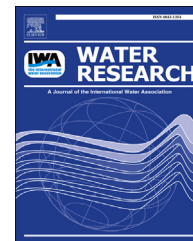




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# Online dissolved methane and total dissolved sulfide measurement in sewers

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## ABSTRACT

Recent studies using short-term manual sampling of sewage followed by off-line laboratory gas chromatography (GC) measurement have shown that a substantial amount of dissolved methane is produced in sewer systems. However, only limited data has been acquired to date due to the low frequency and short span of this method, which cannot capture the dynamic variations of in-sewer dissolved methane concentrations. In this study, a newly developed online measuring device was used to monitor dissolved methane concentrations at the end of a rising main sewer network, over two periods of three weeks each, in summer and early winter, respectively. This device uses an online gas-phase methane sensor to measure methane under equilibrium conditions after being stripped from the sewage. The data are then converted to liquid-phase methane concentrations according to Henry's Law. The detection limit and range are suitable for sewer application and can be adjusted by varying the ratio of liquid-to-gas phase volume settings. The measurement presented good linearity ( $R^2 > 0.95$ ) during field application, when compared to off-line measurements. The overall data set showed a wide variation in dissolved methane concentration of 5–15 mg/L in summer and 3.5–12 mg/L in winter, resulting in a significant average daily production of 24.6 and 19.0 kg-CH<sub>4</sub>/d, respectively, from the network with a daily average sewage flow of 2840 m<sup>3</sup>/day. The dissolved methane concentration demonstrated a clear diurnal pattern coinciding with flow and sulfide fluctuation, implying a relationship with the wastewater hydraulic retention time (HRT). The total dissolved sulfide (TDS) concentration in sewers can be determined simultaneously with the same principle.

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## 1. Introduction

Sewer systems are an important and integrated component of the urban water infrastructure. Sewer networks collect and

transport wastewater through underground pipelines to wastewater treatment plants for pollutant removal prior to environmental discharge. They protect our urban society against sewage-born diseases, unhygienic conditions and

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obnoxious odours, allowing us to live in ever larger and more densely populated cities (Pikaar et al., 2014). However, sewer emissions are a notorious problem that water utilities have to deal with (Ganigue et al., 2011; USEPA, 1974). The production and emission of hydrogen sulfide is a well-known problem for decades. Under anaerobic conditions, sulfate is reduced to sulfide through a biological process mediated by sulfate-reducing bacteria (SRB). Dissolved hydrogen sulfide thus produced can be released from wastewater as the hydrogen sulfide gas, which is the primary cause of sewer odors and corrosion (Joseph et al., 2012; Santry, 1966). However, hydrogen sulfide is not the only harmful emission from sewer networks. Methane can also be generated in anaerobic sewers through methanogenesis (Foley et al., 2009; Guisasola et al., 2008; Jiang et al., 2013). Methane is a highly potent greenhouse gas which is significantly contributing to climate change (IPCC, 2013). It is also explosive with a lower explosive limit (LEL) of approximately 5% by volume (vol), and thus poses a serious safety issue (Spencer et al., 2006). In addition, methane generation in sewers may consume a significant amount of soluble chemical oxygen demand (COD), which is detrimental to nutrient removal in downstream wastewater treatment plants (WWTPs).

There is currently a major effort aimed at understanding and quantifying methane production in WWTPs (Bani Shahabadi et al., 2009; Daelman et al., 2012; Wang et al., 2011). Significant progress is enabling better management of methane emissions in treatment plants. In contrast, very little attention has been paid to methane formation in sewer networks.

Recent studies have shown substantial methane production and emission from sewers by manual sampling followed by off-line laboratory analysis of CH<sub>4</sub> content in wastewater samples using gas chromatography (GC). Over a 6 h measurement campaign with half-hourly manual sampling of sewage, Guisasola et al. (2008) detected dissolved methane with concentrations of 5–25 mg/L at two rising mains. Foley et al. (2009) also measured dissolved methane concentrations of 1.5–9 mg/L during a 4 h manual sampling campaign with a sampling interval of one hour. However, because sewers are dynamic systems, manual sampling campaigns may not capture dynamic variation in methane production (Sharma et al., 2008), imposing a serious limitation on accurate quantification. In addition, manual sampling is not practical for long-term, intensive quantification of methane concentration in complicated sewer networks with a large number of sampling sites.

The overall aim of this study is to capture online dynamic methane data from sewer networks, to better understand and quantify methane production and emission from sewers. To achieve this goal, an online dissolved methane sensor suitable for sewer measurement is required. Although a limited number of commercial sensors for online detection of dissolved CH<sub>4</sub> are available (Camilli and Hemond, 2004; Lamontagne et al., 2001; Tsunogai et al., 2007) (Table S1), these are mainly designed for measuring methane in clean water bodies by using gas-permeable membranes to extract methane gas from water. This method cannot be used for measuring domestic sewage containing a large amount of

impurities as well as sulfide at high concentrations (Boulart et al., 2010).

In this study, a measuring device was specifically designed and developed for dissolved methane measurement in sewer systems. The device comprises a stripping vessel, where dissolved methane is stripped to gas phase, and a measuring chamber, where an online gas-phase methane sensor is used to acquire the methane data, which is then converted to liquid-phase dissolved methane concentration according to Henry's Law. The measurement performance was verified through both laboratory experiments and field applications. The sensor was then applied to long-term monitoring of dissolved methane concentrations at the end of a rising main sewer network for a period of three weeks in both summer and winter. To the best of our knowledge, this is the first reported study of in-situ continuous quantification of dissolved methane concentration in sewers. In addition, simultaneous dissolved methane and total dissolved sulfide (TDS) were achieved by the same principle, and validated under field sewer conditions.

## 2. Materials and methods

### 2.1. Method development

Fig. 1 shows the laboratory experimental set-up for in-situ dissolved methane and TDS measurement. The principle is to strip the dissolved gas in the liquid into the gas phase, and then convert this gas phase concentration under equilibrium back to liquid phase concentration using Henry's Law (Schulthess and Gujer, 1996). In order to carry out the measurement, a wastewater sample (500 mL) was pumped into the stripping chamber using a feed pump (Masterflex L/S, 77800-50, flow rate 1 L/min). For CH<sub>4</sub> measurement, once the feed pump was stopped, the air recirculation pump (Xinweicheng, FML201.5, flow rate 1.5 L/min) and stirrer (Heidolph, MR Hei-Standard, 1400 rpm) were started, enhancing the transfer of dissolved methane from liquid to the headspace with a total gas volume of 1070 mL including 370 mL headspace in the stripping vessel and 700 mL of the gas measurement chamber. A chilling unit was incorporated between the stripping and measurement chambers to eliminate the effect of moisture on the gas sensor by maintaining the relative humidity to a level below 70% (Liu et al., 2014). The recirculation continued until equilibrium was attained (about 4–5 min from stripping, as confirmed by the sensor reading curve as well as GC analysis). Based upon the equilibrated gas phase measurement using a GasTech S-Guard methane detector (range, 0–5.00% vol, and resolution, 0.05% vol), the liquid phase methane concentration under equilibrium was calculated using Henry's Law (Eq. (1)). The total amount of methane in the original sewage sample is calculated as the sum of the measured amount of methane in the headspace and the calculated amount of methane in the sewage sample after stripping (Eq. (2)).

$$C_{w,eq} = H \cdot R \cdot T \cdot C_{g,eq} \quad (1)$$

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