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To ensure that heat from one trial did not affect another, a two minute wait was held between each iteration. The duration of this wait was found through trial and error. Another wait of five minutes was held between each different test sample. This was partly due to the need of cooling but also to ensure that the sample was completely still once measurements were taken.
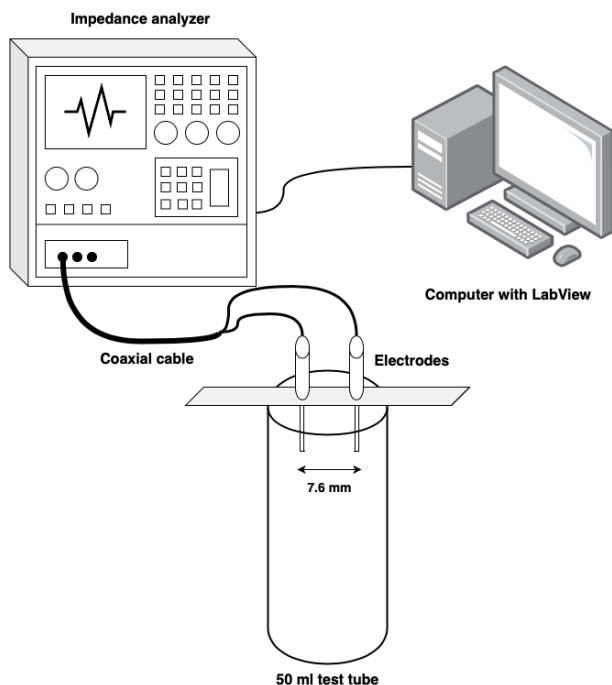
3.4   Impedance

A *Hewlett Packard 4194A Impedance/Gain-phase analyzer* was used to measure the impedance of the contaminants. The analyzer gave an absolute value for the impedance, i.e. both resistance and reactance, and a value for the phase angle for a given frequency. The analyzer was able to measure over a given range of frequencies, known as a sweep over frequencies. Over this set 1000 measurements were taken with even increments. Two hollow injection needles in stainless steel were used as electrodes. The electrodes were glued onto a piece of circuit board and partly covered in plastic in order to have the electrodes at a set depth in the test solutions and have a set surface area of the electrodes exposed to the solution. The tip of the needles were sealed with glue in order to avoid contamination inside the hollow electrodes. The electrodes were 7.6 *mm* apart, had a diameter of 1.15 *mm* and the length of the electrodes that were not covered in plastic were 28 *mm*. A coaxial cable was used to connect the analyzer to the electrodes. The setup can be seen in *figure 12*. Labview was used to visualize the results and to export the data to MATLAB. The electrodes were dipped in deionized water after each test to avoid measurements errors due to contamination of the electrodes. The tests were performed with increasing levels of concentration of the contaminants as to avoid the high concentrated solutions affecting the results of the lower concentration solutions.

The test solutions were made of a mixture of the contaminant and deionized water. More information regarding the test solutions can be found in *Test solutions*. The deionized water and the contaminants were kept in room temperature for such a long time that the temperature in the samples was assumed to be constant.

3.4.1   Session one

The impedance measurements were made during two sessions. During the first session, two different ranges of frequencies, 110 *Hz* - 10 *MHz* and 110 *Hz* - 10 *kHz* were tested. Each test solution was tested over both sets of frequencies once directly after each other. The contaminants tested were identical to those presented in *table 7*, apart from calcium. In order to examine if calcium detection was at all possible, a significantly higher concentration than that presented in *table 7* was prepared. This concentration simulated "very hard water" and contained approximately 200 *mg/l* calcium.

The test solutions were prepared in 50 *ml* plastic test tubes and filled up to 45 *ml* each time to get a constant submerging of the electrodes. It was made sure that the electrode reached the liquid in the test sample and not the foam

Figure 13: *Setup used for impedance measurements.*

at the top as this would result in defected measurements. The test tubes were shaken before each test to make sure that sample had homogeneous distribution of the contaminant. The measurements were then taken once the water in the test tube had become visually still.

3.4.2   Session two

During the second session of tests, two other ranges of frequencies were tested, 1 *kHz* - 100 *kHz* and 40 *kHz* - 40 *MHz*. 40 *MHz* was the maximum frequency achievable by the analyzer. The concentrations of contaminants tested during this session was identical to the concentrations in the first session, apart from calcium. All samples were mixed into deionized water and the measurements were repeated five times for both sets of frequencies. A more extensive study of calcium was conducted during this session. A starting concentration of 500 *mg/l* was continuously diluted evenly nine times to obtain ten different concentrations of calcium. The calcium was mixed with tap water and the measurements were repeated three times for both set of frequencies.

Lastly, it has to be mentioned that selectivity occurred when taking the measurements. The coaxial cable that connected the electrodes to the analyzer was sensitive and if moved created measurement errors. An example of such obvious error can be seen in *figure 26*. The analyzer displayed the measurements taken in real time and it was often possible to see whether the measurement was defective or not. If this was the case, the measurement was redone without it being recorded as one of the iterations.

4   Results

4.1   Ultrasound

4.1.1   Initial testing

The initial testing was performed through trial and error and will thus be presented as a summary rather than with figures. The ultrasound sensors with frequency of 2 *MHz* produced more clear echos than the 0.5 *MHz* sensors and were hence chosen to be tested further. The optimal amount of amplification was found to be 10 *dB*, this gave a clear signal while still being under the 5 *V* limit maximum reading of the oscilloscope. Above this level, some of the frequency information was in risk of being lost. Air bubbles created in still water caused the signal to disappear for a short period of time before returning, looking identical in comparison with the signal before the disturbance.

When adding NaCl into the water container the received signal seemed to have moved closer to the reference signal. The cursors in the oscilloscope were used to estimate the change in time of flight over a distance of 2 *cm* to around 400 *ns* faster in water containing NaCl compared to tap water.

When testing on circulating water and using one sensor as both transmitter and receiver, no echo could be observed. When starting off in still water, a clear and strong echo could be seen on the oscilloscope, but directly after the circulation had started, the output signal became noisy which drowned the echo. However, when using two separate sensors a clear signal could be observed even after the circulation was started.

4.1.2   Still water

The most interesting results are presented below. The difference in time between the slowest and fastest Time of Flights out of the 10 iterations, for all the solutions, are presented in *table 8*. These values are named delta time and indicate the precision of the results.

Figure 13 and *figure 14* show the result of the ToF measurements in still water. The x-axes indicate the time in seconds between the reference pulse and the first received pulse. The y-axes indicate the number of samples. The ToF for the iterations of tap water are between 25.90 and 25.93 μs. This can be observed in both of the figures. The ToF for the different concentrations of NaCl can be seen in *figure 13*. The strongest concentration, *C*, 16 *g* NaCl per litre of water, is displayed in red. The ToF for the iterations of this concentration are between 25.66 μs and 25.68 μs. This means that the sound travel approximately 250 *ns* faster in 16 *g/l* NaCl than what it does in tap water. *Figure 14* show the same comparison but for conditioner and shampoo.

Sound travels faster in shampoo than what it does in tap water. It travels slower in the conditioner solution.

Figure 15 show the frequency content of the received signals in tap water and in the three concentrations of NaCl. The amplitude of the peak at 2 *MHz* is larger for all concentrations of NaCl compared to tap water.

| Delta times [ns] - Still water			
Concentration	*A*	*B*	*C*
Conditioner	*30*	*70*	*30*
NaCl	*30*	*30*	*20*
Shampoo	*30*	*10*	*30*
Tap water	*30*	*-*	*-*

Table 8: *The difference in time between the slowest and fastest ToF out of the 10 iterations for each contaminant.*
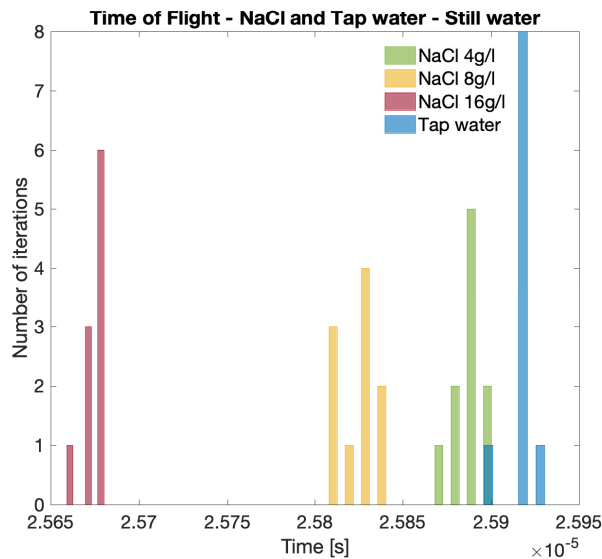
Figure 14: *ToF for three concentrations of NaCl compared to tap water, in still water.*
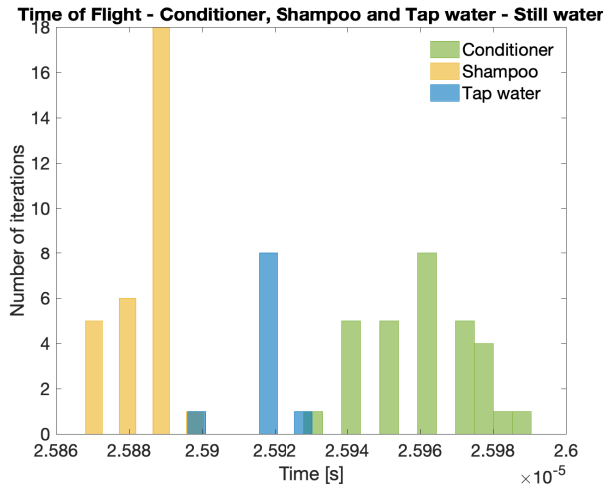
Figure 15: *Time of Flights for shampoo and conditioner in still water compared to tap water. 10 iterations of water and 30 iterations each of shampoo and conditioner. Note that the iterations of the personal care products are a mix of three concentrations.*
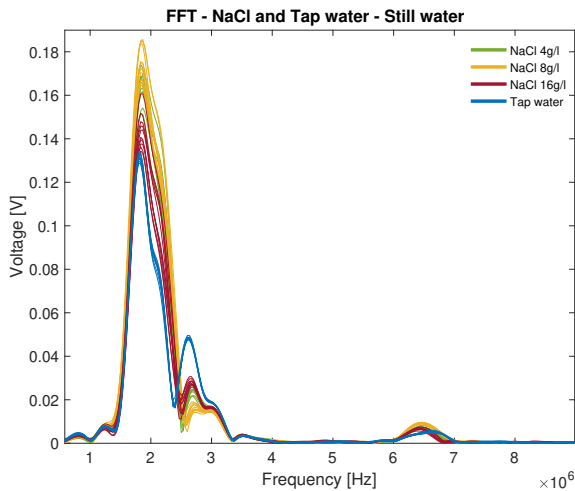
Figure 16: *FFT spectrum of the different concentrations of NaCl compared to tap water. This is the same iterations of data as presented in figure 13.*

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4.1.3   Circulating water

Due to foam, no signal was detected for concentration B and C for shampoo. Concentration A generated a signal, but the results could not be distinguished from tap water. The results from these tests have thus not been included. The difference in time between the slowest and fastest ToF out of the 15 iterations, for the tested solutions, are presented in *table 9*.

Figure 16 show that the difference in ToF between NaCl and tap water is also observable in circulating water. *Figure 17* show the measurements for conditioner and tap water, where no clear difference can be observed.

The frequency content of NaCl and tap water are shown in *figure 18*. There is no difference in amplitude between the peaks. *Figure 19* show a difference between the frequency content of tap water and conditioner. A distinct difference in amplitude of the frequency peak can be observed. The frequency content, in *figure 19*, is further normalized with the total signal in time domain and presented in *figure 20*. This figure show the "FFT peak to Total signal ratio", described in *Theory*, on the x-axis and the number of iterations on the y-axis. A difference between tap water and the iterations of conditioner can be observed.

Delta times [ns] - Circulating water			
Concentration	*A*	*B*	*C*
Conditioner	*250*	*220*	*200*
NaCl	*40*	*40*	*50*
Shampoo	*30*	*-*	*-*
Tap water	*80*	*-*	*-*

Table 9: *The difference in time between the slowest and fastest ToF out of the 15 iterations for each contaminant.*
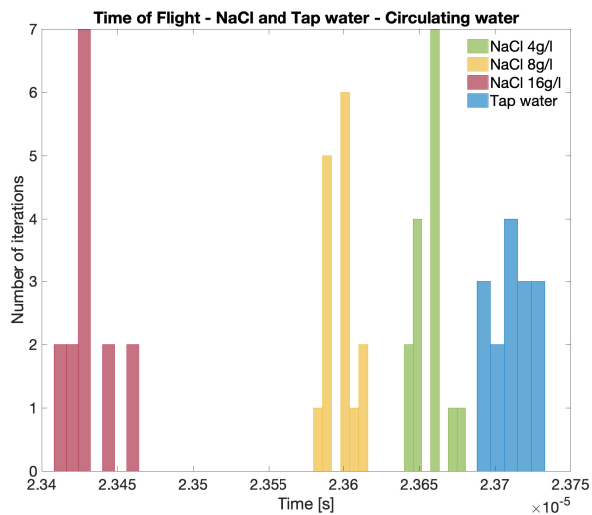
Figure 17: *ToF for three concentrations of NaCl compared to tap water, in circulating water.*
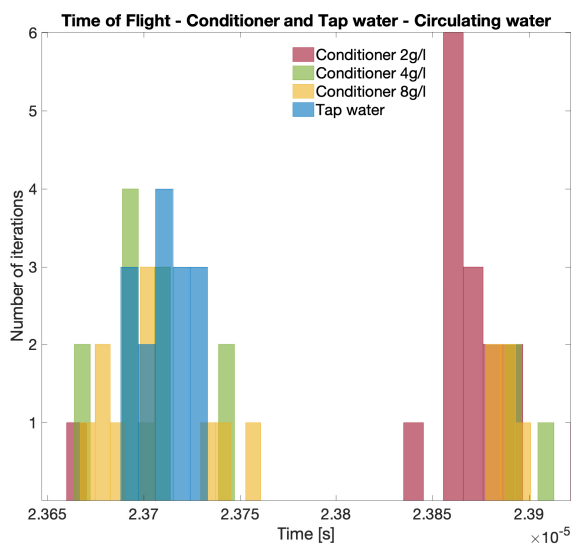
Figure 18: *ToF for three concentrations of conditioner compared to tap water, in circulating water.*
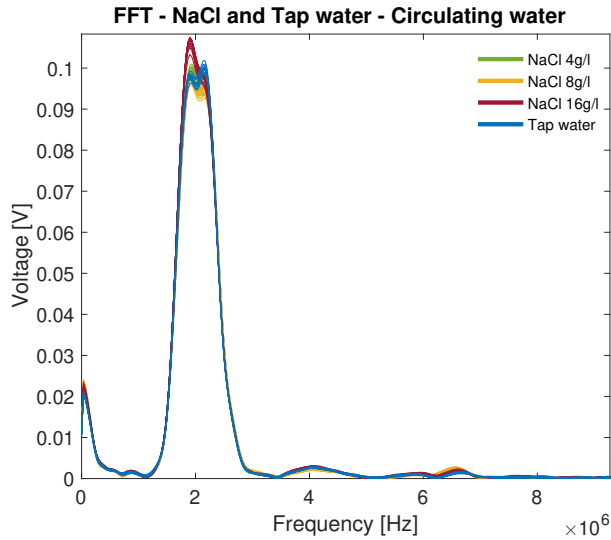
Figure 19: *FFT spectrum of the different concentrations of NaCl compared to tap water. This is the same iterations of data as presented in figure 16.*
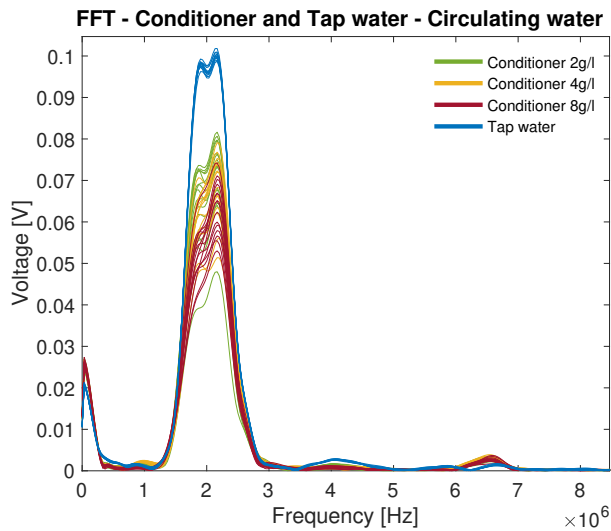
Figure 20: *FFT spectrum of the different concentrations of conditioner compared to tap water. This is the same iterations of data as presented in figure 17.*
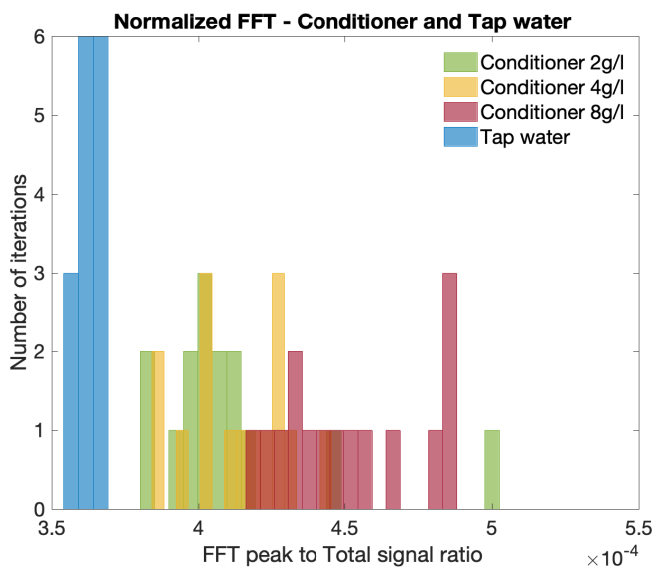
Figure 21: *Showing the "FFT peak to total signal ratio" for the different concentrations of conditioner compared to tap water.*

4.2 IR

4.2.1 Initial testing

A wide range of tubes in different materials, shapes and sizes were reviewed and tested in order to find a suitable tube for the test setup. This resulted in the cylindrical transparent plastic tube mentioned in the *Method*. The distance from the LED to the photodiode was determined by a tradeoff between signal response and practical feasibility. An even smaller distance would generate a greater signal response but make the test setup more difficult to create.

The increase in temperature creating a voltage drop over time seemed to be the same, independent of the initial voltage and thus independent of what solution that was examined. This resulted in taking measurements over a period of one minute instead of instantaneous measurements.

4.2.2 Still water

Figure 21 show the mean value, of concentration B, from all test solutions. The time, from 0 to 60 seconds, is displayed on the x-axis. The output voltage measured by the photodiode is displayed on the y-axis. The output voltage is highest for tap water (dark blue) and lowest for blood (dark red). This figure show the result using the LED with an output wavelength of 1200 *nm*. *Figure 22* show the corresponding results using the 1300 *nm* LED. A distinct difference between tap water and three test solutions can be observed. These three are blood (dark red), conditioner, (green) and calcium, (yellow).

Figure 23 show every measured iteration made for calcium using the 1300 *nm* LED. Three iterations of each of the three concentrations. The measurements are compared to three iterations of tap water (blue). A difference can be observed between all of the concentrations. The same presentation is made for the 1200 *nm* LED. The difference between concentrations for four test solutions can be seen in *figure 24*. The largest difference can be observed for blood, in the upper right subfigure.
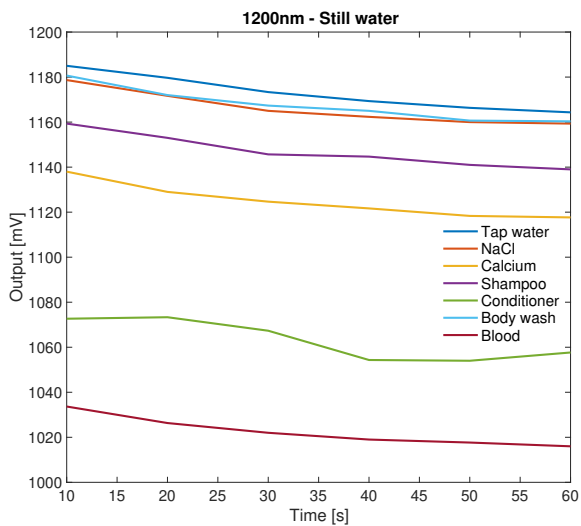
Figure 22: *Show a mean value of concentration B for all test solutions. Wavelength 1200 nm.*
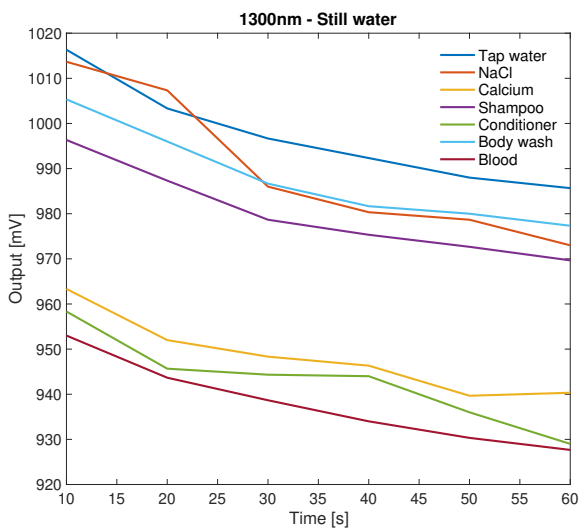
Figure 23: *Show a mean value of concentration B for all test solutions. Wavelength 1300 nm.*
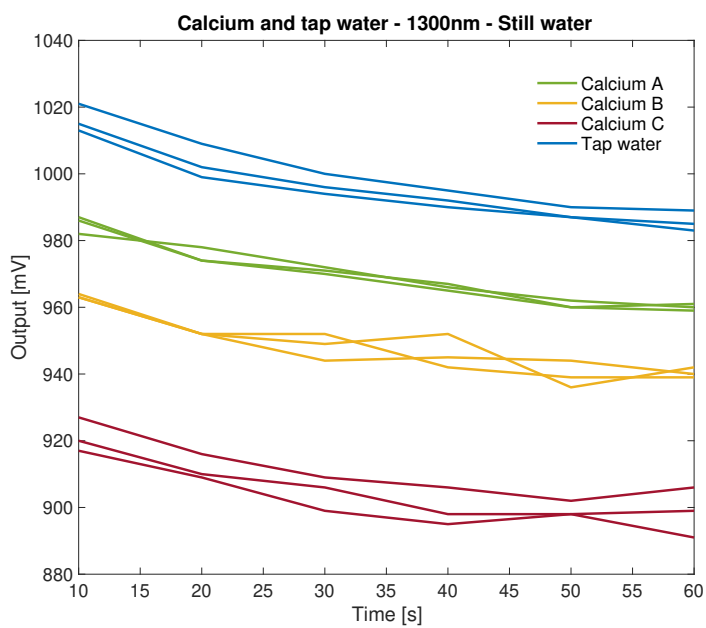
Figure 24: *Show the iterations from the three concentrations of calcium and tap water. Wavelength 1300 nm.*
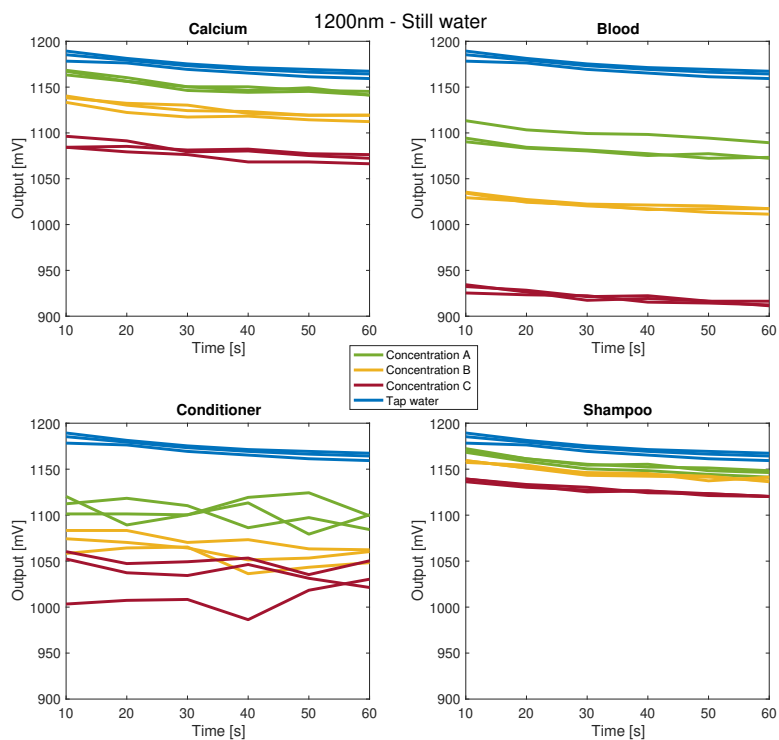
Figure 25: *Show the iterations of the three concentrations for four different test solutions compared to tap water. Calcium (upper left), blood (upper right), conditioner (lower left) and shampoo (lower right). Wavelength 1200 nm.*

4.3   Impedance

The results from the two test sessions are presented in the figures below.

4.3.1   Session one

Figure 25 show the impedance spectrum for frequencies from 110 *Hz* to 10 *MHz*. Impedance is displayed on the y-axis and frequency on the x-axis. It can be observed that all solutions, except NaCl, converge for frequencies above 1 *MHz*. The solution of highest impedance is conditioner (green), which can be seen on the top of the plot. The solution of lowest impedance is NaCl (yellow). The three different concentrations of NaCl is shown in *figure 26*. Note that this figure shows a measurement error for concentration B (yellow). The frequency range of this measurement is 110 *Hz* - 10 *kHz*.
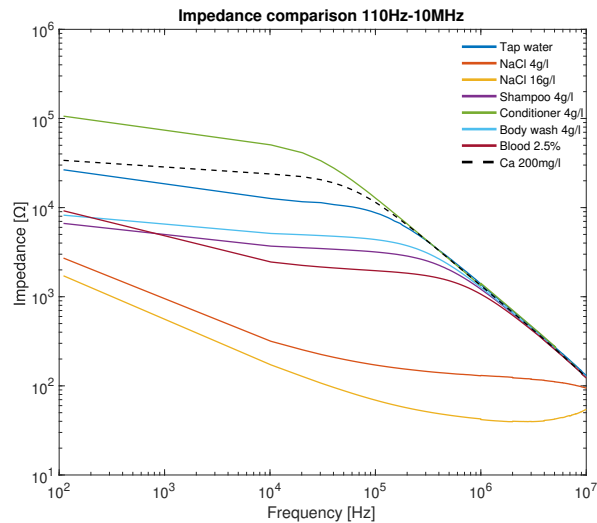
Figure 26: *Impedance spectrum from 110 Hz to 10 MHz for all test solutions. The concentration of each contaminant is described in the figure. The data presented are based on one iteration of each contaminant.*
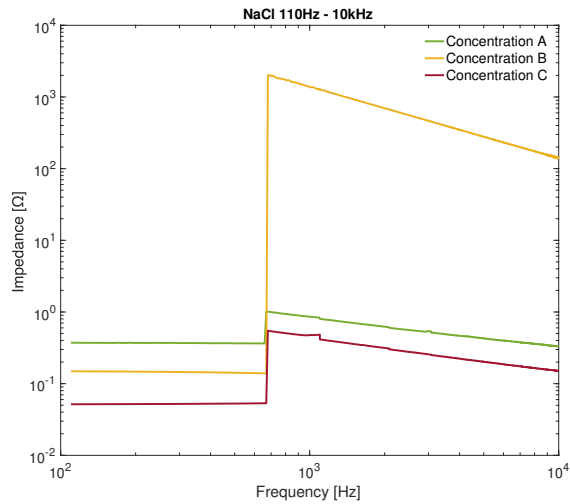
Figure 27: *Impedance spectrum from 110 Hz to 10 kHz for three concentra-tions of NaCl. Note that concentration B (yellow) is a measurement error.*

4.3.2 Session two

As can be seen in *figure 25*, the majority of the test solutions converge at a certain frequency. An enlarged view of the frequencies prior to the convergence in *figure 25* is displayed *figure 27*. This spectrum covers frequencies from 1 *kHz* to 100 *kHz*. The data presented are based on a mean value of five iterations, of concentration B, from each test solution. Deionized water (orange) differs from tap water the most for frequency 1 *kHz*. NaCl (yellow) differs from tap water the most for frequency 100 *kHz*. Body wash (light blue) is the contaminant which differs the least from tap water (dark blue) for all frequencies. The difference between the three concentrations of body wash can be observed in *figure 28*.

Figure 29 show the impedance for frequencies up to the maximum capacity of the impedance analyzer, 40 *MHz*.
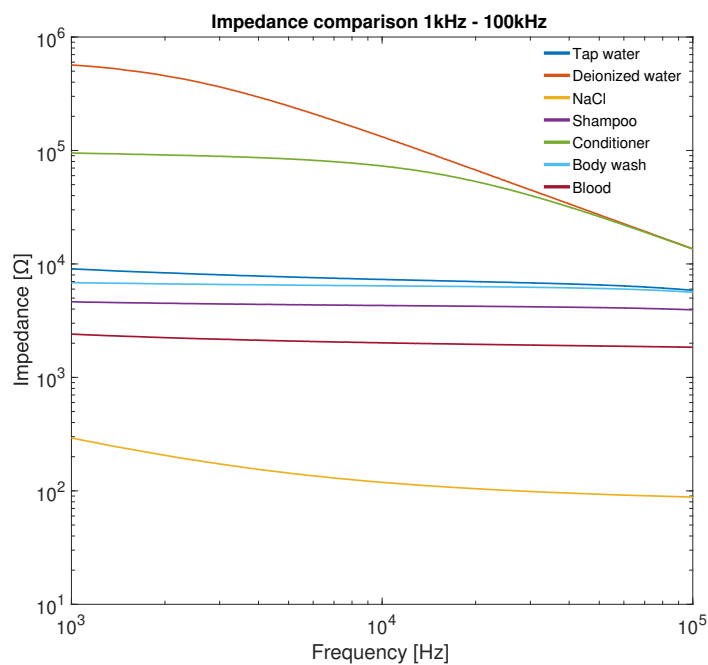
Figure 28: *Impedance spectrum from 1 kHz to 100 kHz for all test solutions except calcium. All the test solutions are of concentration B. The data presented are based on a mean value of five iterations of each contaminant.*
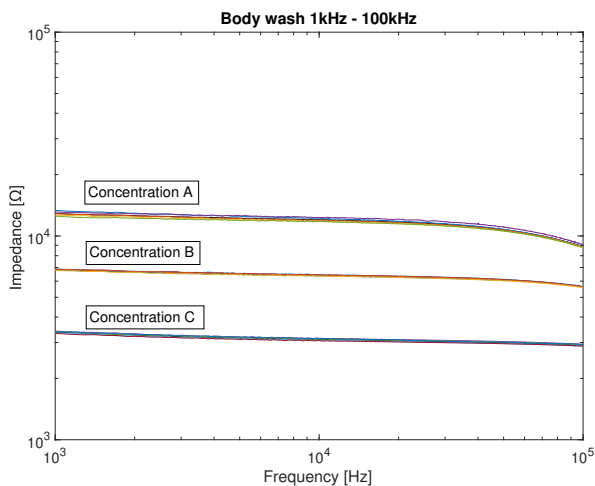
Figure 29: *Impedance spectrum from 1 kHz to 100 kHz for the three concentrations of body wash. Five iterations of each concentration.*
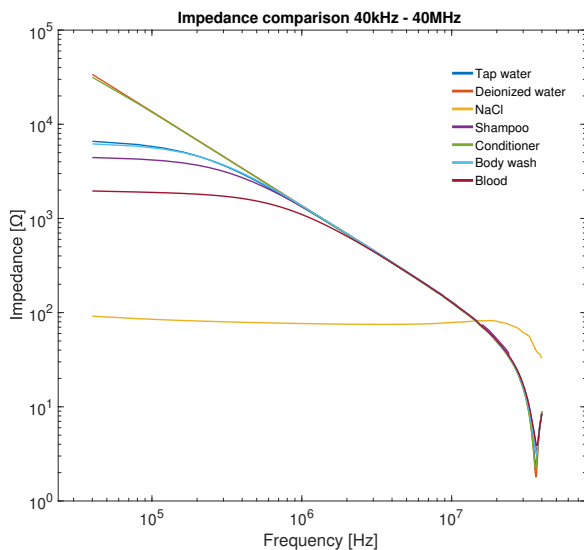
Figure 30: *Impedance spectrum from 40 kHz to 40 MHz for all test solutions except calcium. All the test solutions are of concentration B. The data presented are the mean values of five iterations of each contaminant.*

5   Discussion

5.1   Ultrasound

The contaminants shampoo, conditioner and NaCl were distinguishable from tap water in the still water measurements. *Figure 14*, for example, shows the sound waves moving faster in shampoo solution and slower in conditioner solution compared to tap water. These three contaminants were then again tested with circulated water. No difference compared to tap water could be seen in any analysis for either blood, calcium or body wash, regardless of concentration. These results are thus omitted.

The delta time values for the ToF analysis is an indicator of the precision of the sensor technique. One should note that the variation is very small compared to the whole signal. A delta time of 40 *ns* on a signal that is 24 *μs* long is equivalent to a percentage of 0.16% which is a small variation. A similar variation is obtained in all measurements apart from conditioner in circulating water where the delta time rises to 200 *ns* and above.

Foam created by body wash and shampoo greatly affected the results negatively. As stated in the *Results*, no measurements could be made for concentration B or C for neither of the two products. Foam is essentially trapped air which has completely different acoustic properties compared to water. The set up was designed to avoid foam by placing the ultrasound sensors parallel to the ground in the tube and trying to measure the water in the centre of the tube while having the foam at the top of the tube. This worked well for the lower concentrations, but the surfactants in concentration B and C created too much foam for any signal to be recorded. It should be pointed out that the effect the used Topsflo pump had regarding foam in the system was not fully investigated. It is possible that another pump would cause less foam but this is however just conjecture.

As previously stated, the ToF analysis of conditioner in circulating water could not distinguish the solution from tap water. However the FFT analysis showed better results as can be seen in *figure 19*. The amplitude of the 2 *MHz* frequency peak for all the conditioner measurements are noticeably lower than that of tap water. It can be argued that this decrease in amplitude is due to the particles in the conditioner generating a lower powered signal. These particles are further discussed later on in this section.

In order to make sure that there indeed was a change in frequency, a normalization of the values of both tap water and conditioner was made. The process, FFT peak to total signal ratio, is described in the method, and the results can be viewed in *figure 20*. The figure shows a difference between

tap water and conditioner. There is an overlap between the different concentrations of conditioner but none of the measurements overlap with tap water. From this, the conclusion can be drawn that for further investigation regarding conditioner, FFT is the analysis of choice ahead of ToF.

The best results were achieved measuring NaCl solutions, *figure 13* and *16*. An increasing concentration of NaCl resulted in a shorter ToF, not only in still water, but also in circulating water. This relationship was mentioned in literature and it is assuring that it can be seen in the *Results*. Limited literature supporting general detection of contaminants in liquid using ultrasound was found during the literature study of this thesis. However, the presented results show that detection of NaCl using ToF is possible. It is interesting to see that there is no overlap between the different concentrations of NaCl. This means that it is not only possible to differentiate NaCl solution from tap water, but also to detect differences in NaCl concentration. Hypothesising, it could then be possible to measure the NaCl concentration during a whole shower session and relate this to the hydration level of the user. This is however beyond the scope of this work and will not be speculated further. No conclusions can be drawn from the FFT analysis of NaCl, *figure 15* and *18*, which means that ToF is the most suitable analysis method for future work.

A limitation to the ultrasound measurements is the quality of the ultrasound sensors and the signal generator. As mentioned in method, the received signal could be different from iteration to iteration regarding if it started with a positive or negative half period. This complicated the data analysis. The appearance of the signal in frequency domain also varied. In *figure 15*, one clear peak at 2 *MHz* can be observed as for all the other frequency spectra. However this figure contains a second smaller peak at around 2.8 *MHz*. On the contrary, *figure 18* show an absence of this second peak. Two distinctive peaks are preferable as the peak values can be divided with each other to obtain a value without any dimension. If the contaminant differs from water on both peaks, then a quotient can show an even bigger difference. This was attempted on the ultrasound data, but the inconsistency of appearance made it very difficult. For future measurements, other equipment should be tested in order to see if a more repeatable signal can be achieved.

One vital factor that needs to be constant when measuring ToF is the distance between the transmitter and the receiver. The sensors were approximately 35 *mm* apart (no accurate distance measurement could be made once the sensors were integrated in the set up). For example, if the distance was changed by as little as 0.1 *mm*, then the ToF would change with 70 *ns*, which is a lot in the context of these measurements. Literature claims that the accuracy possible to achieve is better than 0.1 *m/s* [26], which over the given distance

of 35 *mm* corresponds to an accuracy of 1 *ns*. Hence the fixation of the sensor is of utmost importance. The sensors in this thesis were fastened with glue and there was no indication that they had loosened over time. However, with circulating water, there is water pressure in the system that has not been measured. The pressure had most likely not moved the sensors during testing in this work but it is a risk one should bear in mind for future experiments. Although, the sensors were moved when changing the set up from still to circulating water. Hence no comparison have been made between ToF of the two different scenarios. Two other possible sources of error mentioned in literature are turbulence and air bubbles [30, 31]. Multiple actions were taken in order to minimize these. An outlet for air was placed at the highest point of the test set up and the measurements were taken once no visual air bubbles were present in the system. Turbulence was minimized by having tubes with as small cross section area as possible in the setup and by placing the sensors far away from any curvature.

As can be seen i *figure 17*, the iterations of the conditioner seems to be divided into two groups. Both of the groups contain all three concentrations of the conditioner solution. This separation is simply due to the appearance of the received signal. The signals of the group to the left, which have the lower time of flight, are starting with a distinct negative half period that reaches the threshold. The received signals in the group to the right either start with a positive half period or with an indistinct negative half period whose amplitude does not reach the threshold. The fact that a half period of a 2 *MHz* signal is comparable to the difference in time, 220 *ns*, between the two sets, substantiates this claim. In this situation, the problem could indeed be solved by only using the first positive half period for triggering the threshold instead of using the absolute value. Throughout the signal processing, three different approaches regarding threshold were evaluated, negative amplitude, positive amplitude and the combination of an absolute value. The latter was found to give the most promising results in still water and was therefore continuously used during the analysis. Still, the variation within the two sets of iterations in *figure 17* is noticeably greater than that for still water. The reason for this increase is probably due to the fact that conditioner is not completely soluble in water. Visible particles can be seen in any conditioner sample regardless of the duration of sturing. These particles seemingly did not affect the delta times of the still water but disturbed the circulated water tests. This is not surprising as it already has been stated that air bubbles greatly affect the speed of water and the conclusion that visible particles should disturb signal could almost have been made without any tests.

Interesting parameters to investigate in future testing would be how a change in transmitted frequency or distance between the sensors would affect the results. Numerous studies [28, 41] have used similar distances and transmitting frequencies as this work for determining concentrations using FFT. It would however be interesting to examine whether an increased distance would result in a bigger change in frequency or ToF. An approach would for example be to place the sensors along a tube rather than across. The transmitted frequency would then have to be decreased as to be able to travel this increased distance, as mentioned in *Theory*, a decrease in frequency results in an increased depth penetration. Additionally, it would be interesting to analyze a number of echos of the received signal. One could then see if their frequency content changes more than the one analyzed in this work, that only traveled through the analyte once. One study [41] analyzed six repeated echoes and found increasing frequency difference with increasing number of echo. It would hence be interesting to see whether FFT of a third or fourth echo resulted in better opportunity of detection.

In summary, the opportunities and risks of using ultrasound from testing.

Opportunities:

- Shampoo, conditioner and NaCl were detectable

- Different concentrations of NaCl were distinguishable

- High precision in ToF measurements

- Interference due to air bubbles and turbulence was minimized

Risks:

- Blood, calcium and body wash were not detectable

- Foam from personal care products affected the results greatly, especially in circulating water

- The signal generator used did not generate a constant signal

5.2   IR

The most noticeable remark about the results from the IR tests is that all graphs have a negative gradient. This is due to heat generated by the LED affecting the photodiode. The photodiode was very sensitive to heat which caused a decrease in the measured signal over time. This drift was accounted for by taking measurements from the multimeter every 10 seconds during a minute and by turning of the LEDs to let the photodiode cool off between trials and concentrations. This sensitivity for temperature is not optimal, but since the scope of this work only includes evaluation of the techniques, rather than implementation of the sensors, it is sufficient to state that there is a problem and that it has been accounted for.

Out of the three different LEDs with wavelengths of 1200, 1300 and 1450 *nm*, only 1200 and 1300 *nm* gave presentable results. All tests made with the 1450 *nm* LED gave no or close to no signal at all. The LED generated a clear signal when tested in air but as soon as any contaminant mixed with water was tested, the signal disappeared. This is most likely due to the fact that at 1450 *nm*, water has a distinctive absorbance peak. This was acknowledged during the literature study but the magnitude of the peak was larger than expected. Therefore, a clear conclusion that can be drawn is that an IR sensor operating at a wavelength of 1450 *nm* is a poor choice when trying to detect contaminants in a mixture of water. Furthermore, one should be cautious when making comparisons between the magnitude of output for a given contaminant for the two different LEDs of 1200 and 1300 *nm*. Firstly the two LEDs have different optical power. At 50 *mA* the 1200 *nm* LED has an optical power of 5 *mW* while the 1300 *nm* LED only has 3.5 *mW*. Since the current applied to the LEDs has remained constant at 60 *mA*, the optical power has not been equal between the two LEDs. Secondly, the photodiode used does not respond equally to the different wavelengths, rather it is around 10% more responsive at 1300 *nm* than 1200 *nm* (the full emission spectra for the LEDs and the spectral response of the photodiode can be found in the *Appendix*). These two factors lead to the fact that comparisons between the output values of the LEDs should be kept to a minimum. Be that as it may, conclusions can still be made regarding how the different contaminants have deviated from tap water for the two wavelengths.

Promising results can be seen in *figure 21* and *22*, which shows a comparison between all the different contaminants for the two wavelengths 1200 and 1300 *nm*. Blood, conditioner and calcium are the contaminants most distinguishable from tap water. There is a small difference between tap water and shampoo, while there is close to no difference between tap water and body wash or sodium chloride. Blood differs around 150 *mV* from tap water at

1200 *nm* and around 60 *mV* at 1300 *nm*. A similar pattern can be seen for conditioner where it differs around 110 *mV* at 1200 *nm* and 55 *mV* at 1300 *nm*. Calcium on the other hand differs around 50 *mV* for both wavelengths.

Further, *figure 23* and *24* show all iterations for the three concentrations of different contaminants. It can be noted that there is no overlap between the concentrations in blood at 1200 *nm* or calcium for neither 1200 or 1300 *nm*. This is interesting as it indicates that the contaminants not only can be distinguished from tap water, but also that a future sensor might be able to draw conclusions regarding the concentration of the contaminant. This is particularly interesting with calcium as high levels in water can cause deposits in pipes. The fact that the different concentrations do not overlap demonstrates high precision of the technique. This is preferable as it limits the risk of outlying measurements that mistakenly can be detected as water. It further tells us that using the mean value to represent the different iterations is an acceptable approach. The graph in the lower left corner of *figure 24* tells us once again that the conditioner measurements had high variation. This is most likely due to the particles that arise in conditioner due to its poor solubility in water. This is already mentioned under the *Ultrasound* section and will not be discussed further.

The aim of the IR technique was to detect molecules by having them vibrate and absorb light at their characteristic frequency. However, the consideration has to be made whether it is vibration of the molecules that changes the output or if it is the loss of transparency in the solution. All of the three contaminants that show the greatest difference from water, i.e. blood, calcium and conditioner, make the water lose transparency. The effect is visible to the naked eye, but it is unclear if the haziness of the sample only exists in visible light or if it also is present for light in the IR spectra. This is something that can be further explored using an IR camera and try to observe if the haziness exists for IR wavelengths. Another approach would be to use a turbidity sensor and compare the results with an IR sensor. One could then investigate if the loss in visible light transparency is carried over to the IR spectra or not. An interesting observation is that the solutions shampoo and body wash also are more turbid than water. Despite this, those solutions gave poor results using IR. An example of this can be seen in *figure 24*. The difference in output between shampoo and tap water is smaller compared to the difference in output for the other contaminants showed in the same figure.

The next step would be to test the IR technique on circulating water. This was attempted but the setup was not optimal and the results were hence not reliable. The reason for the poor setup was time constraint. Sufficient time was set aside to do measurements on both still and circulating water but the

outbreak of Covid-19 delayed the delivery of the LEDs. The results from the tests made on circulating water are consequently not fully presented in this report but it should be said that they were in accordance with the results for still water and that the use of a IR sensor in a product such as the Orbital shower is very plausible. However, as stated in the beginning of this paragraph, this must be more extensively tested.

It should lastly be noted that the wavelengths used in these test have been selected based upon educated guesses supported by literature. The more optimal approach, mentioned in the *Theory*, is to perform a FTIR and from those results evaluate what wavelengths are most suitable for the detection of the contaminants in question. A FTIR will also make it possible to use higher wavelengths then possible using a LED. As literature suggest, many of the distinctive peaks for molecules found in personal care products are found at over 2500 *nm* [33] and using these wavelengths would be interesting even though a FTIR is next to impossible to integrate into a future product. For future work, an approach using FTIR is recommended.

In summary, the opportunities and risks of using IR from testing.

Opportunities:

- Shampoo, conditioner, blood and calcium were detectable

- Different concentrations of blood and calcium were distinguishable

Risks:

- The photodiod was sensitive to heat generated by the LEDs

- 1450 *nm* was totally absorbed by water

- Uncertainty whether the molecules absorb light or the transparency of the fluid limits the light penetration

5.3   Impedance

It was determined through discussions with supervisors that phase values were irrelevant for the purpose of this project as it mostly gives information regarding the system, such as the surface area of the electrodes exposed, rather than information about the solution being tested. Hence, focus was put on the magnitude or absolute value of the impedance and conclusions will be drawn from those results.

Before drawing conclusions from the impedance results, one should first note that the measurements taken during the first session were not as reliable as the results from the second session. The equipment was not very user friendly and it was difficult for novice users to see whether a measurement is faulty or not. As previously mentioned, the coaxial cable was sensitive and could cause incorrect measurements. An example of this can be seen in *figure 26*, which contains measurements made in NaCl solutions. Concentration B (yellow) has around three times higher impedance value than the other two concentrations. The fact that incorrect measurements could occur lead to greater caution and selectivity in the second session. In that session all the measurements were repeated five times to ensure repeatability. The improved caution is illustrated in *figure 28*, as all the different trials generated very similar results and there was no outlying values. In other words high precision was achieved. This made it possible to use mean values rather than medians of the trials when presenting the results.

Figure 25 shows a number of test solutions over a wide range of frequencies. As mentioned, the results from this first session are rather difficult to draw conclusions from. A disadvantage of measuring over a wide range of frequencies is that when the values are plotted on a logarithmic scale, the higher frequencies receive many more data points than the lower frequencies. This occurs since the analyzer measures a fixed amount of values with even increments. However, one can still see that the biggest differences between the test samples can be found between 1 *kHz* and 100 *kHz*. This particular frequency range was further examined in the second session of tests. *Figure 25* further shows that all test solutions, with the exception of NaCl, converge at frequencies over 100 *kHz*. The frequency range 40 *kHz* - 40 *MHz*, was thus further examined in order to see if the samples continued to converge and if the NaCl samples too followed the same pattern. It would have been interesting to examine even higher frequencies at *GHz* levels. This was not possible with the equipment available. *Figure 29* shows this second set of frequencies and a clear convergence of all tested samples, apart from the NaCl solution can be observed.

As mentioned in the *Theory* section, impedance is a measurement of both resistance and reactance. The NaCl solution had a big resistance entity due to the many ions in the solution. Resistance is not dependent on frequency, and hence the lack of decrease in impedance. The other solutions on the other hand had a larger reactance. This reactance is most probably close to pure capacitance. Pure capacitance has no resistance entity and the impedance value becomes:

$$Z = 1/j\omega C$$

This means that the impedance will decrease with increasing frequency, as all solutions apart from NaCl in *figure 29* do. The reason for the convergence was due to the fact that all solutions are similar to water, and at some point the chemical properties of water are too dominant for any difference between the solutions to be observed.

The sudden increase that can be seen in *figure 29* at around 30 *MHz* was most likely an insignificant resonance peak due to the machine rather than the chemical properties of the samples. This is supported by the fact that 30 *MHz* is very close to the 40 *MHz* maximum limit of the analyzer.

The most interesting conclusions can be drawn from *figure 27*. The graph shows the mean value of the five trials of concentration B (8 *g/l* for NaCl, 4 *g/l* for personal care products and 5% vol blood) of all contaminants together with deionized water and tap water. All solutions show differences in impedance values. An optimal detection is obtained at the frequency with the biggest difference between the contaminants and tap water. The biggest difference can be found in the left side of the figure between 1 - 2 *kHz*. Over this range, conditioner differ greatly from tap water. Shampoo and blood also differ significantly, while body wash is the contaminant most similar to tap water. If multiple impedance sensors can be used at once, then one sensor could measure at 1 *kHz* and another could measure at 100 *kHz*. 100 *kHz* is the area where the biggest difference can be seen between NaCl and tap water. This sensor could focus only on detecting salt which is frequent in fluids from the body, such as urine or sweat.

No conclusive results were obtained from the calcium measurements and were thus omitted from the report. One reason for the inconclusive results might be that the concentrations of calcium attempted to detect were much smaller than that of personal care products or NaCl. Another reason can be that calcium ions are worse at conducting electricity than Na^+ ions. This is supported by a study that claims that Na^+ ions have substantially higher conductivity than Ca^{2+} ions, due to their higher degree of dissociation, which is the ability for cathodes and anodes to separate in a solution [42]. It is much

more common in industry to use titration as a method to measure hardness of water and to achieve similar results with impedance seems unlikely.

One distinct difference between the solutions used in the impedance tests compared to the solutions used in the other tests is that these solutions were mixed in deionized water rather than tap water. This was done in order to ensure that the chemical properties of the contaminants were measured without the chemical properties of water being to dominant. A mixture with tap water would most probably converge at a lower frequency, compared to the contaminants in *figure 25*. On the other hand, the contaminant in a mixture of tap water is more similar to what a sensor in a product would be exposed to. In future work, it would be interesting to test all the contaminants mixed in tap water and compare. Similarly to the other techniques, it would be interesting to perform measurements on circulating water.

In summary, the opportunities and risks of using impedance from testing.

Opportunities:

- All contaminants apart from calcium could be differentiated from tap water

- High precision of measurements

Risks:

- Calcium was not detected

- The values can not be compared to that of a sensor that can be integrated into a product such as the Orbital shower

- Phase values only give information regarding the system

- The contaminants were mixed with deionized water rather than tap water

5.4   General discussion

In order to better be able to discuss the differences between the sensor techniques and to discuss what technique best detects certain contaminants, *table 10* below has been created. A score ranging from one to five was given to the different techniques for each contaminant. A score of one represents that no detection was possible, three represents that detection was possible and five represents that not only detection of the contaminant was possible, but clear differences could be seen between the concentrations. It should be emphasized that these scores are estimated by the authors of this thesis and not scientifically calculated.

	Ultrasound	*IR*	*Impedance*
Shampoo	3	3	3
Body wash	1	2	3
Conditioner	3	4	3
Sodium chloride	5	1	5
Blood	1	5	4
Calcium	1	5	1
Total	14	20	19

Table 10: *Score table demonstrating the performance of each technique. A score of one means that the contaminant is not detectable using the technique whilst a five means that the contaminant is both detectable and a difference between its concentrations could be observed.*

Although, the concentrations were kept constant for all techniques, there were some parameters that varied. For instance, the impedance analyzer was a big piece of very accurate and expensive equipment. It can not be implemented in a product such as the Orbital shower.

Rather, a smaller less accurate sensor measuring impedance at one particular frequency, is possible to integrate in the product. It is thus not fair to compare the far more advanced analyzer to the IR or ultrasound sensors tested. In addition, the impedance analyzer measured over a large range of frequencies, while the IR LEDs and ultrasound sensors measured at one frequency each. A more optimal comparison would be to compare the results of a impedance analyzer to a FTIR. These inequalities were regarded when assigning the scores. Furthermore, since measurements on circulating water only was carried out for the ultrasound technique, results on still water were analyzed when deciding scores.

Before conclusions are drawn from *table 10*, a few examples of reasoning behind the scores should be given. For example, NaCl for ultrasound analyzed with ToF, *figure 13*, shows clear differences between both the different concentrations and from tap water, hence the score five. Conditioner with the IR technique was instead given a four. This was due to the fact that it could be distinguished from tap water, but the output value between the different concentrations overlapped, *figure 24*. The contaminants blood and calcium was given scores of five as their concentrations did not overlap. The attempt of detecting body wash using impedance was given a three. The reasoning behind is that its impedance is very close to water, but clear differences between concentrations can be observed in *figure 28*.

The total score for the techniques in the bottom of *table 10* are interesting to analyze. One can see that IR and impedance are both better than ultrasound. The only contaminant that ultrasound can detect well is NaCl which also is easily detected using impedance. The combination of an IR and an impedance sensor is compelling to hypothesize about. The techniques would complement each other well as one can detect a contaminant that the other would completely fail to detect. For instance, NaCl can be detected with impedance and a possible calcium contamination can be noticed by an IR sensor. Both techniques are equally capable of detecting personal care products, whereas limited information can be found regarding their concentration. As mentioned in the *Theory* of IR, personal care products are preferably measured using wavelengths above that of the near IR spectra. It would be intriguing if the problems of using such an IR sensor could be overcome. A sensor system using multiple impedance sensors and multiple IR sensors where one can measure above 2500 *nm*, would presumably not only be able to detect, but to some extent quantify all contaminants in this thesis. It is hence recommended that future research is made on the combination of the techniques IR and impedance as means of detecting contaminants.

A frequently disturbing factor during the measurements was foam created by the surfactants in personal care products. The greatest amount could be seen in measurements of shampoo, a significant amount could be seen when measuring body wash and hardly any for conditioner. All of the evaluated techniques are used to measure liquids rather than air. The sudden change of medium from water to air greatly affected the results. Foam caused the complete absence of a signal when using ultrasound especially in circulating water. It should be acknowledged that an absence of a signal is a form of detection. If foam is a direct consequence of the presence of personal care products in water than an absent signal can be viewed as a detection. Nevertheless, a very small amount of foam will cause an absence of a signal in ultrasound, which means that water otherwise deemed uncontaminated and

acceptable to be recycled, could be discarded. The effect foam has on the sensor techniques and whether is can be used as a mean of detection is something that needs to be investigated more before any real conclusions can be drawn.

In future work, deeper investigation of the effects that circulating water can have on the techniques, is needed. It has been observed that a greater amount of foam was present in the system when testing all personal care product and for shampoo in particular. The effects this would have on impedance and IR are yet to be investigated. Circulating water will most likely also result in a more noisy signal. This might limit the information that can be obtained about the concentration of the contaminant, but detection should still be possible.

There exist a number of minor limitations to this work that briefly need to be mentioned. Firstly, only one type of product was used as representation for shampoo, body wash and conditioner. One of the requirements when choosing the product for the tests was that they were supposed to contain ingredients frequently found in those product. This requirement was applied, but even so it is not possible for one product to represent all when there is a such big variety of brands and content in the products. Secondly, the cattle blood used differs from human blood in a number of ways, the optimal, still reasonable, choice would be pig blood as it is more similar to human blood, but this was not obtainable. Lastly, as mentioned, tap water parameters differ greatly depending on location and time. The Orbital shower accounts for this by calibrating all of its sensors in the beginning of a shower cycle. Such a calibration was not possible for the tests carried out in this work. The parameters were kept as constant as possible, for example by filling up a large water container in the beginning of each test session. The risk of variation is nevertheless something that one needs to bear in mind.

An interesting aspect not covered by the tests in this report is what the simultaneous occurrence of two different contaminants would do with a sensor system. For instance, an impedance sensor could be exposed to a mixture of conditioner, that have higher impedance than tap water, and NaCl that have lower impedance than tap water. Theoretically, a mixture of these two could cancel each other out and be detected as water. A possible solution to this is to use another sensor technique where the different contaminants are not polar opposites of each other. A mixture of contaminants could then be mistaken as water by one sensor but detected by another. This is another strong argument for the use of a combination of sensor techniques rather than one sole type of sensor.

Temperature is a cause of big variation in sensor measurements. The temperature was attempted to be kept as close to constant as possible but in a real shower temperature differences that might affect the sensors can occur. To compensate a sensor for temperature is however something common. It is therefor highly unlikely that this should hinder any sensors presented in this thesis to be integrated into a product such as the Orbital shower.

6   Conclusion

A wide range of techniques potentially suitable for detection of contaminants in greywater were investigated through literature study. Based on theoretical potential and practical feasibility, the most promising techniques; ultrasound, IR and impedance, have been further analyzed and practically tested for a number of contaminants.

All three techniques were, with individual variation, able to detect some contaminants and failed to detect others. The range of contaminants detected using IR and impedance complement each other the best and thus a combination of the two techniques is preferable. Technical parameters regarding each technique have been discussed in order to optimize detection.

For future work, more extensive studies about circulating water need to be carried out. The results of this thesis can give a good sense of direction of what sensor technique to use, but further investigation is essential before any sensors can be implemented into any product such as the Orbital shower.

References

[1] Jorgensen, S. E., Fath, B. D. (2014). Encyclopedia of ecology. Newnes.

[2] Hybrid device for streamlining water consumption, Mehrdad Mahdjoubi, School of Industrial Design - Lund University (unpublished)

[3] Amjad, Z., & Demadis, K. D. (Eds.). (2015). Mineral scales and deposits: scientific and technological approaches. Elsevier.

[4] Eriksson, E., Auffarth, K., Henze, M., & Ledin, A. (2002). Characteristics of grey wastewater. Urban water, 4(1), 85-104

[5] VASYDs website. https://www.vasyd.se/Artiklar/Dricksvatten/Hart-och-mjukt-vatten. Accessed: 26/03-20

[6] Engebretsen, K. A., Bager, P., Wohlfahrt, J., Skov, L., Zachariae, C., Andersen, A. M. N., ... Thyssen, J. P. (2017). Prevalence of atopic dermatitis in infants by domestic water hardness and season of birth: cohort study. Journal of Allergy and Clinical Immunology, 139(5), 1568-1574

[7] Undersökning, S. G. (2013). Bedömningsgrunder för grundvatten. Uppsala: Sveriges geologiska undersökning.

[8] Schock, M. R., Neff, C. H. (1988). Trace metal contamination from brass fittings. Journal-American Water Works Association, 80(11), 47-56.

[9] Zhang, Y. (2009). Dezincification and Brass Lead Leaching in Premise Plumbing Systems: Effects of Alloy, Physical Conditions and Water Chemistry (Doctoral dissertation, Virginia Tech).

[10] Roy, S. (2018). Interplay of Water Chemistry and Entrained Particulates in Erosion Corrosion of Copper and Nonleaded Alloys in Potable Water Systems (Doctoral dissertation, Virginia Tech).

[11] Grayman, W. M., Rossman, L. A., Deininger, R. A., Smith, C. D., Arnold, C. N., Smith, J. F. (2004). Mixing and aging of water in distribution system storage facilities. Journal-American Water Works Association, 96(9), 70-80.

[12] Widmaier, E. P., Raff, H., Strang, K. T., Vander, A. J. (2008). Vander's Human physiology: the mechanisms of body function. Boston: McGraw-Hill Higher Education.

[13] Costill, D. L. (1977). Sweating: its composition and effects on body fluids. Annals of the New York Academy of Sciences, 301(1), 160-174.

[14] Loeffler, J. M. (2010). The golden fountain–is urine the miracle drug no one told you about?

[15] Humphrey, S. P., Williamson, R. T. (2001). A review of saliva: normal composition, flow, and function. The Journal of prosthetic dentistry, 85(2), 162-169.

[16] Shirreffs, S. M., Maughan, R. J. (1997). Whole body sweat collection in humans: an improved method with preliminary data on electrolyte content. Journal of Applied Physiology, 82(1), 336-341.

[17] Baker, L. B. (2017). Sweating rate and sweat sodium concentration in athletes: a review of methodology and intra/interindividual variability. Sports Medicine, 47(1), 111-128.

[18] Roberts, S. B., Lucas, A. (1985). Measurement of urinary constituents and output using disposable napkins. Archives of disease in childhood, 60(11), 1021-1024.

[19] Putnam, D. F. (1971). Composition and concentrative properties of human urine.

[20] Sakamoto, K., Lochhead, R., Maibach, H., Yamashita, Y. (Eds.). (2017). Cosmetic science and technology: theoretical principles and applications. Elsevier.

[21] D'Souza, P., Rathi, S. K. (2015). Shampoo and conditioners: What a dermatologist should know?. Indian journal of dermatology, 60(3), 248.

[22] Miteva, M. I. (2018). Alopecia. Elsevier Health Sciences.

[23] Draelos, Z. D. (Ed.). (2015). Cosmetic dermatology: products and procedures. John Wiley Sons.

[24] Abu-Zidan, F. M., Hefny, A. F., Corr, P. (2011). Clinical ultrasound physics. Journal of Emergencies, Trauma and Shock, 4(4), 501.

[25] Henning, B., Rautenberg, J. (2006). Process monitoring using ultrasonic sensor systems. Ultrasonics, 44, e1395-e1399.

[26] Hauptmann, P., Lucklum, R., Püttmer, A., Henning, B. (1998). Ultrasonic sensors for process monitoring and chemical analysis: state-of-the-art and trends. Sensors and Actuators A: Physical, 67(1-3), 32-48

[27] Chen, C. T., Chen, L. S., Millero, F. J. (1978). Speed of sound in NaCl, MgCl2, Na2SO4, and MgSO4 aqueous solutions as functions of concentration, temperature, and pressure. The Journal of the Acoustical Society of America, 63(6), 1795-1800.

[28] Chen, A. Q., Freear, S., Cowell, D. M. J. (2007). Measurement of solid in liquid content using ultrasound attenuation. In 5th world congress on industrial process tomography, Bergen, Norway (pp. 820-826).

[29] Weber, F. J. (2004). Ultrasonic beam propagation in turbulent flow.

[30] Strunz, T., Wiest, A., Fleury, A., Fröhlich, T. (2004, July). Influence of turbulence on ultrasonic flow measurements. In 5th IGHEM Conference.

[31] Yildiz, A. (1988). Attenuation of sound waves due to scattering from bubbles and liquid drops. Journal of Sound Vibration, 121, 127-133.

[32] Berthomieu, C., Hienerwadel, R. (2009). Fourier transform infrared (FTIR) spectroscopy. Photosynthesis research, 101(2-3), 157-170.

[33] Salvador, A., Chisvert, A. (Eds.). (2011). Analysis of cosmetic products. Elsevier.

[34] Sze, S. M., Ng, K. K. (2006). Physics of semiconductor devices. John wiley sons.

[35] Madejová, J., Sekeráková, Ľ., Bizovská, V., Slaný, M., Jankovič, Ľ. (2016). Near-infrared spectroscopy as an effective tool for monitoring the conformation of alkylammonium surfactants in montmorillonite interlayers. Vibrational Spectroscopy, 84, 44-52.

[36] Haroon, K., Arafeh, A., Martin, P., Rodgers, T., Mendoza, Ć., Baker, M. (2019). Use of inline near-infrared spectroscopy to predict the viscosity of shampoo using multivariate analysis. International journal of cosmetic science, 41(4), 346-356.

[37] Peters, R. D., Noble, S. D. (2019). Using near infrared measurements to evaluate NaCl and KCl in water. Journal of Near Infrared Spectroscopy, 27(2), 147-155.

[38] Laub-Ekgreen, M. H., Martinez-Lopez, B., Jessen, F., Skov, T. (2018). Non-destructive measurement of salt using NIR spectroscopy in the herring marinating process. LWT, 97, 610-616.

[39] Sjöberg, D., Gustafsson, M. (2007). Kretsteori, ellära och elektronik. Elektro- och informationsteknik.

[40] Khan, L. B., Read, H. M., Ritchie, S. R., Proft, T. (2017). Artificial urine for teaching urinalysis concepts and diagnosis of urinary tract infection in the medical microbiology laboratory. Journal of microbiology biology education, 18(2).

[41] Muhammed Sabri, S., Fareq, M., Malek, A., Naseer, S., Noaman, M. N., Khairudi, M. J., ... Talib, A. (2013). A new ultrasonic exponential decay pulser technique for low concentrations detection and measurements.

[42] Levy, R., Katchalsky, A. (1973). Electrical conductivity of sodium and calcium forms of the synthetic inorganic exchanger zeolite type A. Journal of Colloid and Interface Science, 42(2), 366-371.

7 Appendices

7.1 Test solutions

Deionized water
Kemetyl T-Batterivatten
Maximum conductivity of 5 $\mu S/cm$

Shampoo
Barnängen Normal Shampoo

Ingredients: *Aqua, Sodium Laureth Sulfate, Cocamidopropyl Betaine, Sodium Chloride, Hydrolyzed Soy Protein, Niacinamide, Magnesium Chloride, Panthenol, Simmondsia Chinensis Seed Oil, Disodium Cocoamphodiacetate, PEG-7 Glyceryl Cocoate, Sodium Benzoate, Citric Acid, Cocamide MEA, Glycol Distearate, Parfum, Polyquaternium-10, Laureth-4, Hydrogenated Castor Oil, PEG-40 Hydrogenated Castor Oil, Hexyl Cinnamal, Propylene Glycol, Limonene, Geraniol*

Body wash
Neutral shower gel

Ingredients: *Aqua, Sodium Laureth Sulfate, Cocamidopropyl Betaine, Glycerin, Sodium chloride, Sodium Cocoyl Glutamate, Citric acid, Sodium benzoate, Sorbic acid*

Conditioner
Garnier Fructis

Ingredients: *Aqua / Water, Cetearyl Alcohol, Behentrimonium Chloride, Starch Acetate, Niacinamide, Saccharum Officinarum Extract / Sugar Cane Extract, Hydroxyethylcellulose, Hydroxypropyltrimonium Lemon Protein, Phenoxyethanol, Chlorhexidine Digluconate, Limonene, Camellia Sinensis Leaf Extract, Linalool, Benzyl Alcohol, Benzyl Salicylate, Isopropyl Alcohol, Pyrus Malus Fruit Extract / Apple Fruit Extract, Pyridoxine Hcl, Geraniol, Cetyl Esters, Citric Acid, Citronellol, Citrus Limon Fruit Extract / Lemon Fruit Extract, Citrus Grandis Peel Oil / Grapefruit Peel Oil, Citrus Limon Peel Extract / Lemon Peel Extract, Leuconostoc/Radish Root Ferment FILtrate, Hexyl Cinnamal, Parfum / Fragrance*

Sodium chloride
Falksalt finegrained without iodine

Ingredients: *NaCl 99,8 %, lump preventants (E535/E536)*
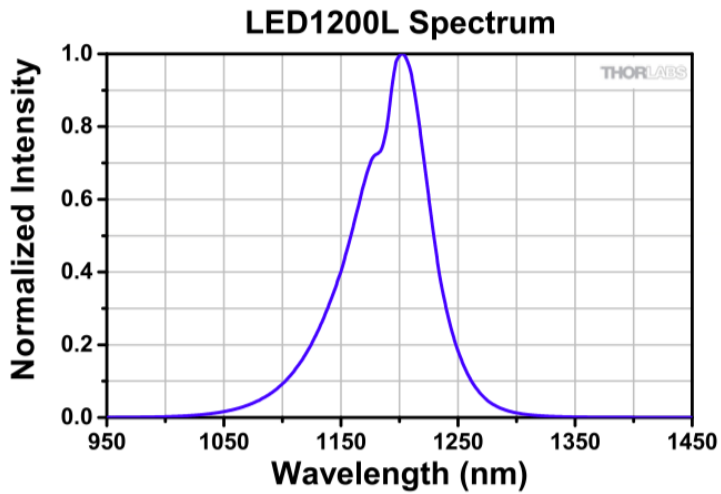

Blood
Cattle blood

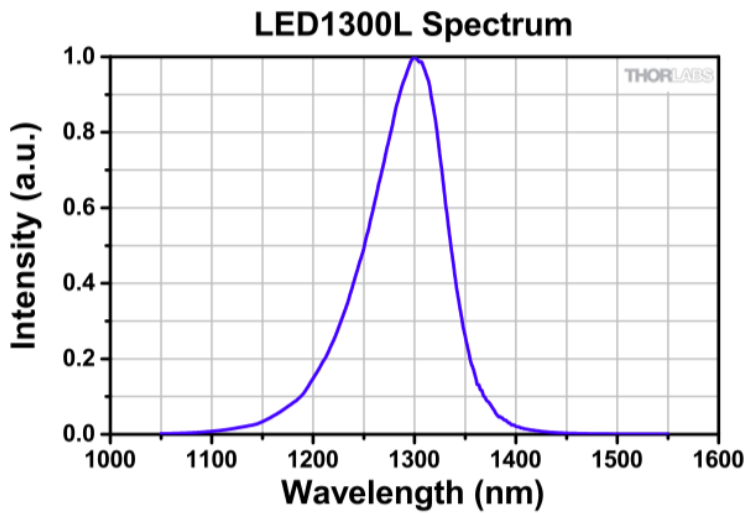**Ingredients:** *Cattle blood, antioxidant E331*

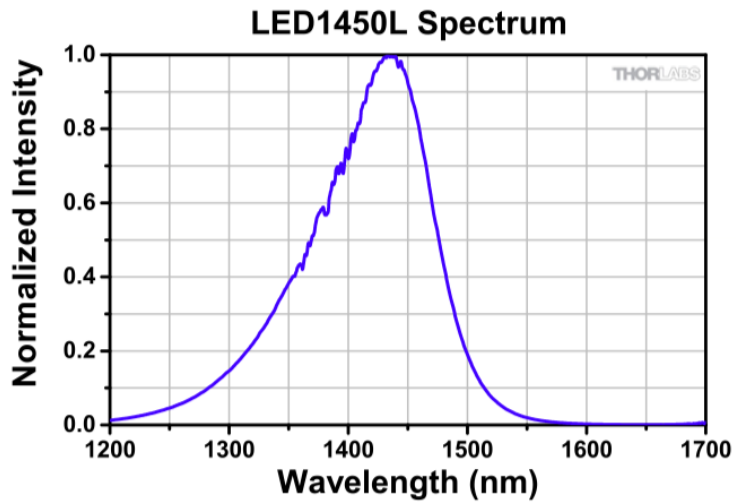## Calcium
Calciumcarbonate

Ingredients: *$CACO_3$, E170*

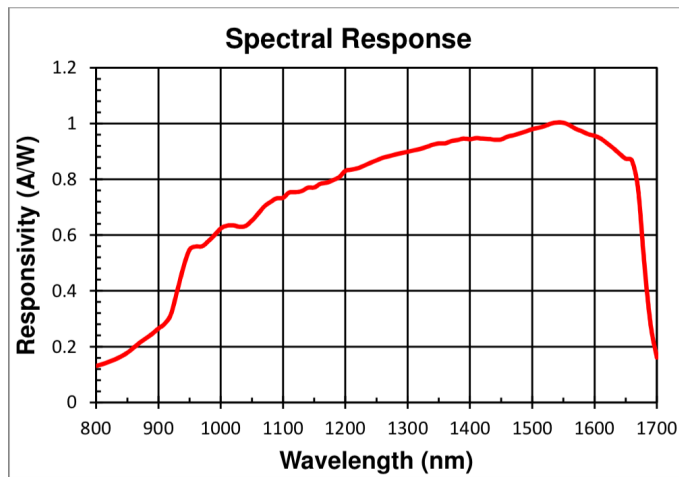## 7.2 IR components

Figure 31: *Normalized intensity. LED 1200 nm.*

Figure 32: *Normalized intensity. LED 1300 nm.*

Figure 33: *Normalized intensity. LED 1450 nm.*

Figure 34: *Spectral response of the photodiode.*