Application of Portable Fluorescence Spectrophotometry for Integrity Testing of Recycled Water Dual Distribution Systems

Adam C. Hambly,[a,*] **Rita K. Henderson,**[a] **Andy Baker,**[b] **Richard M. Stuetz,**[a] **Stuart J. Khan**[a]

[a] Water Research Centre, School of Civil and Environmental Engineering, The University of New South Wales, Sydney 2052 Australia
[b] Connected Waters Initiative Research Centre, The University of New South Wales, Sydney 2052 Australia

Water utilities supplying recycled water to households via a "third-pipe" or "dual reticulation" system have a need for a rapid, portable method to detect cross-connections within potable water reticulation networks. This study evaluates portable fluorimetry as a technique for cross-connection detection in the field. For the first time, an investigation of a full-scale dual reticulation water-recycling network has been carried out to identify cross-connections using a portable fluorimeter. We determined that this can be carried out with a 3 mL water sample, and unlike methods that are currently in use for cross-connection detection, can be achieved quickly without disruption to water flow or availability within the network. It was also revealed that fluorescence trigger values could be established with high levels of confidence by sampling less than 2.5% of the network. Fluorescence analysis was also able to uncover a single, real cross-connection event. As such, this paper is a fundamental demonstration of fluorescence as a reliable, highly portable technique for cross-connection detection within dual reticulation water recycling networks and further establishes the abilities of fluorescence devices as valuable field instruments for water quality monitoring.

Index Headings: **Cross-connection; Monitoring; Fluorescence; Dual reticulation; Water recycling.**

INTRODUCTION

Dual reticulation, or third-pipe, systems are increasingly common in new housing developments for the partial substitution of potable water with non-potable recycled water via a secondary network. These networks are generally implemented in situations where both distribution systems (potable water and recycled water) can be installed at the same time, such as in greenfield housing estates, apartments, and new commercial buildings. Such systems have been used to supply a secondary source of water for uses such as toilet flushing, firefighting, and irrigation.[1] As wastewater recycling has gained traction, with clear benefits from potable water conservation and environmental perspectives, recycled water has become a common source of non-potable water that is commonly used within dual reticulation water networks. Recycled water dual reticulation networks exist worldwide, with notable examples in Australia,[2–8] the United States,[9–12] Europe,[13] Japan,[14] and the Middle East.[15]

A cross-connection between potable water and recycled water distribution systems is an inherent risk that comes with the construction and maintenance of dual reticulation networks.[16,17] Since the source and treatment levels of recycled water may vary significantly according to local regulations and intended uses, the hazards that a cross-connection may present to consumers can also vary to a great extent. At the very least, cross-connections could increase the perceived risk of using recycled water, which in turn may negatively impact confidence in the safety of dual reticulation systems and attitudes toward water recycling in general.[18] The ability to rapidly and reliably test for cross-connections would greatly help to strengthen public acceptance of these networks, allowing the continued growth of such water recycling schemes and therefore the sustainable management of municipal water.

Currently, a widely used method for identifying cross-connections involves shutting off the supply of one distribution system at a property boundary and testing for a lack of flow within the other.[17,19–22] However, this method is unable to detect cross-connections that originate beyond individual properties and, furthermore, is time consuming, costly, and disruptive to consumers. These limitations become increasingly problematic within larger networks. A logical alternative to such a mechanical method is the differentiation of recycled water and potable water by their intrinsic properties. This has been investigated previously by way of parameters such as electrical conductivity (EC), dissolved organic carbon (DOC), turbidity, and ultraviolet (UV) absorbance among others.[23,24] However, the success of these techniques has been limited, since there is typically an overlap between recycled water and potable water in the ranges of these measured parameters. Research has shown fluorescence to be a more promising method in terms of such sensitivity, in addition to being a fast and noninvasive analysis technique. Its use in monitoring potable and recycled water treatment systems has therefore attracted significant research interest.[25–29] Recently, the potential for using fluorescence to differentiate between recycled and potable water was demonstrated in a series of studies that used a bench-scale fluorescence spectrophotometer,[30–32] where the fluorescence intensity of finished recycled water was found to differ from that of potable water by up to a factor of 10 across three Australian dual distribution systems. Fluorescence was also found to outperform UV absorbance at 254 nm, EC, DOC, pH, and turbidity in their

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[*] Author to whom correspondence should be sent. E-mail: adaha@aqua.dtu.dk.

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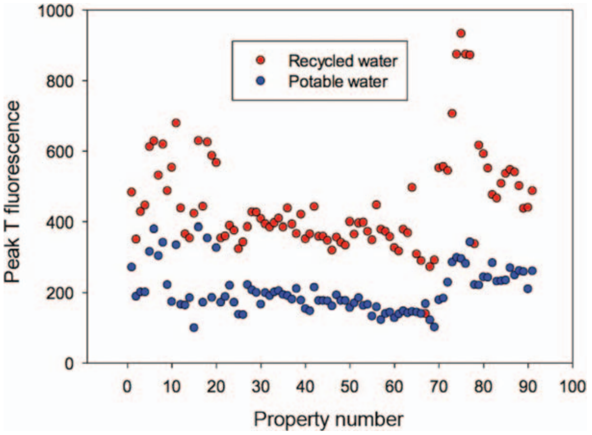

FIG. 1. SMF4 Fluorescence of recycled water and potable water from sampled houses at Aurora Estate and Highlands Estate.

ability to distinguish between recycled water and drinking water, and the most appropriate single wavelength pair for differentiating between recycled water and drinking water using fluorescence was identified as $\lambda_{ex/em} = 300/350$ nm[30]–within the fluorescence region commonly referred to as "Peak T".[33] Fluorescence within this region has been associated with wastewater microbial activity,[34,35] and its persistence within finished recycled water suggests that the Peak T region is a tracer of the remnants of wastewater constituents. This also implies that the recycled water treatment train will ultimately impact the sensitivity of cross-connection detection using Peak T fluorescence.

Fluorescence spectrometers have been employed in situ for water quality monitoring in a number of studies, including to measure the variability in river water dissolved organic matter,[36] to measure protein-like fluorescence in rivers and wastewaters as a surrogate for biochemical oxygen demand,[35] to detect oil contamination in seawater,[37] and to characterize reverse osmosis permeates.[38] They have also been used to quantify viruses in aquatic environments[39] and detect low levels of coliform and *Escherichia coli* bacteria in river and drinking water.[40] However, until a few years ago, portable, commercially available devices capable of monitoring fluorescence in the short UV region were not widely available. Recent developments in this analytical field mean that Peak T portable fluorimeters are now being marketed, and this has increased possibilities for fluorescence analysis to extend into more complex studies, where geographical portability as well as speed and reliability are paramount. Portable Peak T fluorimeters have been successfully used in monitoring ground and surface waters; hence, there is potential to apply them in the monitoring of dual reticulation networks. This paper details the first field-scale assessment of a portable Peak T fluorimeter to assess its in situ ability to reliably detect cross-connections within a full-scale dual reticulation water recycling system.

MATERIALS AND METHODS

Sample Site. Highlands Estate and Aurora Estate (Victoria, Australia) are dual reticulated housing estates that consist of approximately 2000 domestic properties in total, using recycled water for purposes such as toilet flushing, laundry, garden irrigation, car washing, and other non-potable purposes. These estates are supplied from the same water systems, and recycled water has been supplied to the dual reticulation system since 2009 from the Aurora Sewage Treatment Plant and Recycled Water Treatment Plant (operated by Yarra Valley Water), which is located between the estates (approximately 3 km away). The treatment plant uses activated sludge, tertiary filtration, and UV disinfection before water is subjected to advanced treatment in the form of ultrafiltration, further UV disinfection, and chlorination to achieve its finished recycled product, which is then distributed to the dual reticulation system. This treatment train is considered typical of a wastewater treatment plant where the output is intended to be used within non-potable recycled water applications.

Field Survey. The field survey consisted of sampling at 91 individual properties, representing just less than 5% of the dual reticulation network. Sampling was carried out in February 2011, at the same time that the water utility had scheduled a 20% property audit of cross-connections to meet environmental regulatory requirements.[41] Houses were selected at random from throughout the dual reticulation systems and were only subjected to testing if the property owner was available and agreed to have the testing performed (91 properties were sampled within the week). Individual properties were investigated for evidence of cross-connections by turning off each water supply at the individual property level and testing all taps for appropriate flow.

Portable Fluorescence Monitoring. A portable surface monitoring fluorimeter (SMF4) supplied by Safe Training Systems (Wokingham, UK) was used for analysis of fluorescence in the Peak T region. The fluorimeter was one of the first portable devices for monitoring in this short UV excitation range and has been successfully implemented for aquatic monitoring,[40] although now many other portable Peak T devices exist, and the availability and competition between manufacturers is steadily increasing. The fluorimeter monitors a single wavelength pair using an light-emitting diode excitation light source of 280 (±3) nm and measures emission at 360 (±3) nm, which therefore falls within the Peak T region. Data were recorded internally on the instrument and downloaded for analysis using customized Terminal (version 1.9b) software supplied by the manufacturers. The SMF4 used a 10 mm path length, 4 mL volume quartz cuvette (Starna, Australia), and the instrument was zeroed with a sealed, 10 mm path length quartz cuvette of MilliQ water (Varian, Australia). Potable and recycled water taps were opened at high flow for 30 seconds before sampling directly into the quartz cuvette.

RESULTS AND DISCUSSION

Field Survey. The recycled water fluorescence that was measured at the individual properties was typically of greater intensity than potable water fluorescence during the field survey (Fig. 1), which is consistent with previous studies comparing recycled and potable waters.[31] Specifically, the mean (±1 SD) fluorescence intensity of recycled water was more than double that

TABLE I. Mean, standard deviation, and median of recycled and potable water fluorescence intensity in arbitrary fluorescence units (afu) from sampled properties at Aurora Estate and Highlands Estate. Includes values calculated from the data as well as fitted values from the PDFs.

	Potable water fluorescence (afu)		Recycled water fluorescence (afu)	
	Data value	Fitted value	Data value	Fitted value
Mean	206	209	456	469
Standard deviation	64	70	135	185
Median	190	208	414	414
Minimum	100	89	273	295
Maximum	385	+∞	933	+∞

of potable water (456 ± 135 afu and 206 ± 64 afu, respectively; Table I). Previous studies at other dual reticulation systems showed that Peak T fluorescence was typically observed to be from three to ten times that of potable water,[32] where the fluorescent character of recycled water was shown to be dependent on the treatment processes applied and also to vary between treatment plants. Recycled water fluorescence was also observed to have a large range, varying between 273 and 933 afu, whereas potable water fluorescence varied between 100 and 385 afu (Table I). No clear distinctions could be made between the data from each estate (Aurora Estate $n = 54$, Highlands Estate $n = 37$), and a significant overlap between the ranges of fluorescence data for each water source was observed.

In order to better assess the extent of the overlap between fluorescence, the probability density function (PDF) of potable water and recycled water were compared. A PDF is a mathematical function that represents a distribution in terms of the probability or frequency of occurrence of specific values within the distribution. A PDF can be conceptualized as a "smoothed out" version of a histogram of occurrences of a range of values. If sufficient values of a continuous random variable are sampled, producing a histogram depicting relative frequencies of output ranges, then this histogram will resemble the random variable's probability density. As such, the potable and recycled water datasets were each fitted to a lognormal PDF with @Risk software (Palisade Corporation; Fig. 2). Due to the significant overlap in ranges during the sampling period (upper 5th percentile of the potable water fluorescence intensity distribution was higher than the lower 15th percentile of the recycled water fluorescence intensity distribution), attempting to distinguish potable water from recycled water based on a single measurement of potable water would be subject to a significant rate of false positives (where a single drinking water point value would falsely indicate a cross-connection with recycled water).

Although there was considerable overlap between the ranges of recycled and potable water fluorescence intensity distributions, recycled water fluorescence was higher than potable water fluorescence in every instance when considered on an individual property basis. This could suggest that temperature had an effect on the fluorescence readings, since fluorescence intensity is inversely related to sample temperature. Investigations of thermal quenching properties of wastewater fluorescence showed that increasing the temperature from 10 to 45 °C resulted in a subsequent 35% decrease in fluorescence intensity.[42] Since the amount of time between individual property analysis (up to 2 h) was much greater than the time between potable and recycled water analysis of individual properties (less than 5 min), this could indicate that sample temperature differences were at least in part responsible for the

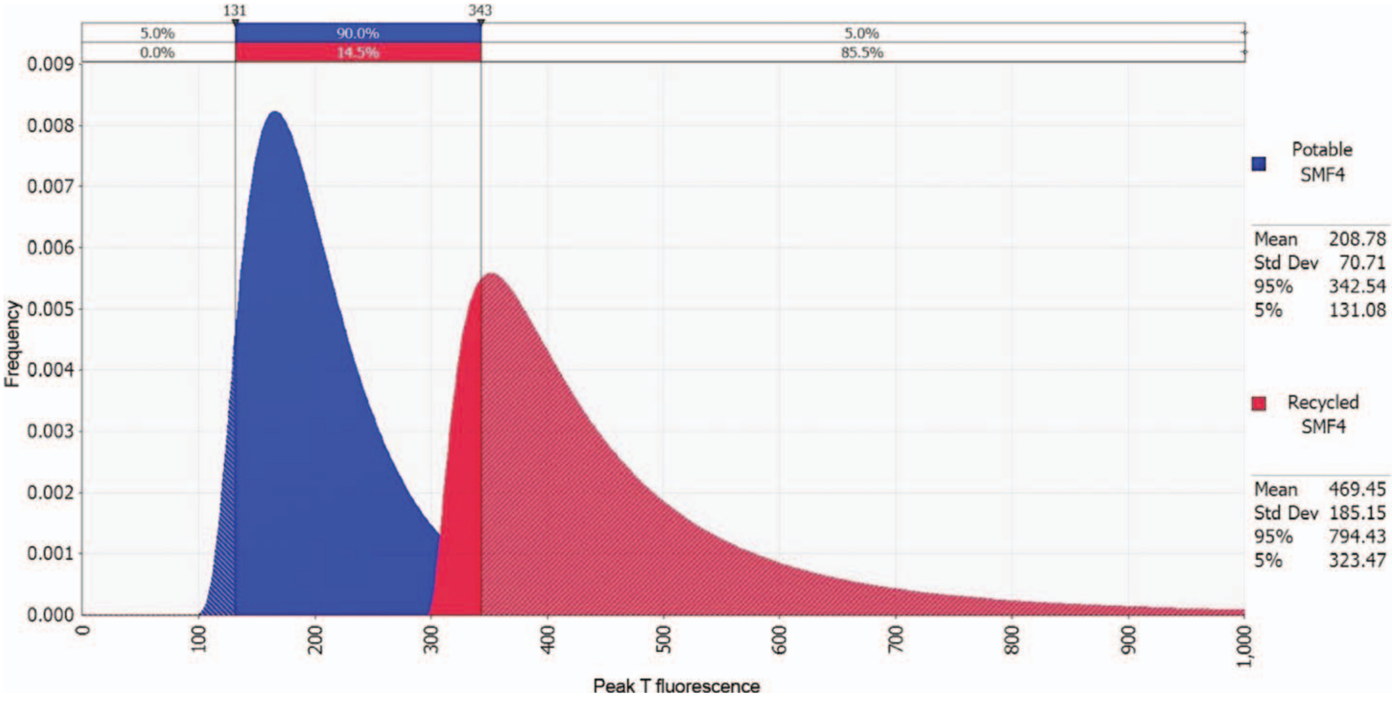

FIG. 2. Overlaid fitted distributions for the fluorescence of recycled and potable water from sampled houses at Aurora Estate and Highlands Estate.

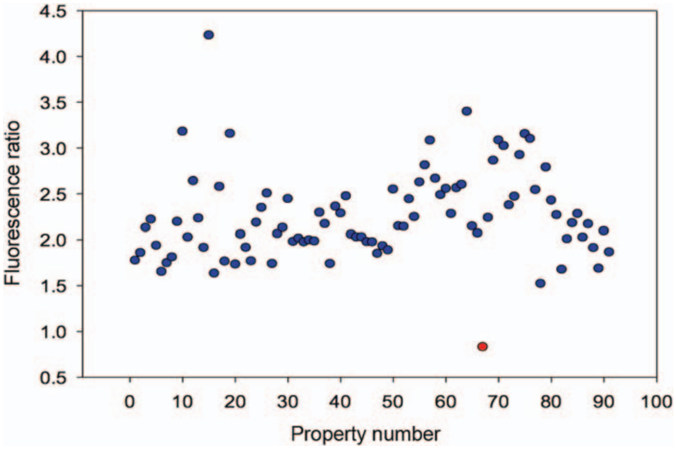

FIG. 3. Recycled water–potable water fluorescence ratio for sampled houses at Aurora Estate and Highlands Estate (N.B: single verified cross-connection is labeled in red triangle).

range of fluorescence intensities over the course of the study. However, since the composition of recycled water is variable throughout a distribution system and changes according to plant input, treatment performance, and usage, the thermal quenching effects of recycled water are also likely to vary between samples taken from across the distribution system. It is therefore more likely that the majority of the fluorescence variation seen is due to variation within recycled and potable water quality throughout the system.

Fluorescence Ratios. On examination of the ratio of recycled to potable water fluorescence, more consistent results were obtained with ratios between 1.52 and 4.23 over all the properties tested with correctly connected pipes, indicating recycled water fluorescence intensity was higher relative to potable water fluorescence intensity for every sampled property (Fig. 3). By using this ratio technique, a single cross-connection event was identified (property number 68; fluorescence ratio of 0.83) at a dual reticulated property during the field survey (Fig. 3). This was confirmed to be a cross-connection using the conventional method of turning off each water source individually and testing for flow.

Assessing Trigger Values. In order to identify a suitable ratio to reliably distinguish real cross-connections from normal variability, the calculated fluorescence intensity ratios of recycled to potable water at each property were fitted to a probability density function (Fig. 4) as previously described. This revealed that the ratio of recycled water to potable water was less than 1.7 for a randomly selected property within the dual reticulation system 5% of the time (5th percentile, 1.696; Fig. 4). By further analyzing the fitted PDFs, it was observed that a fluorescence ratio of 1.52 or less will occur for 0.1% of the fluorescence ratio distribution. This means that for a properly connected dual reticulation system (that is, in the absence of cross-connections) approximately 1 in 1000 properties will return a fluorescence ratio of 1.52 or less. Ultimately, if a recycled water to potable water fluorescence ratio of less than 1.52 is decided to warrant "further investigation," then the likelihood of a false positive occurring is 1 per 1000 properties (Fig. 5), representing a 0.1% rate of false positives. However, care should also be taken not to select a trigger value that is too low, since this will increase the probability of incurring a false negative, which in this instance should be considered as being of greater consequence than a false positive. Unfortunately, it is not currently possible to statistically describe the likelihood of false negatives at specific trigger values, since a large number of actual cross-connections would be required to collect sufficient data for this.

Distribution Sampling Requirements. It is important to note that the typical values and variation in recycled water fluorescence may differ between water recycling

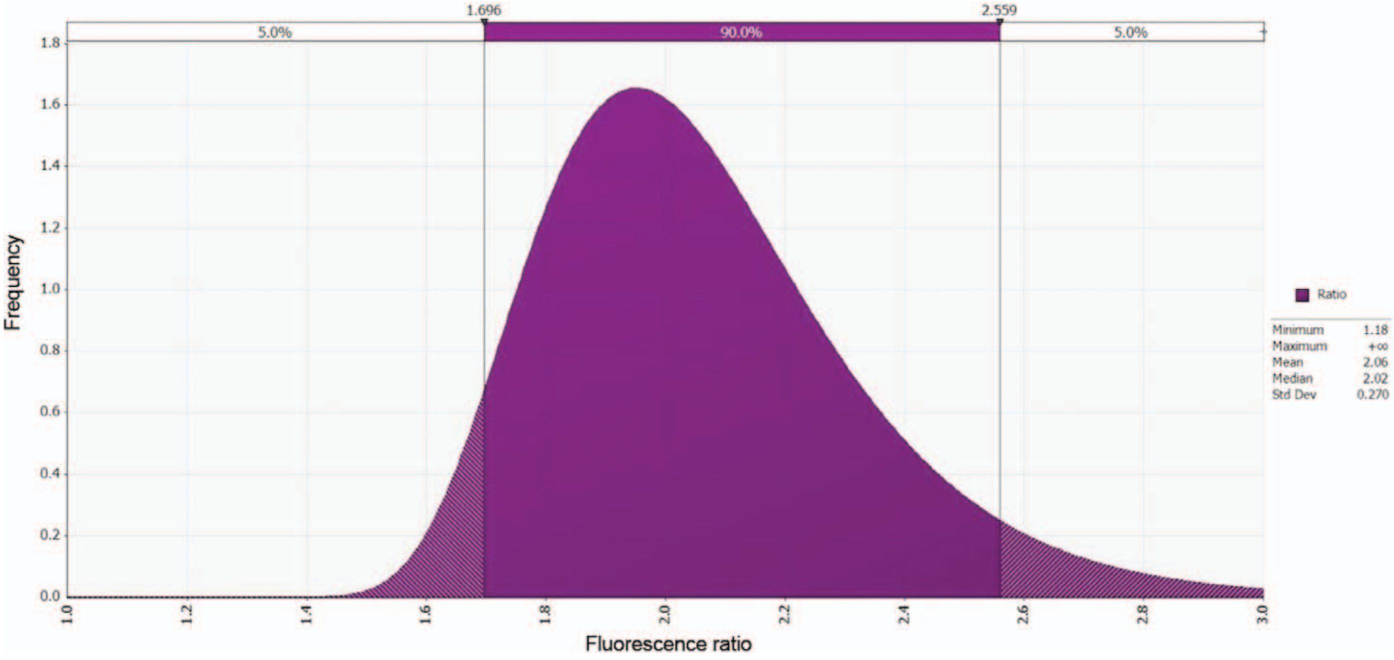

FIG. 4. Fitted PDF of recycled water to potable water fluorescence ratio for dual reticulated properties at Aurora Estate and Highlands Estate.

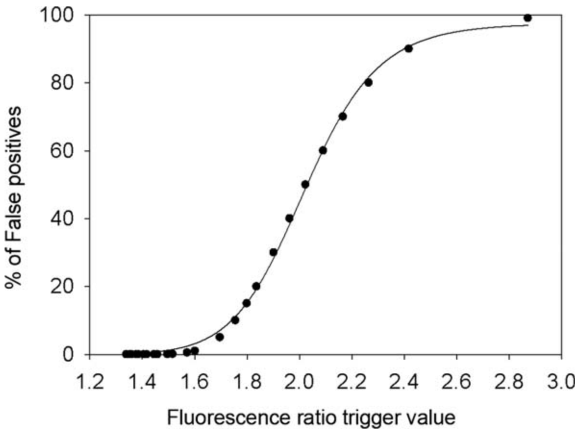

FIG. 5. Calculated "trigger values" for recycled to potable water fluorescence ratio and the percentage of false positives for which they represent.

plants due to factors such as different treatment trains, water quality input, and distribution system retention times.[28,32] As such, this means that the appropriate trigger value for portable fluorescence analysis may also vary between plants, as well as diurnally and seasonally in response to changes in recycled water fluorescence. These factors should be taken into account if a fluorescence ratio monitoring system were to be put in place. The appropriate trigger values for this field study were shown as a result of 91 properties being sampled and analyzed; however, a smaller number of properties sampled may be sufficient at other dual reticulation systems in order to establish trigger values. To evaluate this, random sections of the data were removed (25, 34, 50, and 67%) and refitted to probability density functions and then compared with those constructed with the full dataset (Table II).

The fitted distributions obtained using only 75, 66, and 50% of the data remained very similar to that constructed with the entire dataset, with very little change in the mean and standard deviation. However, when 67% of the dataset was removed, the fitted PDF changed significantly. Specifically, the mean fluorescence ratio decreased to 1.7, and the standard deviation increased to 2.6, resulting in a broadening of the distribution. Two significant conclusions can be drawn from these results. The first is that the number of properties sampled (91) was adequately representative in this case, since halving the number of samples did not have a significant effect on the distribution. Furthermore, this indicated that at least 45 properties were required to be sampled at this particular dual reticulation system (which represents approximately 2.5% of the total system) in order to establish a reliable trigger value. Though this value would ultimately depend on the size of the network, this is a good indication that a relatively low proportion of the dual reticulation distribution system needs to be sampled in order to establish appropriate trigger values and enable reliable ongoing cross-connection detection using portable fluorescence analysis.

TABLE II. Comparison of PDFs constructed with 33, 50, 66, 75, and 100% of surveyed properties.

Data inclusion	Mean	SD	5th percentile	95th percentile
100%	2.06	0.270	1.696	2.56
75%	2.22	0.418	1.604	2.96
66%	2.21	0.411	1.609	2.94
50%	2.31	0.537	1.659	3.32
33%	1.70	2.640	0.150	5.69

CONCLUSIONS

- Cross-connection detection is an area of water recycling network monitoring in which a clear solution is yet to be established. The need for a rapid, portable monitoring device exists to discern recycled water and drinking water from one another and therefore monitor dual distribution water recycling networks for cross-connections in a practical and reliable manner. For the first time, we have shown that portable fluorimetry is one such way of achieving this goal.
- Portable fluorescence was able to uncover a real cross-connection. In this instance, the presence of the cross-connection was already known to the property residents; however, this clearly demonstrates the potential for use of portable fluorimeters in the detection of cross-connections.
- Although the extent to which recycled water can be detected within drinking water may vary significantly between different networks, appropriate trigger values can be established to alert water companies whether further investigation of the piping may be required through an initial survey of as little as 2.5% of the network.

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