HYDRAULIC CHARACTERISATION OF A CONSTRUCTED WETLAND USED FOR NITROGEN REMOVAL VIA A DUAL-TRACER TEST

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ABSTRACT

A dual-tracer study was conducted using Bromine (Br) and Rhodamine WT (RWT) to determine hydraulic characteristics of the Corridor Creek Wetland Filter (CCWF) at Ranger Uranium Mine, in the Northern Territory, Australia. CCWF is a free surface wetland consisting of a series of six cells separated by bunds with interconnecting weirs, and was constructed to remove nitrogen from the effluent of the mine's wastewater treatment plant (nearing completion). The reverse osmosis treatment plant is designed to discharge treated water containing up to 20 mg-N L^{-1} as ammonia and this must be reduced to concentrations around 2 mg-N L⁻¹ following passage through the wetland. Effective nitrogen removal within the CCWF requires oxidation of the ammonia to nitrate followed by denitrification and/or conversion of dissolved nitrogen species to particulate nitrogen through assimilation by plants and other organisms. A key output of the tracer study was measurement of the fluid residence time distributions within the CCWF, useful for future assessment of the efficiency of passive nitrification-denitrification processes within the wetland. Simultaneous use of two tracers afforded economies in sampling, analysis and workload. RWT is easily detected but non-conservative, while Br is conservative but requires laboratory quantification. RWT serves as an inexpensive indicator for Br breakthrough, reducing the number of Br samples and analyses required. Both tracers behaved conservatively during the first ten days of the study with no appreciable loss of RWT fluorescence from either adsorption or photodegradation of the dye as it passed through the first four heavily vegetated wetland cells. RWT fluorescence decayed at a rate of 2.2 - 2.7% per day as the dye passed through the last two cells which consisted of large open water areas with little appreciable shading. The nominal residence time of water in each cell (volume divided by discharge rate) matched the time for the peak tracer concentration to pass between cells. The mean residence time of water in each cell was approximately 50% longer than the nominal residence time. Overall, the wetland exhibited an effective dispersivity of approximately 2000 m² d⁻¹ which situates the CCWF towards the more dispersive end of the range of free water surface wetlands.

1. INTRODUCTION

Water management is a key operational issue for Ranger Uranium Mine, situated in the Northern Territory, Australia. The normal operations of the mine produce effluents that must be treated to remove impurities before the resulting fluids can be disposed of in the surrounding environment. The site experiences large quantities of rainfall during the annual wet season and significant inventories of moderate to poor quality waters are stored on site. Management, treatment and disposal of the wastewater inventories are central components of the site operating strategy. The Corridor Creek Wetland Filter (CCWF) was designed to perform a crucial final polishing of reverse-osmosis permeates released from the Ranger wastewater treatment plant.

This paper describes results from a dual-tracer test designed to quantify hydrodynamic residence time distributions and dispersion phenomena within the wetland. These results will be used to assist the later measurement of net nutrient polishing efficiency, required to underpin the environmental management plan for the mine.

Corridor Creek Wetland Filter

CCWF is an engineered wetland of total area just over 5 ha, to the south of the main operations area at the Ranger mine site. It is constructed in the drainage line of the Corridor Creek system, which drains eastwards towards Georgetown Billabong and ultimately to Magela Creek. CCWF contains six cells, as shown in Figure 1, defined by bunded walls and connected in series by shallow spillways. The wetland is fed by a treated wastewater stream emitted by the Ranger wastewater treatment plant. Presently this wastewater stream arises from treatment of high-quality pond water at the mine, and has a relatively low nutrient content, but as the wastewater treatment strategy expands to incorporate lower quality process water the nutrient concentration may rise to approximately 15 mg L⁻¹ NH₄ (ammonia). The intended function of CCWF is to attenuate the NH₄ concentration to below the environmental regulatory target of 2 mg L⁻¹ for discharge to natural waterways.

The CCWF incorporates shallow, relatively aerated and in some places densely vegetated cells (Cells 1-4) to assist in removing sediment and nutrients from the water stream and also to nitrify ammonia to nitrate. Subsequent cells are larger and deeper to promote denitrification (conversion of nitrate to gaseous nitrogen) which is normally associated with anaerobic microbial processes. Nitrification is normally regarded as a relatively rapid process, whilst denitrification is often a slow process, so fluid residence time can be relatively short in Cells 1 and 2 but may need to be much longer in Cells 5 and 6 to promote efficient conversion.



Figure 1. Schematic of the Corridor Creek Wetland Filter, with idealized flow paths indicated.

2. DUAL TRACER TEST DESIGN

Bromide (as NaBr) and rhodamine WT (RWT) were selected as the two tracers for the test. Of the two tracers, bromide has a more conservative nature, as RWT is known to adsorb onto organic surfaces and to decay photolytically (Dierberg and DeBusk, 2005; Wilson *et al.*, 1986). However, there are drawbacks to using bromide in natural systems. Bromide is typically used in trace concentrations (to avoid undue ecological disturbance) and is invisible to the human eye. Because of the low concentrations accurate measurement in the field is problematic, so fluid samples must be collected for later analysis. The analytical costs are significant, so there is a need to identify only those samples which must be analysed to minimize costs. In comparison, RWT is easy to measure accurately using *in situ* fluorometers and is therefore the better tracer for providing high temporal resolution. Collecting samples for bromide analysis based on the presence of RWT signals achieves the objectives of minimizing sample handling and analysis costs, whilst maximizing temporal and spatial resolution.

Environmental Effects of NABR and RWT

The effect of NaBr on aquatic flora and fauna is well studied, but variations in estimated ecotoxicological thresholds between different studies are not unusual. For common green algae species, NaBr average toxicity (EC50) concentrations over intervals of a few days are as high as 5-20 g L⁻¹ with a no observable effect concentration (NOEC) > 1 g L⁻¹ (USEPA, 2009). For *Daphnia* populations NOEC is 1-10 g L⁻¹ over several days, and over periods of several weeks NOEC is 10-90 mg L⁻¹ (USEPA, 2009). High concentrations of NaBr have been shown to be associated with pathological effects in several freshwater fish species (see, e.g. Wester *et al.*, 1988). These authors estimate the NOEC for guppy and high-eye species exposed to NaBr as 32 mg L⁻¹ over a one month exposure time. For exposure durations shorter than one month, these authors found NOEC levels to increase meaning that fish species can tolerate higher levels of NaBr for short periods of time. This finding was supported by later work by Stormer *et al.* (1996) who found that rainbow trout (*Oncorhynchus mykiss*) were able to tolerate exposure to 80 mg L⁻¹ for 14 days with the only observable effect being an increase of blood plasma Br⁻ and Cl⁻ concentrations. USEPA (2009) data show that the NOEC level is of the order of 250+ mg L⁻¹ (three day exposures) for common fish indicator species. Molluscs and common plants are also reported to be resistant to high NaBr concentrations for durations of several weeks (USEPA, 2009). For these reasons, it is not anticipated that there will be significant side-effects of the use of NaBr as a dissolved phase tracer in CCWF at the concentrations planned and for the intended duration of the tracer trial (of the order of one month).

Bencala and Cox (2005) list US EPA standards for RWT concentrations in the environment (as of August 2004) as 10 μ g L⁻¹ for water entering a drinking water plant and 0.1 μ g L⁻¹ for drinking water. The National Sanitation Foundation Standard 60 states a maximum use concentration of 0.1 μ g L⁻¹. A working group from the German Federal Environmental Agency recommends against using RWT based on their findings of a genotoxic effect (i.e. RWT was found to cause genetic mutations in salmonella and in a mammalian cell culture) (Behrens *et al.*, 2001). However, they did not observe any ecotoxicological effects at concentrations up to 10 mg L⁻¹. Wilson *et al.* (1986) cite a range of studies on the possible toxicity of RWT to aquatic life and found no evidence of problems with oyster eggs and larvae, silver salmon, Donaldson trout nor a range of macroinvertebrates for concentrations of 10 mg L⁻¹, and often much higher, for exposure periods of 1-7 days. There are precedents for the use of RWT at Ranger. Smith *et al.* (1986) report on a tracer study conducted in Magela Creek during 1978 in which 80 L of 20% RWT solution was introduced to Magela Creek approximately 1 km downstream of Georgetown Billabong. This produced concentrations as high as 225 μ g L⁻¹ near Coonjimba, 160 μ g L⁻¹ near Gulungul, and 40 μ g L⁻¹ at Mudginberri. A similar trial was repeated in 1979 by the Australian National University CRES group (50 L dye addition). In 1986 a tracer study by OSS introduced RWT and several radionuclides (including ¹⁹⁸Au, ⁹⁹Tc, ³H) to Magela Creek (G. Douglas, pers. comm.). Due to the absence of any recorded ecotoxicological effects even at concentrations orders of magnitude greater than were employed for this study in the Corridor Wetland trial, the use of RWT as a tracer is considered acceptable.

Conservation of Tracers

Because it is a simple ion, bromide is unlikely to attenuate through sorption or photolytic decay, and is regarded as essentially conservative in surface water environments. However, there are several discussions in the literature regarding the non-conservative behaviour of RWT when used in Australian settings. Smith (1978) conducted a RWT dispersion test in a series of 5 linked lagoons at Tidbinbilla Nature Reserve in the Australian Capital Territory. Flow through the system was slow (5 L s⁻¹) and the experiment took place over more than 37 d. Only 16% of the original tracer injected was recovered at the end of the system. Tracer mass balances showed losses of 34% over 23 days in lagoon #2 and 43% over 37 d in lagoon #3, or about 1.2-1.5% per day. Smith speculated that this loss was most likely due to adsorption onto organic matter. In the Magela Creek Study, Smith et al. (1986) claim essentially conservative behaviour of RWT during the first 21 h. Although their mass balance suggested a loss of about 4.5% of RWT, uncertainty regarding the completeness of mixing and noise in the data could have produced an error of this magnitude. Perhaps the most thorough examination of RWT conservation issues is provided by Dierberg and DeBusk (2005) who used mesocosms containing cattails and submerged aquatic vegetation to assess RWT losses by adsorption onto mineral and organic surfaces as well as photolytic decay. Test conditions were characterised by strong insolation, shallow depth (0.10 m) and high temperatures (32 – 42 °C). They measured a photolytic decay rate of 0.139 d⁻¹ in direct sunlight and 0.027 d⁻¹ in the shade compared to a range of 0.0024 - 0.048 d⁻¹ reported in other literature. RWT sorbed onto both mineral and organic substrates with sorption onto sediment surfaces increasing linearly with organic content. Sorption on sunny samples was 13-25% and onto shady samples it was 33-59%. The net effect was that photolysis plus sorption yielded similar losses of tracer. Based on all of the above experiences, a decay of 3% per day in RWT was anticipated to be a reasonable estimate of the likely loss of tracer during the CCWF tracer test.

Background Fluorescence, Thermal and Photodegradation Effects

Measurement of the background fluorescence in each cell is essential in order to facilitate compensation of the measured fluorescence for the effect of dissolved organic matter (DOM) fluorescence. Based on the experience of Smith *et al.* (1986), background fluorescence was expected to be in the range of 0.02-0.07 μ g L⁻¹ and likely to have negligible impact on this study given the proposed concentration levels for the dye. The background fluorescence was measured in each of the six wetland cells on 8 May 2008 prior to the injection of any tracers. Background fluorescence in the six cells (after blank adjustment) ranged from 0.1 to 1.8 μ g L⁻¹, and was seldom more than a few percent of the peak tracer concentration in any cell.

The thermal sensitivity of RWT fluorescence was directly measured as part of the CCWF tracer test as follows. Two 2 L samples were collected at the exit to Cell 2 to assess both the photodegradation of RWT fluorescence over time and the temperature sensitivity of fluorescence. The containers were made of clear glass with a plastic-lined metallic lid. One container was wrapped in duct tape to provide a dark environment, henceforth referred to as the 'dark sample'. Both containers were left exposed to full sun on the land adjacent to the wetland for the duration of the experiment. This results in much higher exposure to ambient light than is experienced within the wetland and therefore represents an upper bound on the photodegradation likely to be experienced by dye in the wetland. During the course of the day it is possible that the containers experienced some periods of shade (or filtered light) as opposed to full sun due to the surrounding vegetation. On 11 and 22 May 2008 the temperature sensitivity of fluorescence was measured by placing the fluorometer into a sample (light or dark) and then placing the sample in an ice water bath and recording temperature

and fluorescence during approximately 10 minutes as the samples cooled. The temperature correction factor, C_{f_5} was determined by regressing the ratio of fluorescence at 25 °C, RWT_{25} , and the sample fluorescence, RWT, against the difference in temperature, ΔT , between 25 °C and the sample temperature,

$$\frac{RWT_{25}}{RWT_{somple}} = 1 - C_f \Delta T$$

where $\otimes T = 25 - T_{sample}$. Both dark samples exhibited a similar linear temperature dependence between 15 °C and 35 °C, (-10 < ΔT < 10 °C), and conform with the data of Wilson *et al.* (1986) whose data gave a slope of -0.027 for a best fit linear regression (see Figure 2).

The photolytic stability of the fluorescent tracer is shown in Figure 2 which compares the measured fluorescence of light and dark samples over a period of 29 days. The light sample decayed at a rate of 6% per day whereas the dark sample lost only 0.3% of its fluorescence per day. In the field, the loss of fluorescence is expected to be substantially less than the light sample because of attenuation of sunlight within the water column. The light sample was exposed to much higher irradiance than would be the case in the wetland.



Figure 2. Temperature sensitivity (left) and photodegradation (right) of Rhodamine WT fluorescence. A procedural error in the field is evident in the temperature data from the light sample on 22 May 08.

3. CONDUCT OF THE DUAL TRACER TEST

Concurrent use of two tracers, RWT and NaBr, was employed to measure the hydraulic characteristics of CCWF. To ensure a high signal to noise ratio, 5.24 kg of NaBr was dissolved into approximately 50 L of permeate water on 7 May 08. The corresponding mass of Br was 4.07 kg. The fluorescent tracer was made by adding 2 L of RWT concentrate (20%) to permeate water to make up a total volume of approximately 50 L, approximating a RWT total mass of 0.4 kg. The tracer was injected into the permeate discharge pipeline beginning at 11:00 h on 8 May 08 and continued until the pump was turned off at 11:15 h with approximately 12 L of stock solution remaining. The tracer first arrived at the wetland at 11:50 h and was virtually exhausted by 12:03 h. The discharge of permeate from the water treatment plant during the tracer study was maintained as close to $0.0343 \text{ m}^3 \text{ s}^{-1}$ as possible. A record of the discharge is shown in Figure 3. Periods of higher flow occurred when the 3rd RO unit was brought on line. Over the entire test the average flow to the wetland was $0.0347 \text{ m}^3 \text{ s}^{-1}$. All flow to the wetland ceased at 15:30 h on 5 June 08 when the WWTP discharge was redirected to another location. Figure 4 shows photographs of the progress of the RWT through CCWF.







Figure 4. Aerial photography of propagation of tracer through CCWF Cells 1-4. No tracer is visible 1 h after its arrival in Cell 1 (top left cell) due to very dense *Eleocharis* stands. On 9 May the tracer is mainly in Cell 2 although some introduction into Cell 3 is visible. On 10 May the top half of Cell 2 is clear and most of the tracer is in Cell 3 with some tracer present in Cell 4 upstream of submerged berm. On 11 May the dominant tracer mass is visible in the downstream half of Cell 3 and in Cell 4.

RWT Measurements

RWT concentrations were measured using 2 Turner Cyclops-7 rhodamine fluorometers. The fluorometer data were logged using Datataker 505 and Datataker 85 dataloggers. Secondary temperature standards (Thermometrics CSP) were attached to each of the fluorometers to provide accurate (± 0.01 °C) measurements of *in situ* water temperatures. All fluorometers were calibrated against stock dye solutions. Fluorescence measurements during the tracer test were corrected for background fluorescence and thermal effects. The data loggers and a 12 V battery were housed in protective cases (Pelican) with environmental seals to allow unattended operation in the field. The data loggers were programmed to sample the fluorometer output voltage and the temperature sensor every 2 s and record the average values of 15 samples every 30 s. The fluorometer systems were deployed in the exits to adjacent wetland cells. Shortly before the tracer was expected to arrive at the next unmonitored downstream cell, the most upstream system was redeployed (leap frogging) into the exit of the next unmonitored cell.

BR Samples

Water samples for Br (and RWT) analysis were collected using ISCO automated samplers. No ISCO sampler was deployed at the exit to Cell 3 because of relatively difficult access. Each sampler pumped samples from the spillways between wetland cells at the same location as where the fluorometers were deployed. The frequency of sample collection varied between 1 and 4 h, typically, with a higher sampling frequency employed during periods when tracer concentration changes were most rapid and longer periods used to sample the tails of the concentration time series. The sampling period was increased to 8 - 12 h for Cells 5 & 6 towards the end of the experiment as fluorometer readings confirmed very slow changes in concentration following the passage of the concentration peak. Br analysis was performed by the Northern Territory Envrionmental Laboratory using inductively coupled plasma mass spectrometry (ICP-MS).

Density Effects

The RWT-Br tracer concentrate is denser than water. It is essential to minimise the density differences to ensure that the tracer mixes with the water mass rather than forming a density current that moves through the wetland differently from the main water mass. The possible formation of a density current can be mitigated by ensuring that the tracers are well mixed with the influent flow prior to introduction to the wetland. The density of the stock tracer can be estimated from the specific gravity (s.g.) of its constituents. A 20% concentrate of RWT has a s.g. of 1.19. The concentration of NaBr was approximately 10% which has a s.g. of 1.08 (density of sodium bromide from Mettler Toledo (2009)). Combined, the stock tracer solution is estimated to have a s.g. of 1.0842. If injected over 10 min the stock tracer would be diluted by 20000 L of permeate (assumed s.g. of 1.0) to produce a s.g. of 1.0002 at the point of introduction to Cell 1. The difference in s.g. between the injected tracer and the receiving water is equivalent to the density difference caused by a ~0.7 °C change in temperature. Neglecting entrainment of ambient water in Cell 1, the dyed inflow can be expected to form an underflow during the afternoon. The normal diurnal heating and cooling cycle produces water temperature changes of 2 °C or more so we can assume that any underflow would be entrained into the overlying water column as a result of penetrative convection during night time cooling. Afternoon injection reduces the chances of incomplete night-time mixing because the initial entrainment of ambient water will occur at the warmest temperature. There is a negligible chance of incomplete mixing of the tracer within the wetland.

4. TRACER DISPERSION

Tracer breakthrough at the six wetland cell exits is approximated by equation (1):

$$\frac{\partial c_i}{\partial t} = D_i \frac{\partial^2 c_i}{\partial x^2} - \frac{Q}{A_i} \frac{\partial c_i}{\partial x}$$

(1)

where the index *i* denotes the wetland cell (i = 1, 2, ..., 6), Q = Q(t) is the water flux through CCWF, c_i is the tracer concentration measured at the outlet of Cell *i*, A_i is the effective cross-sectional area of Cell *i* and D_i is the effective dispersion coefficient of Cell *i*. The dispersion coefficient for a wetland context is discussed by Kadlec (1994). Solutions of the dispersion equation yield tracer breakthrough curves which contain information on hydrodynamic fluxes and residence times in the wetland. These quantities are conveniently extracted from the breakthrough curves using statistical moment analysis. A simple metric is the nominal detention time, t_{nom} , or mean residence time, which can be expressed for any water body of volume *V* subject to a flow rate *Q* by $t_{nom} = V/Q$, thus yielding a nominal CCWF (volume 38477 m³) residence time of 13.4 days for a WWTP discharge rate of 2.88 ML d⁻¹.

5. RESULTS

Results for Cells 1,2,4 and 6 are shown in Figure 5. In Cell 1, the RWT and Br data overlap closely. Peak concentration occurred at 21:54, 10 h after the dye was introduced. The presence of a second peak at 06:25 h on 9 May 08 suggests either the presence of a second, more dispersive, flow path through this cell or the possible formation of a dense underflow which was mixed throughout the water column during nighttime mixing caused by penetrative convection. By 11 May 01:00 h, 61 h after introduction, 95% of the tracer had passed through Cell 1. The maximum concentration of Br observed in Cell 1 was 4 mg L⁻¹, which is significantly less than ecotoxicity levels discussed earlier. In Cell 2 the peak concentration occurred at 19:50 on 9 May 08, 149 h after introduction and 32 h after the peak passed through Cell 1. By 08:00 h on 15 May 08, 95% of the tracers had passed through Cell 2. This is 164 h after the start of the test. The concentration peak is much more dilute and broader than was observed in Cell 1. Curves for Cell 6 show the concentration peak arrived at around 00:00 on 24 May. The experiment concluded before the full breakthrough curves in Cell 6 could be determined and the mass estimates rely on extrapolation of the breakthrough curves. Loss of RWT fluorescence is prominent with decreases of about 15% on 18-19 May in Cell 5 (not shown) and of 33% on 23-25 May in Cell 6.



Figure 5. Continuous (30 s) RWT concentration (black line) and Br sample values (blue circles) for CCWF Cells 1,2,4,6. Yellow band denotes the period when flow into Cell 1 was reduced by half. Moment analysis data are presented in Table 1. Here the Peclet number Pe is defined as Pe = u L/D where u is the mean velocity, L is the distance travelled and D is the dispersion coefficient.

 Table 1. Tracer moment calculations for Corridor Creek Wetland Filter. Bold figures for Cells 5 and 6 denote results of calculations after adjusting RWT fluorescence for decay. *Extrapolated.

Cell	inject	1	2	3	4	5	6
1 st Appearance	8 May 11:51	8 May 19:49	9 May 04:00	9 May 17:30	10 May 09:00*	12 May 20:00*	14 May 00:00*
Peak time		8 May 21:54	9 May 19:50	11 May 02:20	12 May 13:00	17 May 09:00	24 May 00:00
95% time		10 May 15:00	15 May 08:00	17 May 16:08	19 May 22:00	2 June 14:30	10 June 01:00
Measurement End Time	8 May 12:03:34	13 May 12:00	27 May 12:40	27 May 15:30	27 May 12:30	13 June 00:00*	25 June 00:00*
RWT Mass (kg)		0.34	0.33	0.28	0.27	0.21/ 0.28	0.16/ 0.28
Br Mass (kg)		3.5	3.3	n.d.	2.7	2.8	2.8
Time to Peak (h)		10	32	62.5	97	123	180
Cell Travel Time (h)			22	30.5	34.5	26	47
Time to 95% (h)		51	164	220	274	603	781
Mean Residence Time to End of Cell τ (day)		0.9	2.3	4.1	5.8	13.4/ 14.2	19.3/ 20.4
Exit RWT Pulse Variance σ^2 (day ²)		0.5	5.6	6.6	7.2	33.3/ 37.1	41.1/ 43.9
Normalized Variance σ^2/τ^2		0.64	1.03	0.4	0.21	0.19/ 0.18	0.11/ 0.11
Peclet Number Pe		1.5		3.7	8.4	9.4/10	17/ 17
Dispersion coefficient $(m^2 d^{-1})$		6230		12400	2120	1450/ 1370	1930/ 1930

A loss of roughly 20% of tracer mass between the exits of Cell 2 and Cell 4 suggests the possibility of leakage from the wetland and is being investigated further. The cumulative nominal residence time (volume/discharge) for the wetland cells is quite similar to the measured time for the concentration peak to pass through each cell (Figure 6). Tracer first appears in each cell in slightly less than half this time. The mean residence time, i.e. the average time the tracer spends in the wetland, is approximately 50% longer than the nominal residence time and the time for 95% of the tracer to pass through the wetland is more than twice the nominal residence time. The first three cells of the wetland are extremely dispersive (dispersion coefficient, D, > 6000 m² d⁻¹). From the end of Cell 4 to the end of Cell 6, D remains approximately 2000 m² d⁻¹ which situates CCWF towards the more dispersive end of the range of free water surface wetlands described by Kadlec (1994).



Figure 6. Nominal residence and mean tracer residence times, time for tracer to initially appear, time to peak concentration and time for 95% of tracer to pass through each wetland cell. All data reflect the cumulative performance of the wetland from the WWTP discharge to the end of the cell shown.

6. CONCLUSIONS

The study described here has quantified the residence time distributions of the six cells in CCWF, and the overall residence time distribution of the wetland itself. The use of a dual tracer technique allowed efficient sampling and analysis to be performed, reducing costs considerably whilst also reducing uncertainty in the estimation of tracer breakthrough curves and fluid residence times. Concentration attenuation processes for the RWT tracer were studied and quantified.

For the flow conditions during the tracer study the mean residence time for the wetland was 19-20 days. Cells 1-3 had dense stands of macrophytes and filamentous algae which would be expected to provide a large surface area to support N removal during the first 5 days as water passes through these cells. Passage through Cells 5 and 6 is expected to take place through mainly open water over roughly 14 days and sediment contact is expected to be the dominant mechanism for transformation of nitrogen.

7. ACKNOWLEDGEMENTS

The authors gratefully acknowledge the assistance of EWLS and Ranger Miner staff with the design, arrangement and conduct of the dual tracer test. Particular thanks are due to Louise Frick, Amber Hooke and Michelle Iles.

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