



Assessing Infiltration and Exfiltration on the Performance of Urban Sewer Systems

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Standard Operation Procedure (SOP):

Quantification of Exfiltration from Sewers with artificial Tracers: Continuous dosing (QUEST-C)

Material, Methods and Data Analysis

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Standard Operation Procedure (SOP):**Quantification of Exfiltration from Sewers
with artificial Tracers: Continuous dosing (QUEST-C)****Material + Methods
and
Data Analysis**

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1. General Principles

Some background information is given on the basic principles of the tracer method. This version of the Standard Operation Procedure (SOP) is considered as a practical guide that should enable end users to perform exfiltration measurements with Li^+ and Br^- as ionic tracers.

1.1. Introduction

The watertightness of a drainage system is explicitly mentioned as a basic performance requirement in the EU standard EN 752-2. Exfiltration of wastewater is critical because of its negative impact on soil and groundwater. Although the recent belief was that the self-cleaning capacity of the soil is sufficient to degrade pollutants from eventually occurring seepage, cases are reported where exfiltration has led to environmental pollution, health hazards and even death Bishop et al., 1998.

The following description gives a compendium for the quantification of exfiltration from sewers with Li^+ and Br^- as tracers. It contains information about the method itself, the planing process, the realisation of the experiments and the analysis of the data. When necessary, references are given to provide access to further information.

The method is suited to quantify the exfiltration from long sewer pipes under operation. It is aimed at sewer sections of several hundred meters and it is in this form not applicable to house connections. The method is not intended to replace standard testing procedures for watertightness or CCTV observations (Rieckermann J. and Gujer W., 2002).

The method described below is subject to a PhD thesis at EAWAG and has not been fully developed at this stage of the project.

1.2. Terminology

General terms

QUEST-C - Quantification of Exfiltration from Sewers with artificial Tracers: Continuous Dosing Procedure

exfiltration – losses of wastewater from a leaky sewer pipe.

tracer – a chemical substance that labels the wastewater and is monitored for exfiltration measurement. Ideally behaves conservatively.

Investigation reach – sewer section that is tested for exfiltration.

Sampling cross section – point where sampling pump is installed

Indicator tracer – Tracer that is continuously dosed upstream of the investigation reach. The amount of indicator tracer is reduced by exfiltration and indicates whether exfiltration occurs or not.

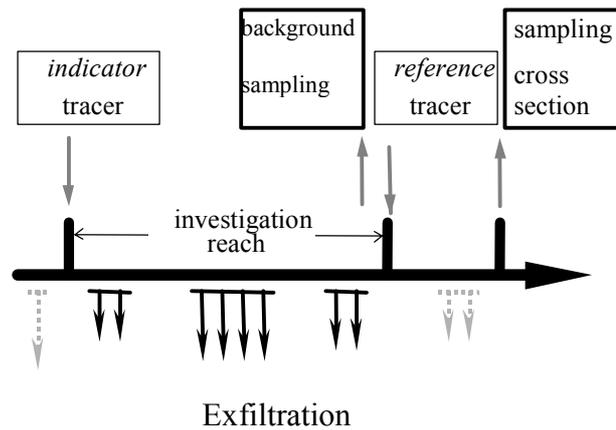


Figure 1 Conceptual sketch of the QUEST-C method to quantify exfiltration

Reference tracer - Tracer that is continuously dosed at the end of the investigation reach. As it is not influenced by exfiltration in the section of interest, it is used as a reference to which the indicator signal is compared.

background sampling – continuous sampling in order to identify the background concentration of the *reference tracer* in the wastewater.

laboratory standards – tracer solutions for each substance that is used in the experiment which is prepared in the laboratory before the experiment

field standard – mixture of LiCl tracer solution and NaBr tracer solution which is obtained in the field after the last sampling. The mixture is obtained by pumping tracer solution from both dosing pumps into the same receptacle under field conditions. This serves as a reference for the real ratio of Br^-/Li^+ dosed during the individual experiment.

1.3. Principles of the QUEST - C method

The basic idea of the QUEST – C method is that exfiltration measurement is feasible by a mass balance of a tracer substance over the desired sewer line. At the beginning of the *investigation reach* a known constant mass flow of tracer is continuously dosed which is defined as the *indicator* tracer (Figure 1). This tracer is passed through the sewer line and if exfiltration occurs, tracer substance is lost with the seeping wastewater Rieckermann et al., 2005.

One of the advantages of the QUEST-C method is that no inline measurement device is needed. Samples can be taken like a) discrete samples and b) time-proportional composite samples with a sampling pump. We recommend time-proportional composite sampling because of the variation of discharge during the sampling period, which causes a variation of the ratio of tracers. Therefore, also flow rate measurement is recommended during the experiment.

1.4. Basic requirements for exfiltration measurements with tracers

The basic requirements and desirable conditions for QUEST-C measurements are: (a) good accessibility to the sewers of interest (b) sufficient and continuous discharge in the

investigation reach (condition is valid also for additional section inflows), (b2) steady flow condition, (c) simple boundary conditions for both Li^+ and Br^- tracer measurements (no or little industrial wastewater flow and a minimal additional wastewater inflow on the investigation length), (d) zero or constant background concentration of both tracers and (e) adequate laboratory equipment for specific ions analysis, equipment for flow rate measurements, etc. that can operate under sewer conditions.

Critical aspects concerning the use of ionic tracers Li^+ , Br^- with dilution method

One possible disadvantage for the application of the tracer dilution method with ionic tracer is any *background concentration* that already exists in the wastewater. To minimise this problem, a background sampling procedure was implemented. In most cases it is only possible to sample the *reference* tracer, as the inflow into the investigation reach cannot be controlled and time synchronisation of the fluid elements between *indicator injection point* and *sampling cross section* is questionable. Therefore, the tracer with the higher variation in the natural background is chosen as the reference tracer.

Under normal circumstances, the bromide content of most drinking waters is small, seldom exceeding 1 mg/L. Natural origins of bromide comes are connate waters and sea waters (intrusion into aquifers or spray-affected precipitation). Industrial brine discharges may also contribute to bromide in water sources.

A minor constituent of minerals, lithium is present in fresh waters in concentrations below 0.2 mg/L. The use of the lithium or its salts in dehumidifying units, medicinal waters, metallurgical processes, and the manufacture of some types of glass and storage batteries may contribute to its presence in waters.

In area of Zurich (Ruemlang, Duebendorf, Opfikon) several wastewater samples were analysed and background concentrations were found to be under the detection limit for Li^+ . For Br^- values up to 0.4 mg/L were measured. Based on this results Lithium was used as *indicator tracer* and Bromide as *reference* tracer.

Because of the local differences of the ionic tracers (Li^+ , Br^-) background concentrations, preliminary investigation at the individual experimental location are recommended.

A second problem is the *instability of the flow during experiment*. The constant-rate injection method depends on steady flow conditions during the experiment. However, sewer flow is never steady and this behaviour introduces an error into QUEST-C measurements. Firstly, average sampling is recommended to minimise this error. Dosing and sampling times should not be less than 90 minutes.

Secondly, it is recommended to identify quasi-steady flow conditions at the measuring cross section during the day (e.g. 10:00-12:00, 13:00-16:00, 03:00-05:00). To have adequate information on the flow pattern, preliminary flow measurements should be performed.

2. Devices and equipment

In this chapter the main devices that are needed for the experiment are explained. Checklists with all the more general material that has been used can be found in Appendix C.

2.1. Equipment for dosing

The basic dosing set-up is shown in figure 2-1. The tracer solution is pumped from its container directly to the sewer with a pump trough flexible tube. The container is placed

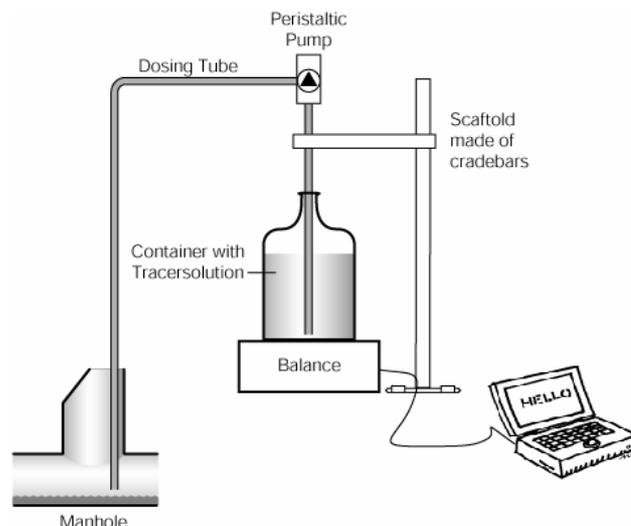


Figure 2 Schematic representation of dosing station with pump, container, balance and data logging system

directly onto the balance. The use of a PC (data logger) that allows a continuous recording of the weight is recommended. To protect balance and container from the influence of the weather (wind, rain) it is recommended to place the whole set-up into a closed box (see figure 3)

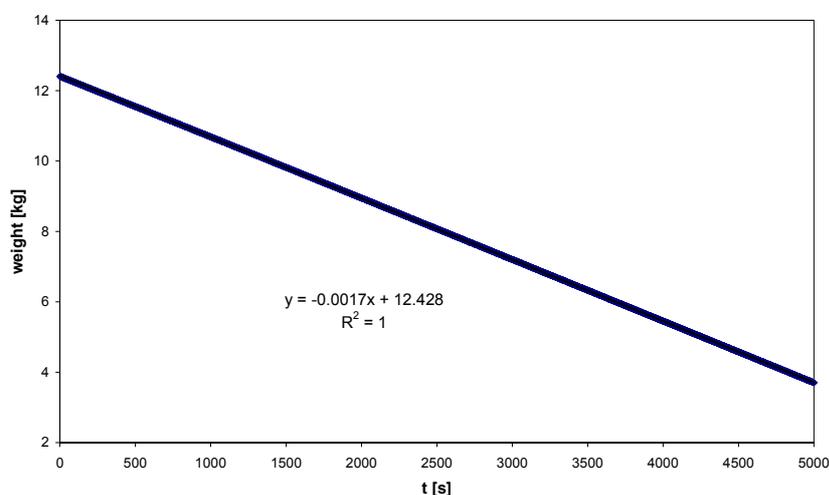


Figure 3 Example of the test result of dosing setup with ISMATEC BVK

The most important property of the dosing pump is a constant pumping rate during the whole experiment. To check accurately the behaviour of the pump a preliminary testing of the set-up should be done in the laboratory. We obtained the best dosing rates with multi-head multi-channel peristaltic pumps (e.g. ISMATEC BVK). The technical specification of the tested dosing pumps are given in Annex B.

At the injection point of reference tracer is necessary to combine dosing of reference tracer with background sampling. This modified set-up is shown in Figure 4.

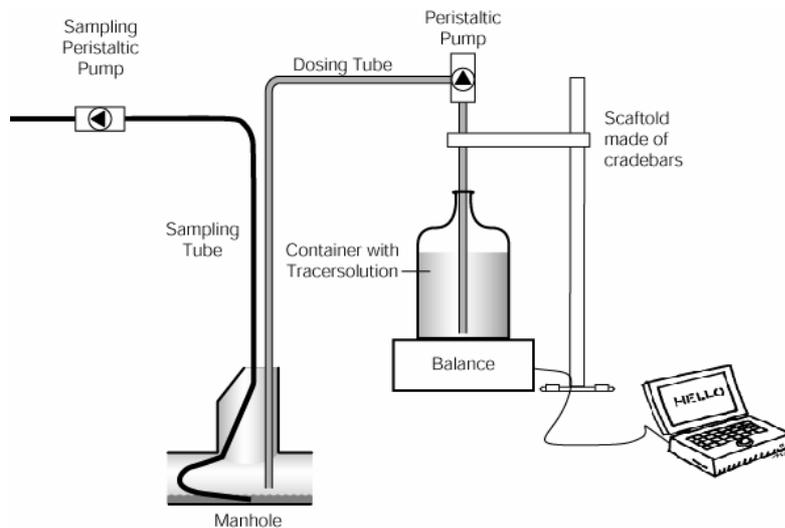


Figure 4 Schematic representation of dosing station with pump, container, balance, data logging system and sampling apparatus

2.2. Equipment for sampling

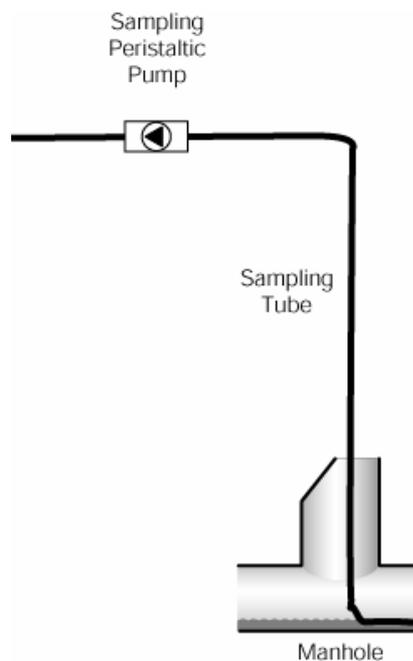


Figure 5 Schematic representation of sampling station with pump

The QUEST-C method is based on continuous sampling of background samples and samples that contain both tracer substances. This aims at minimising the errors due to a varying discharge. Another advantage of the continuous sampling is an easier operational procedure compared to the QUEST method using slug injections.

The most critical aspect is clogging of the sampling equipment (tubes and pumps). Gross sewer solids (toilet papers, food etc.) can immediately block the sampling tube inlet. To avoid this, the optimisation of the following set-up parameters should be considered:

- It is recommended to use low speed multi-channel peristaltic pump with a pumping rate of about 60 ml/min. If possible, the pumps should be placed outside the sewer (explosion safety). It is favourable to use at least 2 channels for sampling (2 separate tubes). A soft, flexible and smooth PVC material (e.g. TYGON) with an inner diameter of about 5 mm should be used. Fixing the tubes in the sampling manhole ca. 1 m above water surface showed an optimal performance. The end of the tubes, approximately 1-2 m, have to leisurely float in the stream. Best results were obtained with a tube made of one single piece without any junctions.
- The combination of the suitable pump rate and diameter of the tube is important because of the inlet inflow velocity. The sampling set-up should be tested before the experiment, preferably at the sampling site of interest. A clogging-free performance must be guaranteed during several hours.
- Typical sampling flasks have a volume from 0.5 to 1 liter, depending on the pump rate and duration of sampling. For easier filtration it should have broad neck, such that wastewater can directly be taken up with a syringe. For the filtrated samples, 50 ml flasks are sufficient, because about 30 ml of filtrate is needed for further analysis. In general, the material of the flasks should be chosen carefully to avoid contamination of the samples or adsorption to the receptacles. We recommend to clean the receptacles before the experiment with a solution of HNO₃ (see section 6.4).

2.3. Equipment for discharge measurement

To avoid gross errors due to rapid variations in the discharge an additional flow measurement is recommended. This should ideally have a high temporal resolution in the order of seconds. The device that has been tested in combination with the dilution method is the AMERICAN SIGMA 950 Flow Meter (American Sigma Inc., Medina, NY, USA). The device measures peak velocities with the *Ultrasonic Doppler Method* and water depth with an air bubbler. Data can be obtained with a minimum resolution of 2 seconds via RS 232. It is flexible in handling and does not influence the sewer flow significantly. For preliminary discharge measurements a time resolution of one minute is regarded sufficient.

Apart from these methods any continuous mobile discharge measurement which offers data in a high temporal resolution can be used (weir, etc.).

2.4. Equipment for field filtration

To avoid the absorption of tracers to suspended solids and flask material the samples should be immediately filtrated in the field. In addition, filtration is required for ion chromatography (IC) analysis in the laboratory.

For filtration syringes, plastic filtration modules, filter papers and tweezers are needed. In our experiments membrane filter papers with a pore size of 0.45 µm and diameter of 47 mm were used (e.g. Schleicher & Schuell, Sartorius). During bad weather condition, filtration can be done in laboratory. Nevertheless, it should be done immediately after the return from the field site.

2.5. Equipment for sample analysis

For the analysis of cations and anions it is recommended to use the IC method. The main advantage of IC method is the possibility to analyse both cations (Li^+) and anions (Br^-) in one run of the ion chromatograph. However, the instrument has to be equipped with two columns (cation column and anion column).

The ICP (Inductively Coupled Plasma) method for the analysis of lithium was also tested at EAWAG. However, in our laboratory, the results were strongly affected by the wastewater matrix and the concentrations were overestimated.

A more detailed description of the used Ion Chromatograph (Metrohm) is given in Annex C.

3. Chemicals and reagents

The chemicals that are used as tracer are Lithium Chloride and Sodium Bromide which are dissolved in water. Both chemicals used in the experiments were standard granular salts in industrial quality. Since a solution with high concentrations of both LiCl and NaBr causes density effects, it is not recommended to use concentrations above half of the solubility limit (LiCl: 415 g/L, NaBr: 240 g/L). Producers and retailers of the chemical are listed in section B.3.

3.1. Toxicology of lithium

Lithium is mixed (alloyed) with aluminium and magnesium for light-weight alloys, and is also used in batteries, some greases, some glasses, and in medicine. Concentration limits of Li^+ in the drinking water are 5 mg/L as a short-term maximum persisting for not more than 1 week and 0.5 mg/L as long term maximum. Experience has shown that in tracing experiments with lithium salts these requirements are satisfied without any difficulty.

As it is mentioned in the report of a working group at the German Federal Environmental Agency (Umweltbundesamt – UBA), there are “indications that lithium chloride is ecologically unsafe, which, however, could not be clarified conclusively by the Working Group. Therefore, in tracing experiments, lithium concentrations in resurfaced groundwater and in surface water should be kept as low as possible.” Behrens et al., 2001

3.2. Toxicology of bromide

Bromide has a low acute and chronic toxicity to mammals and aquatic organisms Flury and Papritz, 1993. Based on the toxicity data, a ground water quality criterion of 1 mg Br^- /L was established in the literature Behrens et al., 2001. Acceptable daily intake values for humans have been proposed at 0.1 and 1.0 mg Br^- /(kg body weight). According to UBA

(Behrens et al., 2001), tracing tests with bromine salts should not be conducted if water treatment facilities could be afflicted that use oxidizing agents. This way, the formation of cancerous compounds such as bromates or bromide hydrocarbons can be prevented.

In general, the use of the tracers Li^+ and Br^- is not considered critical at this stage of the project. Firstly, an impact on drinking water treatment facilities by exfiltrating sewage can mostly be excluded, as sewers are not supposed to be laid within the vicinity of treatment works or near wells. Secondly, the tracer concentration in the samples does not exceed a few mg/L and is further diluted in the reactors of the WWTP. The expected concentrations in the effluent therefor are in the range of a few micrograms per liter and are even more diluted in the receiving waters.

4. General preliminary planning

The general preliminary planning consists of the collection of data as well as some preliminary testing. These two steps form the basis for further planning.

4.1. Collection of data

4.1.1. Constructional data

Among the constructional data of interest are the length, the slope, the profile, the cross section, the junctions, an eventual change of profiles and the material of the investigation reach.

4.1.2. Additional data

Additional sewer structures certainly play a role during the performance of the experiment. For example a spillway may cause better mixing, whereas a retention tank will level out and flatten the tracer signal, which is not regarded favourable.

4.1.3. Hydraulic data

Although in most cases there will not be a continuous measurement of the discharge, most sewer operators can give a rough estimation on the flow in the investigation reach. An estimate with a precision of 30 % is considered the minimum requirement for the design of the experiment. For accurate experimental design a preliminary measurement is recommended.

4.1.4. Background concentration of selective ions in the wastewater

Though the natural background of lithium was found to be negligible at the test sites, bromide background concentrations can vary from location to location because of the

different environmental and geo-chemical conditions. A possible source of both substances are industrial applications. In addition, some road salts seem to contain a small amount of bromide. Both natural and artificial sources can cause considerable variations in the wastewater concentrations. Preliminary measurements at the measuring cross section are necessary to avoid gross errors in the experimental design and to estimate the uncertainty of the experimental results. During the QUEST-C experiment a sampling of the bromide background is recommended.

4.2. Preliminary tests

The following preliminary tests are recommended for the further planning and will help to optimise the realisation of the experiment.

4.2.1. Discharge

Measurements of discharge should be made for at least two days up to one week before the experiment. During the preliminary monitoring, a resolution of a few minutes should be sufficient, but a high resolution is favourable.

4.2.2. Background concentration

The background concentration data of the wastewater should be collected during the periods of the day which look optimal from the point of view of discharge variation. This knowledge is needed to determine which tracer is optimal for the *indicator* and which one for *reference* tracing. Ten mixed samples, each over ten minutes, were used during experiments at Rümlang, CH. Hint of how to implement satisfactory sampling strategies can be found in {Ort, 2005 #532}.

4.2.3. Dispersion, Residence time distribution (RTD)

In order to avoid gross errors due to dispersion effects additional tracing tests have to be conducted to get information on the residence time distribution. The cheapest and most common tracer is sodium chloride, measured with conductivity devices. The conductivity meter should have a time resolution of one second and a measuring range from 0 to 20 mS/cm and two digits after the comma. It consists of the probe and an appropriate device. TetraCon 325 standard conductivity cells of WTW (Wissenschaftlich Technische Werkstätten GmbH, Weilheim, Germany) were used in our experiments. The devices should have a reliable temperature correction which has to be checked in a laboratory trial. Calibrated conductivity probes have to be installed in a clogging-free manner. A model boat (see section D.1) served well for this purpose. It is obligatory to use at least two conductivity probes to compensate for an eventual incorrect operation of one device (clogging).

In the pre-test two or more pulses are added at the two dosing points for indicator- and reference tracer and at the measuring point the conductivity is recorded. Time of the pulse

transit, form, height and width give information about the RTD and dispersion of the tracer cloud and will be necessary for an optimal experimental design.

If tracer tests are not possible a rough estimation of the average residence time can be also done with floating objects such as polystyrene particles, tennis balls or even apples. However, results are coarse and the wastewater must be constantly observed to detect the floating objects downstream.

If an additional submersible pump is used between the dosing point of the reference tracer and the sampling cross section, the pre-test should be done with the pump in operation. It was found that the hydraulic properties of the section are sometimes changed significantly with an operating pump.

The pre-test should be done in the periods of the day which were chosen for QUEST-C exfiltration measurements.

4.2.4. Mixing length

As the tracer has to be fully mixed at the measuring cross section, a sufficient mixing length has to be provided. The relationship between the mixing length and channel width is quadratic $L_{mix} = f(b^2)$. Therefore, it is recommended to distribute the dosing tubes equally across the channel width. Information on mixing modelling can be found in Rieckermann et al., 2004, Fischer et al., 1979, Jirka et al., 1991.

For non-buoyant tracers, Rutherford, 1994 recommends mixing lengths of $100-300 * d$ (d = channel width). Mixing can be considered complete if the coefficient of variation of several probes at different places of the cross section is greater than 0.98. Rutherford, 1994.

If mixing was found to be incomplete, one or more submersible wastewater pumps should be installed upstream of the *sampling cross section* for additional mixing.

5. Experimental design

The experimental design aims at defining dosing duration, tracer masses, and the necessary volumes for dosing. The goal is to find the optimal dosing and sampling protocol. At the present stage, only preliminary recommendations can be given.

5.1. Determining the mass of the indicator tracer

The basic criteria for determining the mass for the *indicator tracer* is the expected sewer discharge during experiment, the total dosing time and the required concentration of *indicator tracer* at the sampling cross section.

5.1.1. Total dosing time of the indicator tracer

The total dosing time $t_{d,tot}$ is calculated after

$$t_{d,tot} = t_s + t_p + t_f \quad (4)$$

- t_s - sampling time (in the Ruemlang experiments: 100 minutes)
 t_p - time required to get a stable plateau concentration in the sampling cross section after first tracer detection (this information can be obtained from the time of passage of the conductivity peak from the RTD pre-experiment).
 t_f - safety time to cover sampling with stable mass flow of tracer. It is recommended to set this value to 15 – 30 min (depends on the length of investigation reach).

5.1.2. Sewer indicator tracer concentration

Because of negligible background concentrations of lithium in the investigation reach and no significant interferences with other substances in the IC analysis the recommended concentration of lithium should be in the range of 1-2.5 mg Li⁺ /L in the sampling cross section.

From these information one can compute the adequate amount of tracer mass, volume of the container and a feasible dosing flow rate. The maximum dosing concentration of the tracer solution should not exceed 415 gLiCl/L (half of the saturation concentration).

5.2. Determining the mass of the reference tracer

The necessary volume of the reference tracer can be computed in analogy to the indicator tracer, but the time required to get stable plateau concentration in the sampling cross section is definitely much shorter.

5.2.1. Sewer reference tracer concentration

The concentration of bromide in reach under investigation was in the range 0 to 0.5 mg Br/L (Rümlang, CH). The recommended concentration of bromide should be in the range of 2.5-5.0 mg Br/L in the sampling cross section, because of slight interferences from other substances (nitrite NO₂⁻, carbonates) in the chromatogram.

From this information one can compute the adequate amount of tracer mass, volume of the container and a feasible dosing flow rate. The maximum dosing concentration of the tracer solution should not exceed 240 gNaBr/L (half of the saturation concentration).

5.3. Sampling protocol

The sampling protocol defines the sampling procedure and determines the total duration of sampling, the number of individual samples and the sampling time. As these are very individual settings, they depend on the local situation of the end user (equipment, financial resources, etc.) and of the conditions in the sewer (tracer background concentrations, variation of discharge, etc.). At the present stage of the project, it is not

possible to give general recommendations regarding the number of samples or the sampling time.

In the experimental trials done at Rümlang, CH, 2*10 samples were simultaneously taken in intervals of 5-10 minutes. In addition, 10 background samples were taken at the dosing point of bromide. To obtain results with less random variations, time-proportional composite samples were taken with peristaltic pumps instead of discrete sampling.

6. Preparation of the experiment

6.1. Time schedule

The time planning should consider the preparations in the lab and in the field, the realisation of the experiment, filtration of samples, preparation of samples for analysis and the cleaning of the equipment after the experiment. A form for the various steps can be found in the Appendix C.1.

6.2. Coordination of activities with local authorities

Before any technical preparation of the experiment the local sewer and traffic authorities have to be contacted. It is absolutely necessary to keep with their safety and security requirements and apply general safety and security standards (Annex A).

Possible dangers for health and environment are among others:

- traffic
- lifting and carrying/ dropping goods (manhole cover)
- falling
- gas
- lack of oxygen
- infection
- an explosive sewer atmosphere
- flooding/ water waves
- contact with highly concentrated tracer solution

6.3. Preparing the tracer solutions

6.3.1. Lithium

The required amount of LiCl is dissolved in warm water which eases the dissolution of the salt. This laboratory standard should be stirred until a homogeneous solution is achieved (for at least 1 hour). Any splashing of water should be avoided. A sufficient amount of this solution should be kept separately to determine the concentration, which is needed to calculate the tracer mass inflow into sewer system.

6.3.2. Bromide

The required amount of NaBr is dissolved in warm water which eases the dissolution of the salt. A homogeneous solution of this *laboratory standard* has to be guaranteed. A sufficient amount of this solution should be kept separately to determine the concentration, which is needed to calculate the tracer mass inflow into sewer system. The drying of the bromide salt has to be done in a ventilated space because bromine vapours are irritating for eye and mucosa.

6.4. Preparing the sampling flasks

During the experiment flasks are required to store the sampled sewage. A typical volume is in the range of 0.5-1 liter, whereas the flasks for the filtrated samples should have a volume of 50 ml. To minimize eventual contaminations from a previous use of the flasks, it is necessary to clean all bottles with an acid. In our experiments the flasks were filled completely with 0.01 mol/L solution of HNO₃ for a minimum time of 2 hours.

6.5. Preparing the field material

The preparation of the material which is needed during the field experiment might optimally take place on the day previous to the experiment. A preliminary checklist has been compiled which can be easily adapted to the individual needs of the end user (see Appendix C).

7. Performing the experiment

7.1. Safety and security

As most flowmeters and pumps are not explosion safe, the neighbouring sewer manholes should be opened to prevent an explosive atmosphere. Furthermore, the installation of a gas warning device is recommended, so that the experiment can be cancelled immediately if there is any sign for a critical sewer atmosphere. In general, make sure that the common safety standards are applied (Annex B).

7.2. Preparation in the field

7.2.1. Dosing point for the indicator tracer (Litihum)

The necessary equipment for the tracer dosing has to be prepared and installed. The tracer solution is pumped directly to the sewer with a pump through a flexible tube. Container with the LiCl solution is placed directly on the balance, which is connected to a PC (data

logger) that allows a continuous recording of the weight. To protect balance and container from weather influence it is recommended to place the whole set-up into a closed box. The end of the dosing tubes should be fixed close to the water surface. For faster mixing the tubes should be uniformly distributed across the width of the water surface.

7.2.2. Dosing point for reference tracer (Bromide)

In principle, the set-up for the dosing of the reference tracer is the same as for the indicator tracer (see above). Additionally, in this manhole the background concentration of the reference tracer has to be sampled. The modified set-up is shown in figure 2-2.

A sufficient number of sampling flasks, the necessary filtration equipment and the flasks for the filtrated wastewater samples should also be prepared.

7.2.3. Sampling cross section

At the sampling cross section the measuring and sampling instruments have to be installed. It is highly recommended to measure the discharge, because it provides redundant information that can be used to check the tracer data for eventual artefacts. Furthermore it is mandatory if a more advanced data analysis procedure is applied Rieckermann et al., 2005.

At this point, it is most important that the sampling tubes have to be installed in a clogging-free manner. If necessary, a mixing pump has to be installed to achieve complete mixing. If so, it is highly recommended to use a sewage pump that operates reliably in the sewer environment. If a flowmeter is used that operates with the *Ultrasonic Doppler* principle, the mixing pump should be placed one manhole upstream the measuring point. It was found that the additional turbulence of the pump may have a negative impact on the SIGMA flowmeter.

As most flowmeters and pumps are not explosion safe, the neighbouring sewer manholes should also be opened to prevent an explosive atmosphere.

7.3. Performing the experiment in the field

The realization of the experiment is effectuated after the sampling protocol described in section 5.3. Before the start of the tracer dosing, the peristaltic pumps have to be warmed up to ensure steady operational conditions. It is advisable to check the operation of the equipment frequently. Especially the sampling tubes in the dosing manhole of the reference tracer have been subsequently clogged by sewer solids during the experiments. At this stage of the project, we still see some optimisation potential for the collocation of the sampling tubes in the flow (e.g. onto a model boat, onto a sewer ring). After the final sample has been collected, the *field standard* has to be prepared.

8. Sample analysis

8.1. Sample preparation

The samples should be filtrated immediately after the sampling procedure in the field. If this is not possible, the filtration must take place in the laboratory immediately after the return from the field site.

Membrane filters with pore size of 0.45 μm should be used (Schleicher&Schuell, Sartorius) together with plastic filter holder units and a 100 ml syringe. To prevent the leaking of any particles to the samples its recommended to do a second filtration step with laboratory filter plastic units. Such membrane filters should have a pore size of 0.45 μm and a diameter from 15 to 25 mm (Sartorius, Orange). During night-time experiments, when the TSS concentration is minimal, these small filters may also be used for the direct filtration of the samples. For IC analysis, the volume of 30 ml is regarded sufficient.

8.2. Preparation of the laboratory standard

To estimate the ratio between the concentration of the dosing solution it is necessary to analyze the standard solutions. To minimize the errors from the dilution of the highly concentrated dosing solutions, it is recommended to dilute a mixture of both liquids together. It is recommended to weight defined amounts of the LiCl solution (e.g. 1 g) and the NaBr solution (e.g 1.5 g) and mix them together in the same beaker. Because of the high concentrations the mixture must be diluted to meet the measuring range of the IC (e.g 1:10 000).

8.3. Analytical method

For the analysis of cations and anions it is recommended to use the ion chromatography (IC) method. The main advantage of IC method is the possibility to analyse both cations (Li^+) and anions (Br^-) in one run of the ion chromatograph. However, the instrument has to be equipped with two columns (cation column and anion column). The detailed description of the device used is given in Appendix B.

The basic principle of the QUEST-C method is that only the ratio of the concentrations of both substances is regarded. In ion chromatography there is in general a linear relationship between the concentration of the substance and the peak area obtained from the device. Since the standards of each experiment are also analysed, any calibration of the IC device is not required.

To avoid systematic errors of the samples, the IC analysis of all samples should be done in random order. It is recommended to analyse control standards and samples of distilled water between a certain number of samples (e.g. each 5th or 10th sample) to check the device for stability.

9. Data analysis

The analysis of the experimental data aims at the computation of the measured exfiltration. Firstly, all the information of the field experiment (logger files, hand-written notes) has to be carefully checked and scanned for gross errors (correct operation of the pumps, correct sampling intervals, etc.). Secondly, the pump rates have to be computed from the information of the scale. Thirdly, before the final computation of exfiltration, the analytical results have to be inspected.

9.1. IC analysis

The results from measurements with an ion chromatograph is a chromatogram in which each substance is represented by a peak. The magnitude of the concentration is represented by the magnitude of each peak. For Bromide and Lithium analysis, the peak area is taken for the computation of exfiltration. At our laboratory, it proved better to determine both substances with the area of the peaks instead of the peak heights.

9.1.1. Peak detection

The first step always must be to visually check the chromatograms of the samples for correct peak detection. Generally, this done by the laboratory personnel, but it is still mandatory to personally work through the data himself/herself to avoid gross errors. Depending on the behaviour and the settings of the device, peak areas are not detected correctly in the chromatogram. To compensate for this error, it is possible to do a manual detection and separation of the peak.

9.1.2. Integration

The same visual check applies to the correct integration of the peak. In some cases there are overlapping peaks or non-linear baselines that are not detected by the device. A manual baseline separation and integration of the peak can mostly compensate for this error, too.

9.2. Computation of mass loss of indicator tracer

With the data obtained from the analytical procedure, tracer mass loss can be computed. The first step is to calculate the ratio of Br^-/Li^+ from the pump rates and the *laboratory standards*. As the *field standard* should contain redundant information, a plausibility check of the obtained results is possible.

The second step is the correction for the background of bromide. The peak areas measured in the background samples simply have to be subtracted from those measured in the samples. Finally, the ratio of Li^+/Br^- in the samples has to be computed and compared

to the ratio of the mass input estimated from the tracer concentrations of the standards and the pump rates.

9.3. Error propagation

To estimate the uncertainty of the computed exfiltration, a Gaussian error propagation is performed. At this stage of the project, it seems reasonable to assume that the random errors are normally distributed and have a relatively small standard deviation. Under these assumptions a linear propagation of the individual standard deviations of each error is possible after

$$\sigma_E = \sqrt{\sum_{i=1}^n \left(\frac{\partial E}{\partial p_i} \right)^2 \cdot \sigma_{p_i}^2}$$

where p_i is the individual parameter (source of error), σ_{p_i} is the magnitude of the error (the standard deviation of its normal distribution) and $\partial E / \partial p_i$ is the partial derivative of the exfiltration equation (eq. 3) to the individual parameter.

The individual parameters considered here are:

- error of the pumping rate Br
- error of the pumping rate Li
- error of the peak area of the laboratory standard (IC)
- error of the area of the scale used to prepare the laboratory standard
- error of the peak area of the samples resulting from the analytical procedure, the sampling procedure and the variation of the flow

The errors of the analytical procedure, the sampling procedure and the variation of the flow rate are not explicitly considered. As they are difficult to separate, a more advanced data analysis is necessary to reduce uncertainty in the mass loss estimate.

The model for data analysis including uncertainty analysis is provided with an EXCEL spreadsheet. An example data set is also provided which serves as a tutorial.

9.4. Dynamic data analysis

In case that the hydraulic properties of the reach during the experiment can be modelled satisfactorily, a dynamic data analysis can be used to obtain more accurate results. Further information is provided in Rieckermann et al., 2005.

10. Interpretation of the results

All computed exfiltration results have to be interpreted carefully, because a detailed analysis of eventual limitations has not been effected at the present stage of the project.

Please consult Rieckermann, 2005 for further information. In case of no inflow and uniformly distributed defects along the investigation reach and the perimeter of the sewer,

a loss of tracer can directly be interpreted as exfiltration. However, sometimes negative mass losses are computed which indicates a gain of tracer mass. Most often, this is due systematic errors due to the variation of natural baseline concentrations or uncertainty in the dosed volume of indicator and reference pulses.

For the assessment of the obtained exfiltration rates, the following points have to be evaluated

- Tracer losses during dosing (pump, tube, splashes at the inflow)
- Tracer losses by adsorption of tracer substance on biofilm or sewer material
- Degradation or conversion of the tracer in the wastewater

Niesel, 2003 shows that the type of dosing has a noticeable influence on the result and that even under stable operating conditions pumps and balances can have an error of a few percents.

Another phenomenon that has to be taken into account is that the mass loss estimate is a relative figure, which relates to the labelled wastewater stream before the first leakage occurs. In a network this might be difficult to determine and often exfiltration in a reach can only be related to upstream discharge values. As discharge generally increases in a reach, this can mostly be taken as a lower bound. In case that discharge in a investigation reach is very non-uniform, the investigation has to be carried out in smaller subsections.

However, the current knowledge does not allow for more comprehensive recommendations on the interpretation of results of tracer loss from experiments in a network situation.

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A. General safety and security guidelines for working activities in sewers

In general, a permission of the local authorities is necessary before opening any manhole cover of or entering the sewer under investigation.

The following safety precautions have to be taken when working in the sewer environment:

- Safeguard the surroundings of the corresponding manholes with respect to traffic
- Use appropriate devices for opening manhole covers
- Make sure that the area at street level is protected to prevent materials or foreign objects from falling into the manhole
- At least one additional person has to be present outside the sewer to organize help in case of an accident. A first aid box, a fire extinguisher and a mobile phone should be available on the site.
- Wear at least the following personal safety equipment: Protective clothing, gloves, a safety helmet and security boots with steel caps. An explosion safe light source is mandatory.
- Secure every person entering the sewer (cable winch and tripod)
- Ensure adequate ventilation and continuous air monitoring with a gas warning device
- Care about proper hygiene, including frequent and routine hand washing, especially before eating, smoking and after the end of the experiment. This may require to bring clean water to the experimental site.

Furthermore, vaccinations for diphtheria and tetanus are recommended. All workers should also have sufficient protection against Hepatitis A and B.

B. Devices specification

B.1. Dosing pumps

ISMATEC BVK standard

Flow rates < 0.003 – 3700 ml/min (depending on mounted pump-head)
Channels 1 – 24 (depending on mounted pump-head)
Speed 2.4 – 240 rpm
Speed setting 1-99.9%, resolution 0.1%, 3-digit potentiometer
Speed control Closed loop control for pressure independent speed
Analog input Speed control (0–5 or 0–10V / 0–20 or 4–20mA)
Analog output Speed monitoring (0–10VDC or 0–12 kHz)
Digital input (TTL level) Start/Stop, rotation direction
Differential pressure 1.5 bar (21.7 psi) possible (depending on pump-head and tubing material); small tubing diameters may allow higher pressures
Motor type DC motor
Power consumption max. 100 W
Mains connection 115/230VAC; 50/60Hz
Protection rating IP 30
Depth / Width / Height 220 x 155 x 260 mm (8 2/3" x 6 1/10" x 10 1/4") (without pump-head)
Weight 5.7 kg (13 lb) (without pump-head)

ISMATEC MV-CA4

8 heads peristaltic pump
max 4 channels
variable rotation speed
CA tubing cassettes
Differential pressure 1.5 bar (21.7 psi) possible (depending on pump-head and tubing material); small tubing diameters may allow higher pressures
Motor type DC motor
Power consumption max. 100 W
Mains connection 115/230VAC; 50/60Hz
max flow rate ca. 120 ml/min/1 tube = 480 ml /min/4 tubes with purple - orange tubing Q = 29 l/hour

B.2. Ionchromatography

Metrohm Compact IC

Anions

Column: Metrosep A SUPP 5 150 mm (particle size 5 µm)
Eluent: 1.0 mmol/L sodium hydrocarbonat/ 3.2 mmol sodium carbonat
Loop: 20 µml

Kations

Column: Metrosep C 2 100 mm (particle size 5 µm)
Eluent: 4 mmol/L tartaric acid, 1 mmol/L dipicolinic acid
Loop: 10 µml

B.3. Chemical specification

Lithium Chloride

Granular, typ. 99.5% LiCl
Product No. 401112
CAS-No. 7447-41-8
White, crystalline granulate
Molecular weight: 42.40
Specific gravity: 2.608 g/cm³
Solubility in water (20°C): 83.2 g LiCl/100 g
Producer:
Chemetall GmbH
Fax +49697165-2053
Email: lithium@chemetall.com
www.chemetalllithium.com
Price: ca. 30 CHF/kg

Sodium bromide

Granular, typ. 99.9 % NaBr
Product No. 81945-330
sack of 25 kg
Solubility in water (20°C): 48.0 g NaBr/100 g
Producer:
Schweizerhall Chemie AG
Elsässerstrasse 231
CH 4031 Basel
Fax +41613268526
Email: irsi.stobler@schweizerhall.ch
<http://www.schweizerhall.ch/>
Price: ca. 10 CHF/kg

C. checklists and forms

C.1. Checklists for planning

Time scheduling

location	task	time
office		
	design experiment (tracer masses, tracer volumes, mixing lengths, dosing protocol, etc.)	
Laboratory		
	preparing of tracer solution, filling into receptacles	
	cleaning of sampling flasks for non-filtrated samples (50ml)	
	cleaning of sampling flasks for filtrated samples (50ml)	
	preparation of equipment used in the field	
Field		
	installation of sampling pumps (+ flow meter)	
	installation of dosing stations	
	continuous dosing of tracer and continuous sampling	
	de-installation of dosing stations	
	preparation of the field standard	
	de-installation of sampling pumps (+ flow meter)	
Laboratory		
	Preparation of samples for IC analysis (eventual 2 nd filtration)	
	cleaning of equipment after experiment	
Office		
	Check of data (chromatograms, discharge, scales, ...)	
	Computation of exfiltration	

C.2. Checklists for equipment

Safety/traffic and Safety/sewer**safety sewer**

	tripod	
	helmets	
	safety harness	
	gas warning device	
	working clothes	
	warning jacket	
	gloves (gum and lab)	
	rubber boots	
	clean water (10 l)	
	soap	
	disinfectant	
	flashlight/ headlamp	
safety traffic	warning triangle	
	material for making a barrier	
	warning cones (Pylonen)	

C.3. Field stations

Indicator tracer station

Scaling	Balance OHAUS 0-150 kg Notebook with cable connection to OHAUS and software	
Dosing	Peristaltic pump (e.g. Ismatec BVP) with tubing (i.d. 3mm) Container with LiCl solution 25 L Holder for tubes	
Power supply	generator 220 V/ batteries+transformer Cable ties	
Reference station	tracer	
Scaling	Balance OHAUS 0-150 kg Notebook with cable connection to OHAUS and software	
Dosing	Peristaltic pump (e.g. Ismatec MV-CA4) with tubing (i.d. 3mm) Container with NaBr solution 25 L Holder for tubes	
Sampling	peristaltic pump for continuous sampling with TYGON ST tubing (i.d. 5 mm) Flasks for background sampling – (e.g. 10 * 500 ml, 10* 50ml) Filtration apparatus – syringe, tweezers, filters (e.g. Schleicher 0.45 um, diameter 47 mm)	
Sampling station		
Sampling	2 channel peristaltic pump for continuous sampling with TYGON ST tubing (i.d. 5 mm) Flasks for background sampling – (e.g. 10 * 500 ml, 10* 50ml) Filtration apparatus – syringe, tweezers, filters (e.g. Schleicher 0.45 um, diameter 47 mm)	
Discharge measurements	Flow meter (e.g. American Sigma) Notebook with connection to Sigma flowmeter and Insight software	
Power supply	Generator 380 V (for submersible pump e.g. AMAREX) + cable ties	

D. General equipment**Miscellaneous**

Synchronized clocks	
Distilled water with squeezing bottle	
Boat or float for the conductivity probes	
data logger + connection cables for logger	
lab gloves	
cleaning tissue/ paper	
towel	
garbage bag	
string/ cord	
duct tape	
water-level	
buckets 15L, 10L	
toolbox + red pliers (Rohrzange)	
pick axe	
cable ties	
cutter	
wooden layer fo placing the balance on	
scissors	
pen + paper	

D.1. Images of equipment**Figure 6 Boat for the conductivity probes**

Dosing equipment



Figure 7 Dosing tubes holder

Dosing station equipment



Figure 8 Box for sheltering of the dosing equipment at a dosing station



Figure 10 Filtration apparatus

Sampling pumps



Figure 11 Peristaltic sampling pumps

Dosing pumps



Figure 12 Dosing pump ISMATEC BVK



Figure 13 Dosing pump ISMATEC MV-CA4

Sampling flasks



Figure 14 1 L sampling containers