Catchment and Drinking Water Quality Micro Pollutant Monitoring Program – Passive Sampling

Report 7 – Winter 2017

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Photo courtesy of Natalia Montero Ruiz

Project Team

Sarit Kaserzon, Kristie Thompson, Chris Paxman, Christie Gallen, Gebriele Elisei, Rachel Mackie, Michael Gallen, Tim Reeks, Hui Jiang, Geoff Eaglesham, Saer Samanipour and Jochen Mueller.

Direct Enquiries to:

Dr. Sarit Kaserzon

Queensland Alliance for Environmental Health Sciences (QAEHS)
Formerly National Research Centre for Environmental Toxicology (Entox)
The University of Queensland
20 Cornwall Street, Woolloongabba, Qld 4102
(p) +61 (0)428 532 053
(m) +61 (0)415 671 348
(f) +61 (0)7 3274 9003
(e) k.sarit@uq.edu.au (w) www.uq.edu.au

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Executive summary

The Catchment and Drinking Water Quality Micro Pollutant Monitoring Program was launched in mid 2014 with the aim of improving the characterisation and understanding of the micro-pollutant risk profile in source water reservoirs through annual summer and winter sampling campaigns. The monitoring program utilising passive samplers was continued in reservoirs in South East Queensland (SEQ) during August - December 2017 and represents the seventh of twelve sampling campaigns (targeting winter/summer from 2014 – 2020). Results presented provide a continued insight into the water quality of the target catchments and drinking water reservoirs.

A wide range of polar and non-polar organic contaminants of interest were targeted by two types of passive samplers and included herbicides, pharmaceuticals and personal care products (PPCPs), organochlorine pesticides (OCPs), other pesticides, and polycyclic aromatic hydrocarbons (PAHs). Sampler extracts were analysed at QAEHS by LC QQQ MS/MS, LC-QTOF MS/MS (polar compounds) and GC-MS/MS (non-polar chemicals) using the latest analytical methods and established protocols.

Chemical analyses of the passive sampler extracts detected a total of 76 different chemicals including 22 OCPs (and pesticides), 9 PAHs, 30 herbicides and insecticides and 15 PPCPs. OCPs were detected at all sampling locations, with endosulfan sulfate, pp-DDD and pp-DDE, being the most prevalent between sites and dacthal showing the highest total concentration. Total ∑OCP water concentrations were ≤ 16 ng L^{-1}. PAHs were detected at 92% of sites with fluoranthene > pyrene > chrysene present at the highest concentrations. Fluoranthene was the most abundant, followed by pyrene. Total ∑PAH water concentrations were ≤ 3.3 ng L^{-1}. Herbicides/insecticides were detected at all sampling locations. The triazines: atrazine > simazine were present in high abundance and/or concentration, as well as diuron and metolachlor. Total estimated ∑herbicide water concentrations for herbicides were ≤ 145 ng L^{-1}. Low levels of fifteen PPCPs were detected in the passive samplers. Water concentrations were above the limit of reporting (LOR) for DEET, carbamazepine, caffeine, codeine and hydrochlorothiazide. DEET and salysilic acid were both detected at 83% of sites, followed by hydrochlorothiazide 39% and carbamazepine 28%. Total estimated ∑PPCP water concentrations were ≤ 26 ng L^{-1}, when excluding DEET levels of 80 ng L^{-1} found at one site.

Drinking water guidelines are available for some of these chemicals, but no chemicals were present in concentrations that exceeded these guidelines. Guidelines for freshwater aquatic systems are also available for some chemicals. The pesticide chlorpyrifos exceeded the 99% freshwater species protection guidelines (0.04 ng L^{-1}) at all sites where it was detected, but not the 95% freshwater species protection guideline (10 ng L^{-1}).

1. Introduction

As the bulk supplier of potable water to South East Queensland and in order to safeguard the regions drinking water sources and ensure water quality is maintained, Seqwater has sustained a *Catchment and Drinking Water Quality Micro Pollutant Monitoring Program*. The aim of this program is to identify and understand the presence of micro-pollutants in the water reservoir areas as well as to recognise any spatial and temporal trends of micro pollutants. An extension of this program has been introduced to include the use of passive sampling technologies in the monitoring of water storages over a six year period (2014 – 2020; summer and winter sampling campaigns), in order to accurately assess the risk they may pose to drinking water quality. The scope of work for this project includes the deployment of passive sampling technologies in two routine sampling campaigns (summer and winter) a year, over a three year period. In addition, passive samplers may be deployed at sites when required to measure specific high rainfall or event periods.

The typically low level concentrations of micro-pollutants present in environmental waters makes sampling methods such as grab sampling challenging, as 1 L grab samples often may not offer sufficient volume for concentration and detection of micro-pollutants and episodic contamination events may be missed when collecting single samples that provide a single point in time estimate of water quality. The use of passive sampling technologies have been introduced to complement and overcome some of these challenges, substantially improving the ability of monitoring chemical pollutants in liquid phases over the last 15 - 20 years. Some of the benefits of passive sampling tools can include *in-situ* concentration of chemical pollutants, increased sensitivity and the provision of time-weighted average concentration estimates for chemicals over periods of ≥ 1 month, increased data resolution and risk profiling using a robust scientific methodology. Passive samplers designed to monitor non-polar (i.e. using polydimethylsiloxane or PDMS) as well as polar (using Empore Disk or ED) chemical pollutants have been chosen for deployment.

The list of target chemicals for inclusion in the monitoring campaign has been identified following a review of all Australian Drinking Water Guideline and Australian and New Zealand Environmental Conservation Council listed parameters and was narrowed down based on an assessment of their possible application in the catchment areas, and assessed from Australian Pesticides and Veterinary Medicines Authority (AVMPA) registered products applications, as well as water solubility and guideline values. This report presents data from the seventh monitoring campaign.

2. Methodology

Passive water samplers were deployed in 36 SEQ reservoirs/waterways from August to December 2017 over a period of between 28 - 33 days (Table 1). The deployment of samplers was conducted in alignment with "Drinking and Catchment Water Quality Micro-pollutant Passive Sampling Procedure" (27 May 2014).
Two types of passive samplers were deployed at each site. Empore Disk™ (EDs) samplers to detect the presence of polar chemicals such as herbicides, and pharmaceuticals and personal care products (PPCPs), and polydimethylsiloxane (PDMS) strips (deployed in stainless steel cages) to detect the presence of non-polar chemicals such as certain organochlorine pesticides (OCPs) and polycyclic aromatic hydrocarbons (PAHs). Passive flow monitors (PFMs) were co-deployed with the passive samplers at each site to estimate water flow conditions at each site during sampler deployment. Table 2 below lists the deployment site locations, site numbers, site codes, dates and lengths of deployment periods, as well as the water velocity measured at each site.

Table 2 Deployment locations, dates and lengths of deployment periods and water velocity measured at each site

SITE #	SITE CODE	SITE NAME	DATE DEPLOYED	DATE RETRIEVED	DAYS DEPLOYED	FLOW VELOCITY (CM S^{-1})	COMMENTS
SEQ1*	MRS-SP012	SEQ-MARY RIVER @ COLES CROSSING	24/08/2017	21/09/2017	28	1.52	PDMS replicate only. Minimum flow of 3.4 used in water concentration estimates (E
SEQ2	LMD-SP001	SEQ-LAKE MACDONALD INTAKE	28/08/2017	25/09/2017	28	5.24	
SEQ3	BOD-SP001	SEQ-BORUMBA DAM	24/08/2017	21/09/2017	28	6.52	
SEQ4	MRS-SP013	SEQ-MARY RIVER @ KENILWORTH	21/11/2017	19/12/2017	28	17.14	Deployed late due to access issues. PFMs both empty.
SEQ5	POD-SP001	SEQ-POONA DAM	23/08/2017	20/09/2017	28	2.86	Minimum flow of 3.4 used in water concentration estimates (EDs)
SEQ6	SOR-SP001	SEQ-SOUTH MAROOCHY INTAKE WEIR	23/08/2017	20/09/2017	28	1.55	Minimum flow of 3.4 used in water concentration estimates (EDs)
SEQ7	YAC-SP001	SEQ-YABBA CREEK @ JIMNA WEIR	23/08/2017	20/09/2017	28	1.02	Minimum flow of 3.4 used in water concentration estimates (EDs)
SEQ8	BPD-SP001	SEQ-BAROON POCKET DAM	28/08/2017	25/09/2017	28	4.25	
SEQ9*	EMD-SP001	SEQ-EWEN MADDOCK INTAKE	28/08/2017	25/09/2017	28	5.15	
SEQ10	SOD-SP010	SEQ-KILCOY WTP OFFTAKE	11/09/2017	9/10/2017	28	3.54	
SEQ11	SOD-SP011	SEQ-KIRKLEAGH	11/09/2017	9/10/2017	28	5.03	
SEQ12*	SOD-SP001	SEQ-SOMERSET DAM WALL	11/09/2017	9/10/2017	28	4.99	PDMS replicate site only.
SEQ13*	WID-SP004	SEQ-WIVENHOE DAM @ ESK PROFILER	29/09/2017	27/10/2017	28	5.13	ED replicate site only.
SEQ14	WID-SP001	SEQ-WIVENHOE DAM WALL @ PROFILER	29/09/2017	27/10/2017	28	9.22	
SEQ15	LOC-SP034	SEQ-LOCKYER CREEK @ LAKE CLARENDON WAY					Site not active
SEQ16	LOC-SP031	SEQ-LOCKYER CREEK @ O'REILLYS WEIR	14/09/2017	12/10/2017	28	2.85	Minimum flow of 3.4 used in water concentration estimates (EDs)
SEQ17	MBR-SP016	SEQ-LOWOOD INTAKE	14/09/2017	12/10/2017	28	8.00	
SEQ18	MBR-SP001	SEQ-MID BRIS RIVER @ MT CROSBY WESTBANK OFFTAKE TOWER	6/09/2017	4/10/2017	28	3.15	
SEQ19	NOD-SP091	SEQ-NORTH PINE RIVER @ DAYBORO WELL	5/09/2017	3/10/2017	28	2.64	Minimum flow of 3.4 used in water concentration estimates (EDs)
SEQ20	NOD-SP001	SEQ-NORTH PINE VPS	5/09/2017	3/10/2017	28	5.32	
SEQ21	LAK-SP001	SEQ-LAKE KURWONGBAH	5/09/2017	3/10/2017	28	7.19	
SEQ22	NOD-SP023	SEQ-NORTH PINE RIVER @ PETRIE OFFTAKE	13/09/2017	11/10/2017	28	2.35	Minimum flow of 3.4 used in water concentration estimates (EDs)
SEQ23	NSC-SP001	SEQ-HERRING LAGOON	21/08/2017	18/09/2017	28	1.96	Minimum flow of 3.4 used in water concentration estimates (EDs)
SEQ24	LHD-SP005	SEQ-LESLIE HARRISON DAM	13/09/2017	11/10/2017	28	4.30	
SEQ25	WYD-SP001	SEQ-WYARALONG DAM WALL	27/09/2017	26/10/2017	29	4.58	
SEQ26	MOD-SP027	REYNOLDS CREEK @ BOONAH	27/09/2017	26/10/2017	29	1.68	Minimum flow of 3.4 used in water concentration estimates (EDs)
SEQ27	MOD-SP002	SEQ-MOOGERAH DAM @ OFFTAKE	27/09/2017	26/10/2017	29	3.98	
SEQ28	LRS-SP017	SEQ-LOGAN RIVER @ KOORALBYN OFFTAKE	19/09/2017	22/11/2017	64	5.01	Samplers over deployed: subsequently replaced (see below).
SEQ28	LRS-SP017	SEQ-LOGAN RIVER @ KOORALBYN OFFTAKE	22/11/2017	20/12/2017	28	6.35	Replacement Results.
SEQ29*	MAD-SP004	SEQ-MAROON DAM WALL @ OFFTAKE W2 BUOY	27/09/2017	26/10/2017	29	7.05	
SEQ30	LRS-SP013	SEQ-LOGAN RIVER @ HELEN ST	19/09/2017	2/11/2017	44	10.41	1 PFM and EDs lost. Replaced (see below)
SEQ30	LRS-SP013	SEQ-LOGAN RIVER @ HELEN ST	22/11/2017	20/12/2017	28	23.97	Replacement Results.
SEQ31	LRS-SP016	SEQ-RATHDOWNEY WEIR	19/09/2017	17/10/2017	28	2.71	Minimum flow of 3.4 used in water concentration estimates (EDs)
SEQ32	CAC-SP001	SEQ-CANUNGRA CREEK @ OFFTAKE	5/10/2017	2/11/2017	28	3.14	Minimum flow of 3.4 used in water concentration estimates (EDs)
SEQ33	LND-SP014	SEQ-LITTLE NERANG DAM	27/09/2017	25/10/2017	28	3.21	Minimum flow of 3.4 used in water concentration estimates (EDs)

SEQ34*	**HID-SP001**	**SEQ-HINZE DAM UPPER INTAKE**	9/11/2017	7/12/2017	28	3.36	Minimum flow of 3.4 used in water concentration estimates (EDs)
SEQ35	**HID-SP002**	**SEQ-HINZE DAM LOWER INTAKE**	9/11/2017	7/12/2017	28	3.44	
SEQ36*	**MBR-SP013**	**SEQ-DOWNSTREAM FERNVALE STP @ SAVAGES CRC**	6/09/2017	4/10/2017	28	4.12	
SEQ37	**LRS-SP012**	**SEQ-LOGAN RIVER @ CEDAR GROVE**	19/09/2017	17/10/2017	28	2.74	Minimum flow of 3.4 used in water concentration estimates (EDs)

* Indicates replicate sites

** A minimum flow velocity of 3.4 cm s^{-1} is required in order to assess flow velocity using Passive Flow Monitors (PFMs), where flow velocities are lower than this value, this minimum value is applied to flow correction modelling.

2.1 Passive sampler preparation and extraction

Passive flow monitors (PFMs), Empore Disk (ED) passive samplers (for the sampling of polar organic pollutants) and Polydimethylsiloxane (PDMS) passive samplers (for the sampling of more hydrophilic organic pollutants) were all prepared and extracted according to previously published procedures and methods described in Kaserzon et al. 2017)

Figure 1 Preparation of Empore Disk (ED) passive samplers for deployment

2.2 Analytical methods

Chemical analysis was performed at QAEHS using established protocols. EDs were analysed by LC/MS QToF and/or LC/MSMS QQQ for polar herbicides and PPCPs (75 chemicals) with detect/non-detect screening conducted for an additional 45 chemicals. PDMS samplers were analysed for non-polar chemicals comprising of 29 OCPs and 16 PAHs via GC/HRMS (Appendix 1). The analytical methods for herbicides and PPCPs (LC-QQQ MS/MS), OCPs and PAHs (GC-HRMS) and Non-target herbicide and PPCPs (LC-QTOF MS/MS) have all been detailed in previous published reports (Kaserzon et al. 2017)

2.3 Data modelling and reporting of results

Passive sampling enables time integrated estimates of water concentrations (C_w) of a wide range of organic pollutants to be calculated based on the amounts of chemicals accumulated in the sampler within a given exposure period (Vrana *et al.* 2005; Kot *et al.* 2000) The uptake of these chemicals into the sampler is initially linear but eventually reaches steady state whereby equilibrium of the concentration in the sampler and the concentration in

the water is reached. The size and polarity of the contaminant and other environmental factors such as flow, turbulence and temperature can affect the rate of uptake or sampling rate (R_s) which is measured as volume of water sampled per day (L day^{-1}). The duration of the deployment period is another critical factor determining whether time integrated sampling or equilibrium phase sampling is occurring for a given analyte in a sampler.

Equations 1 and 2 describe the estimation of water concentration based on linear or equilibrium phase sampling, respectively.

Equation 1

$$C_W = \frac{C_S \ x \ M_S}{R_S \ x \ t} = \frac{N_S}{R_S x \ t}$$

Equation 2

$$C_W = \frac{C_S}{K_{SW}}$$

Where:

C_W = the concentration of the compound in water (ng L^{-1})
C_S = the concentration of the compound in the sampler (ng g^{-1})
M_S = the mass of the sampler (g)
N_S = the amount of compound accumulated by the sampler (ng)
R_S = the sampling rate (L day^{-1})
t = the time deployed (days)
K_{SW} = the sampler –water partition coefficient (L g^{-1})

Calibration data (typically sampling rates or sampler-water coefficients) obtained in laboratory or field studies were used to derive these concentration estimates. Together with the sampling rates calibration data, deployment specific PFM data are used as a means to assess site-specific effects of water flow on the sampling rates of chemicals and correct for the influence of flow (O'Brien *et al.* 2009). For chemicals detected where no calibration data was available, results were reported as ng $sampler^{-1}$. Methodologies used to calculate site specific sampling rates during the deployment periods are fully described in Kaserzon et al. (2017).

2.4 Quality control and assurance procedures

In order to ensure quality control and to identify any instances of laboratory contamination, blank passive samplers were prepared, extracted and analysed in parallel with exposed samplers for each deployment period (*n* = 3 for each sampler type; ED and PDMS). Laboratory blanks were prepared before each deployment but were not exposed to air or water for the duration of the deployment. These samplers were included in each batch of samples that were extracted and analysed. In cases where chemicals were detected in blanks as well as exposed samples, the concentration in the exposed sample had to exceed three times the concentration in the blank sampler for it to be included in the data. Results were not subtracted for detections in blank samples. Results for all blank samples have been reported in the Appendix 1.

Replicate ED and PDMS passive sampler sites were randomly chosen and deployed in sites 9, 29, 34 and 36. ED replicates only were deployed at site 13 and PDMS replicates only were deployed at sites 1 and 12.

Acceptable replicate values (within < 30 %) were typically observed for passive sampler replicates deployed. Up to 60% was observed in instances were levels were very low (i.e. close to reporting limits). Only values that were significantly above blank background levels (> x3 blk level) are reported.

Recovery of chemicals was verified by spiking blank and exposed samplers with various surrogates prior to extraction, and internal standards prior to analysis. Non-extracted side spikes (solvent blanks spiked with surrogates and recovery standards) were prepared in parallel to spiking and extracting exposed samples. These represent 100% recoveries and are essential in recovery correction calculations.

All QAEHS laboratory procedures are performed by fully trained staff according to established SOPs. QAEHS used the following internal SOPs for the preparation, extraction and analysis of samplers.

NTX-P-004: Preparation of Empore Disks (EDs)
NTX-P-005: Extraction of EDs
NTX-P-008: Pre-cleaning and preparation of PDMS samplers
NTX-P-001: Extraction of PDMS from water
NTX-S-009: Preparation of Flow Monitoring Devices (PFMs) for use with Water Passive Samplers
NTX-A-003: GC/HRMS Method for Pesticide and PAH Analysis
NTX-A-005: LC/MSMS-QQQ method for herbicide and PPCP analysis
NTX-A-004: LC-ESI-QTOF-MS/MS – Target and Non-target polar herbicides and PPCP analysis

3. Results and discussion

3.1 PFM results

Two PFMs were deployed at each sampling site with good agreement observed between duplicate PFMs (> 80%). Average flow velocities estimated from PFMs over the deployment period ranged between 1.02 (Site 7 - YAC SP001 Yabba Creek) – 23.97 cm s^{-1} (Site 30 - LRS SP013 Logan River at Helen St). Low flow which falls below the linearity loss rate range of the PFM (i.e. < 3.4 cm s^{-1}; O'Brien *et al.* 2009) was observed at fourteen sites (Table 2 and Figure).

Under stagnant to very low flow conditions there is little difference in the mass lost from the PFM and therefore the PFM cannot provide an accurate prediction for the effect of flow on R_s (i.e. below a threshold flow of 3.4 cm s^{-1} or PFM loss rate equal to 0.58 g d^{-1}; O'Brien *et al.* 2009; 2011b). When correlating PFM mass loss rate with chemical sampling rates in passive samplers, both the PFM and R_s require minimum flow or turbulence before any effects of flow begin to influence loss rate and chemical accumulation, respectively (i.e. via linear loss rate in PFMs and linear chemical accumulation in passive sampling). This is because the rate of diffusion across the passive sampling membrane under near stagnant conditions is independent from environmental conditions (Kaserzon *et al.* 2014; O'Brien *et al.* 2011b). Therefore, in order to remain within the accurate mathematical modelling range for PFM-based flow velocity prediction, we applied a minimum flow rate of 3.4 cm s^{-1} for the sites showing flow below this threshold and the minimum atrazine equivalence R_s. This may result in a slight over-estimation of R_s and under-estimation of water concentration estimates (C_w), though we do not expect this to be significant.

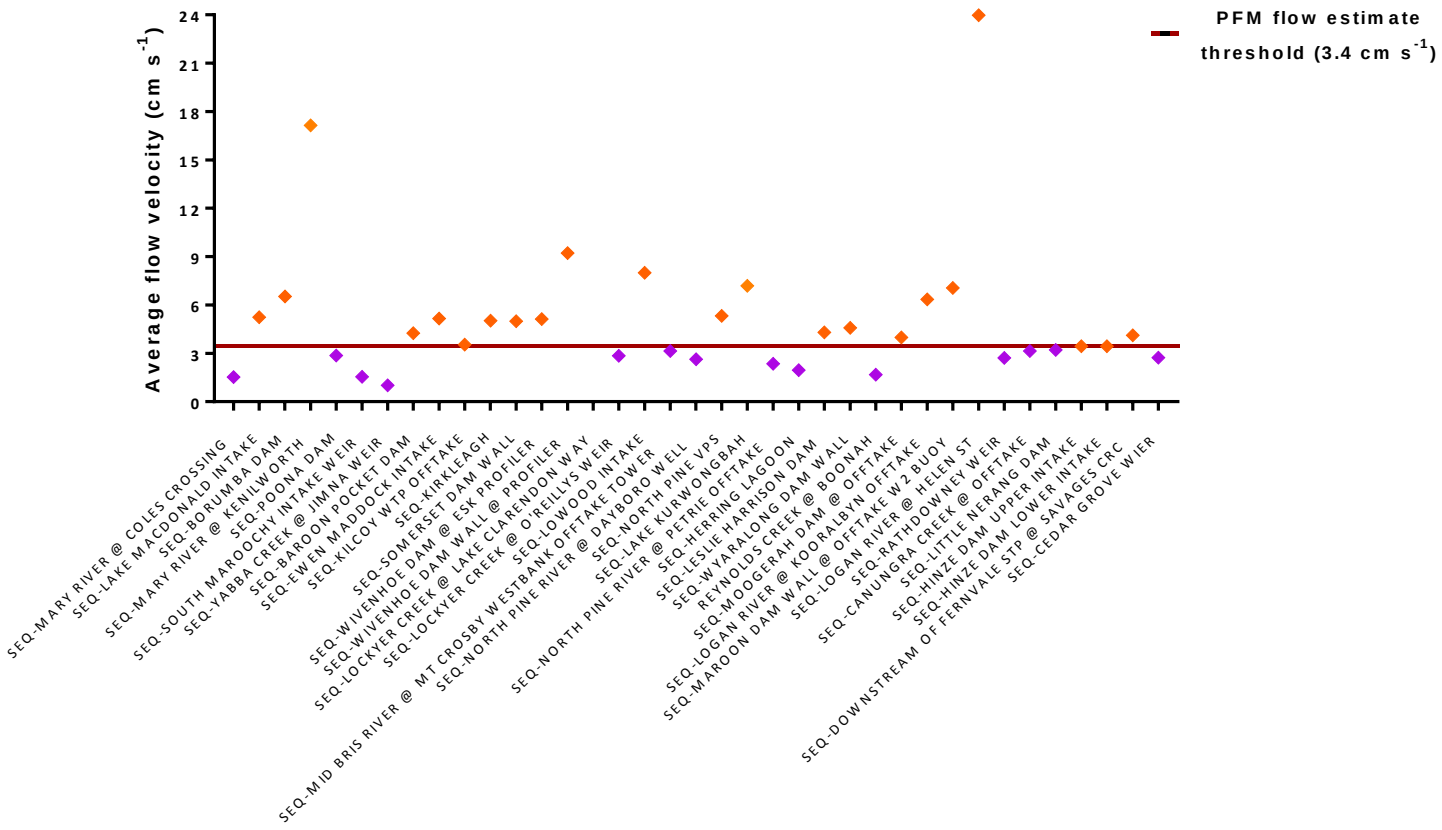

Figure 2 PFM based average flow rate estimations at the deployment sites

3.2 Chemical analysis results

A summary of the number of chemicals detected at the sampling sites, the percent detection of each chemical and mass accumulation range (ng sampler^{-1}) is presented in Table and 3 below. Table summarises the non-polar chemicals detected with PDMS (OCPs, pesticides and PAHs). A total of 22 OCPs and pesticides and 9 PAHs were accumulated in samplers with percent detection at sampling sites ranging from 3% – 97% (for OCPs) and 22% – 56% (for PAHs).

	Number of sites detected (n = 36)	% Detection	Min. Detected (ng PDMS^{-1})	Max. detected (ng PDMS^{-1})
OCPs				
endosulfan sulfate	35	97	0.1	3.1
pp-DDD	35	97	0.03	4.9
pp-DDE	31	86	0.12	10
heptachlor epoxide B	29	81	0.12	7
dacthal	26	72	1.3	660
a-HCH	25	69	0.03	0.3
op-DDD	18	50	0.1	3.4
dieldrin	17	47	3.8	33
op-DDT	15	42	0.01	0.14
pp-DDT	15	42	0.04	1.6
endrin	13	36	0.06	0.14
chlorpyrifos	10	28	22	380
op-DDE	9	25	0.015	0.2
cis-chlordane (a)	6	17	0.037	1.6
PeCB	5	14	0.9	1.4
trans-chlordane (r)	5	14	1	6.4
aldrin	4	11	0.56	1.1
endrin ketone	2	6	0.56	1.7
a-endosulfan	1	3	1.1	
b-HCH	1	3	0.11	
heptachlor	1	3	2.1	
heptachlor epoxide A	1	3	0.1	
PAHs				
Chrysene	20	56	3	14
Fluoranthene	17	47	12	180
Benzo (bjk) fluoranthene	16	44	0.6	3.6
Indeno (1,2,3-cd) pyrene	15	42	0.12	1
Benzo (a) anthrancene	11	31	0.76	10
Benzo (e) pyrene	11	31	1.1	4.3
Pyrene	9	25	23	170
Benzo (a) pyrene	8	22	0.51	1.4
Benzo (g,h,i) perylene	8	22	0.37	2.7

Table summarises the polar chemicals detected with EDs (herbicides, insecticides and PPCPs). A total of 30 herbicides and 15 PPCPs were accumulated in samplers with percent detection at sampling sites ranging from 3%-97% (for herbicides and insecticides) and 3% - 83% (for PPCPs). The full data reporting sheet listing individual masses and estimated water concentrations of all analytes for each site are provided in Appendix 1.

Table 2 Summary of the number of chemicals accumulated in PDMS, percent of detection (%) at the sites and the range of mass accumulated over 28-29 days (ng PDMS^{-1})

	Number of sites detected (n = 36)	% Detection	Min. Detected (ng PDMS^{-1})	Max. detected (ng PDMS^{-1})
OCPs				
endosulfan sulfate	35	97	0.1	3.1
pp-DDD	35	97	0.03	4.9
pp-DDE	31	86	0.12	10
heptachlor epoxide B	29	81	0.12	7
dacthal	26	72	1.3	660
a-HCH	25	69	0.03	0.3
op-DDD	18	50	0.1	3.4
dieldrin	17	47	3.8	33
op-DDT	15	42	0.01	0.14
pp-DDT	15	42	0.04	1.6
endrin	13	36	0.06	0.14
chlorpyrifos	10	28	22	380
op-DDE	9	25	0.015	0.2
cis-chlordane (a)	6	17	0.037	1.6
PeCB	5	14	0.9	1.4
trans-chlordane (r)	5	14	1	6.4
aldrin	4	11	0.56	1.1
endrin ketone	2	6	0.56	1.7
a-endosulfan	1	3	1.1	
b-HCH	1	3	0.11	
heptachlor	1	3	2.1	
heptachlor epoxide A	1	3	0.1	
PAHs				
Chrysene	20	56	3	14
Fluoranthene	17	47	12	180
Benzo (bjk) fluoranthene	16	44	0.6	3.6
Indeno (1,2,3-cd) pyrene	15	42	0.12	1
Benzo (a) anthrancene	11	31	0.76	10
Benzo (e) pyrene	11	31	1.1	4.3
Pyrene	9	25	23	170
Benzo (a) pyrene	8	22	0.51	1.4
Benzo (g,h,i) perylene	8	22	0.37	2.7

Table 3 Summary of the number of chemicals accumulated in EDs, percent of detection (%) at the sites and the range of mass accumulated over 28-29 days (ng ED^{-1})

	Number of sites detected (n = 36)	% Detection	Min. Detected (ng ED^{-1})	Max. detected (ng ED^{-1})
Herbicides and Insecticides				
Desisopropyl Atrazine	35	97	0.08	4.3
Atrazine	34	94	0.06	44.4
Diuron	34	94	0.20	7.5
Metolachlor	32	89	0.10	69.7
Simazine	32	89	0.10	10.2
Desethyl Atrazine	30	83	0.14	7.0
Metsulfuron-Methyl	29	81	0.32	7.4
Tebuthiuron	29	81	0.10	15.0
Hexazinone	24	67	0.21	30.6
2,4-D	23	64	0.36	15.2

Tebuconazole	23	64	0.05	1.1
Imidacloprid	22	61	0.16	5.7
Terbuthylazine des ethyl	21	58	0.06	0.58
MCPA	19	53	0.22	11.0
Terbuthylazine	17	47	0.06	0.3
Metalaxyl	16	44	0.10	3.1
3,4 Dichloro Aniline	12	33	0.06	0.14
Isoxaflutole	10	28	0.05	0.10
Triclopyr	10	28	0.10	1.3
Propazine	9	25	0.10	0.34
Propiconazole	6	17	0.05	0.20
Haloxyfop	5	14	0.24	1.2
Propoxur	3	8	0.20	0.45
Prometryn	2	6	0.15	1.5
Ametryn	1	3	0.06	
Bromacil	1	3	0.38	
bromoxynil	1	3	0.09	
Fluazifop	1	3	0.06	
Methomyl	1	3	0.14	
Pendimethalin	1	3	0.07	
PPCPs				
DEET	30	83	3.20	102
Salicylic acid	30	83	0.60	2.8
Hydrochlorothiazide	14	39	0.05	0.39
Carbamazepine	10	28	0.32	6.3
Iopromide	9	25	0.10	10.3
Acesulfame	9	25	0.10	0.9
Paracetamol	6	17	0.20	0.40
Caffeine	5	14	19.00	29.5
Gabapentin	5	14	0.21	2.6
Temazepam	3	8	0.90	1.2
Atenolol	1	3	1.10	
Codeine	1	3	2.80	
Ibuprofen	1	3	8.00	
Tramadol	1	3	0.17	
Triclosan	1	3	0.08	

3.3 OCPs

In total, twenty two OCPs and pesticides were accumulated in PDMS samplers over the 28 – 29 day deployment period (Table , Figure , Appendix 1), with the amount of ∑OCPs accumulated ranging between 0.25 – 662 ng $PDMS^{-1}$ for sites 19 (North Pine River @ Dayboro well) and 11 (Kirkleagh), respectively.

The highest frequency of detection was observed for endosulfan sulfate and pp-DDD with 97% detection for each, followed by pp-DDE with 86%, heptachlor epoxide B with 81% and dachthal with 72% detection. Highest accumulation was observed for dacthal at 660 ng $PDMS^{-1}$ (at site 11, Kirkleagh) followed by chlorpyrifos at 380 ng $PDMS^{-1}$ (at site 30, Logan river @ Helen St).

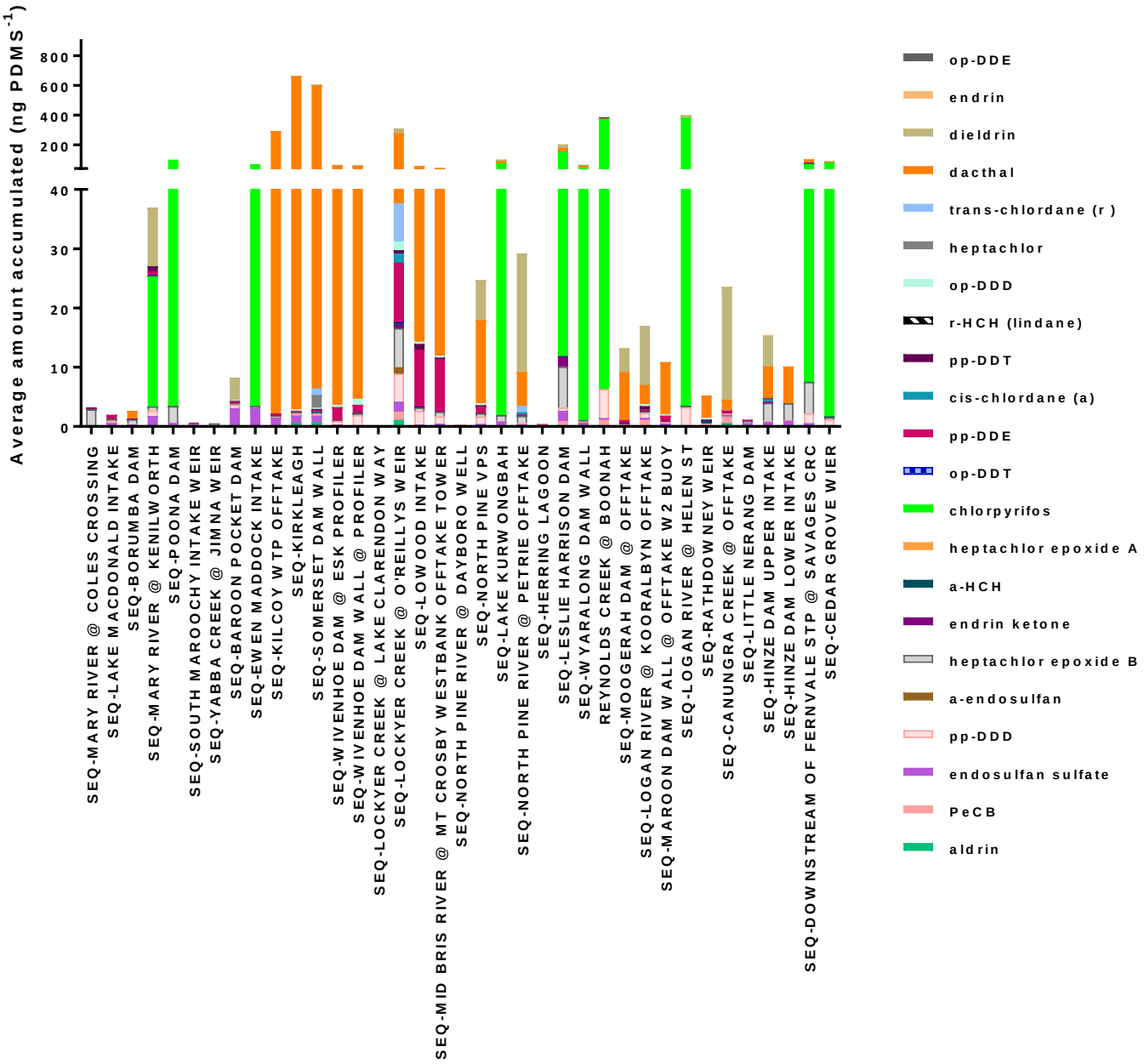

Figure 3 Total amounts of 22 ΣOCPs accumulated in PDMS passive samplers

The conversion of OCP masses accumulated in passive samplers to average water concentrations over the deployment period revealed an estimated water concentration range of ∑OCPs between 0.01 – 15 ng L^{-1} for sites 19 (North Pine River @ Dayboro well) and 11 (Kirkleagh), respectively (Figure 44). Followed by site 12 (Somerset Dam Wall) with ∑OCPs of 14 ng L^{-1}.

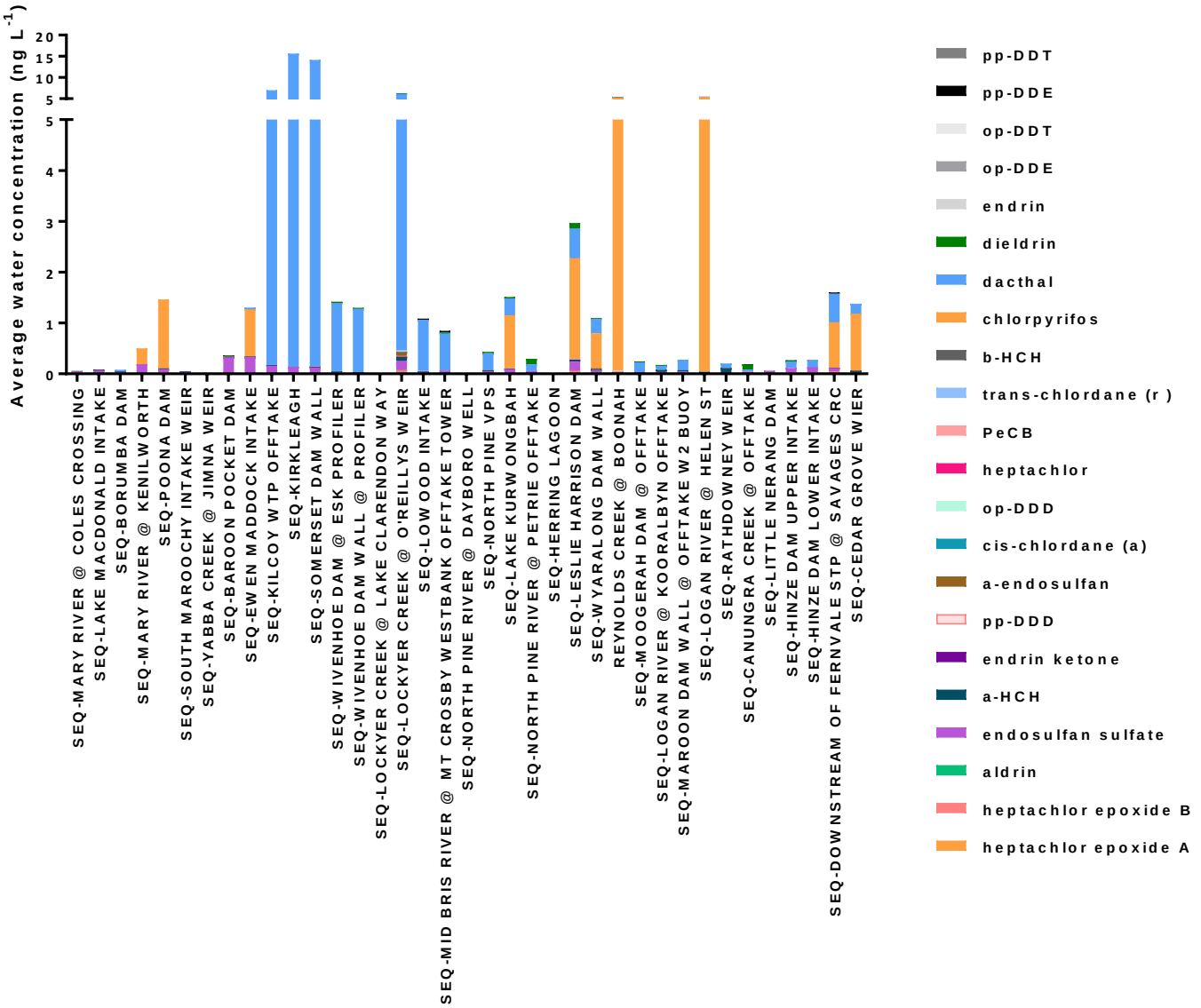

Figure 4 Total estimated water concentrations of 22 ΣOCPs derived from accumulation in PDMS

3.4 PAHs

In total, nine different PAHs were accumulated in PDMS samplers with an average amount of ∑PAHs accumulated ranging between 0.2 – 347 ng $PDMS^{-1}$ for sites 18 (Mid Brisbane River @ Mt Crosby westbank offtake tower) and 29 (Maroon Dam wall @ offtake W2 bouy), respectively (Table 2, Figure , Appendix 1). The highest frequency of detection was observed for chrysene with 56% detection, followed by fluoranthene with 47% and Bezo (bjk) fluoranthene with 44% detection frequency. The PAH accumulated in the greatest abundance between sites was fluoranthene > pyrene > Chrysene.

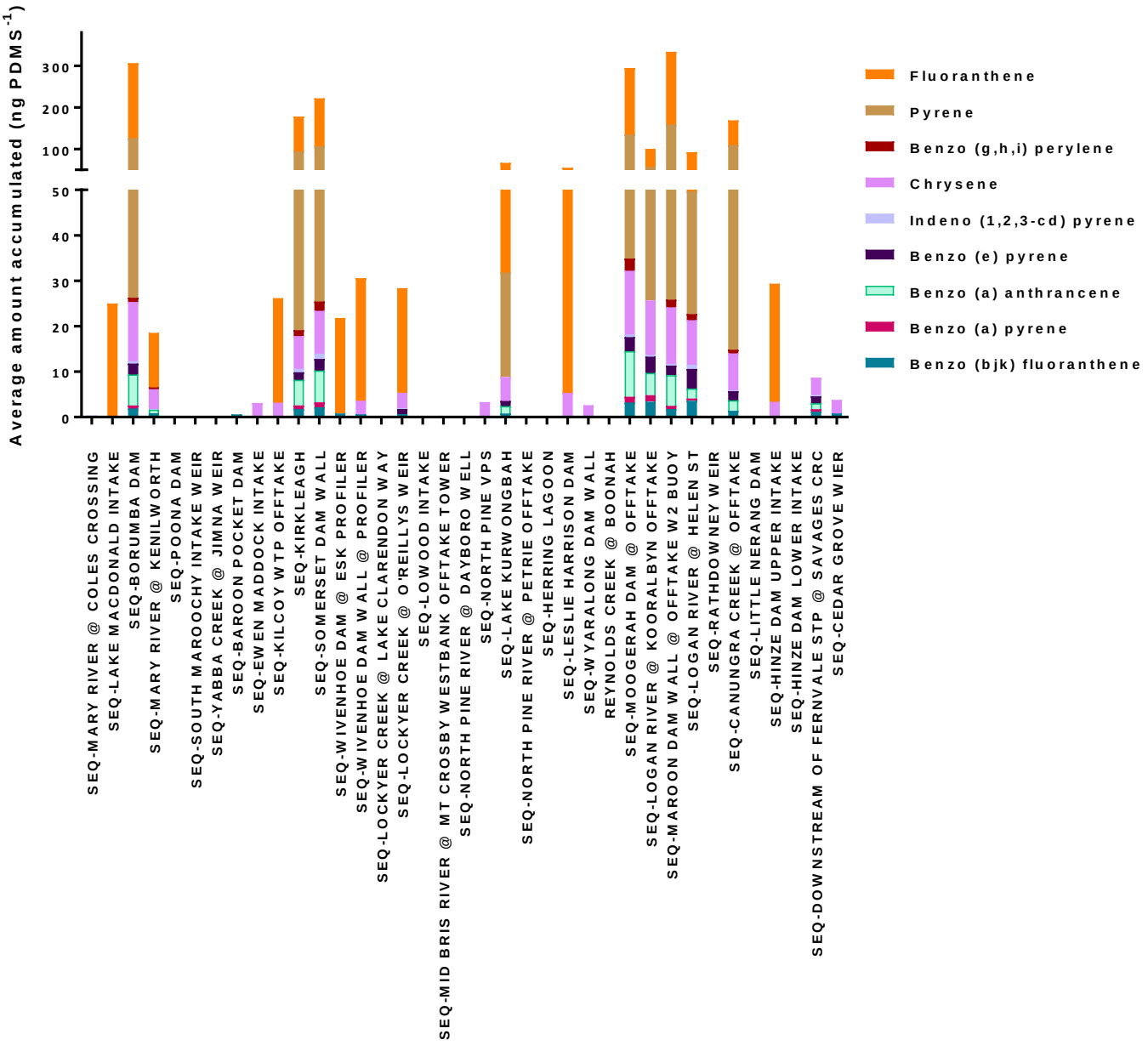

Figure 5 Total amounts of 15 ΣPAHs accumulated in PDMS passive samplers

When converting the masses of accumulated PAHs in passive samplers to average water concentrations over the deployment period, concentrations of ∑PAHs ranged between 0.001 – 3.4 ng L^{-1} (Figure 6)

for sites 18 (Mid Brisbane River @ Mt Crosby westbank offtake tower) and 27 (Moogerah Dam @ offtake), respectively. Thirty sites had reportable water concentrations of PAHs. Highest ∑PAH concentrations were observed at sites 27 (Moogerah Dam @ offtake) followed by site 32 (Canungra creek @ offtake) site 3 (Borumba Dam) and site 29 (Maroon Dam wall @ offtake W2 bouy) with concentrations of 3.4, 2.5, 2.3 and 2.3 ng L^{-1}, respectively.

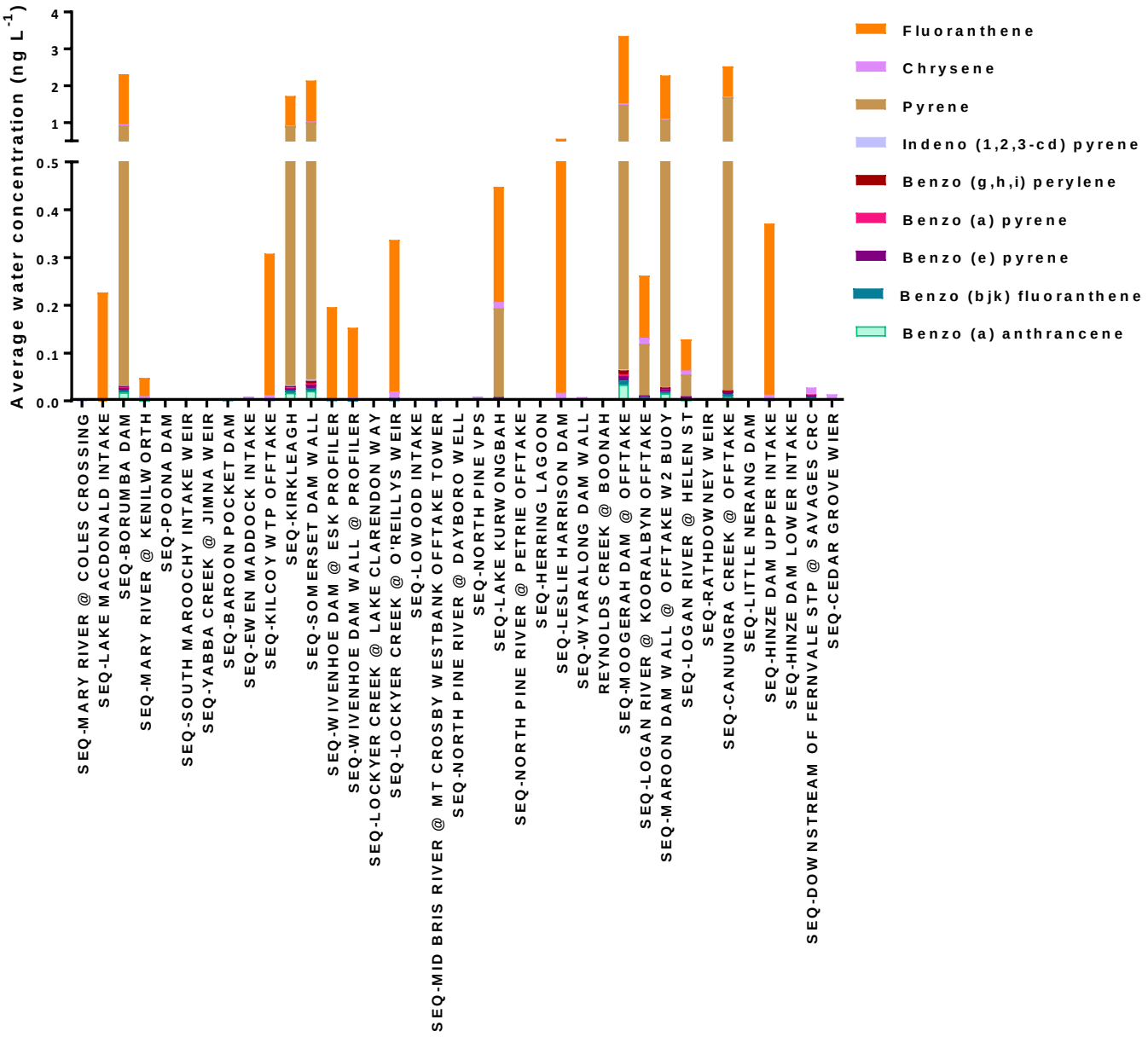

Figure 6 Total estimated water concentrations of 9 ΣPAHs

3.5 Herbicides and insecticides

Over the 28-29 day deployment period, 30 herbicides and insecticides accumulated in ED passive samplers (

	Number of sites detected (n = 36)	% Detection	Min. Detected (ng PDMS^{-1})	Max. detected (ng PDMS^{-1})
OCPs				
endosulfan sulfate	35	97	0.1	3.1
pp-DDD	35	97	0.03	4.9
pp-DDE	31	86	0.12	10
heptachlor epoxide B	29	81	0.12	7
dacthal	26	72	1.3	660
a-HCH	25	69	0.03	0.3
op-DDD	18	50	0.1	3.4
dieldrin	17	47	3.8	33
op-DDT	15	42	0.01	0.14
pp-DDT	15	42	0.04	1.6
endrin	13	36	0.06	0.14
chlorpyrifos	10	28	22	380
op-DDE	9	25	0.015	0.2
cis-chlordane (a)	6	17	0.037	1.6
PeCB	5	14	0.9	1.4
trans-chlordane (r)	5	14	1	6.4
aldrin	4	11	0.56	1.1
endrin ketone	2	6	0.56	1.7
a-endosulfan	1	3	1.1	
b-HCH	1	3	0.11	
heptachlor	1	3	2.1	
heptachlor epoxide A	1	3	0.1	
PAHs				
Chrysene	20	56	3	14
Fluoranthene	17	47	12	180
Benzo (bjk) fluoranthene	16	44	0.6	3.6
Indeno (1,2,3-cd) pyrene	15	42	0.12	1
Benzo (a) anthrancene	11	31	0.76	10
Benzo (e) pyrene	11	31	1.1	4.3
Pyrene	9	25	23	170
Benzo (a) pyrene	8	22	0.51	1.4
Benzo (g,h,i) perylene	8	22	0.37	2.7

Table , 7, Appendix 1). The average amount of ∑herbicides and insecticides accumulated ranged between 0.1 - 145 ng ED^{-1} for sites 23 (Herring Lagoon) and 16 (Lockyer Creek @ O'reillys weir), respectively. Out of the 28 priority herbicides and pesticides, 14 were found among sites. The most frequently detected herbicide were Desisopropyl Atrazine and atrazine (97% and 89%, respectively) followed by diuron (94%), metolachlor (89%) and simazine (89%). All sites had positive detects with site 16 (Lockyer Creek @ O'reillys weir) expressing the highest accumulated amount (145 ng ED^{-1}) followed by new site 37 (Cedar Grove weir) introduced this season (107 ng ED^{-1}), with both sites showing the profile of high accumulated levels of metolachlor.

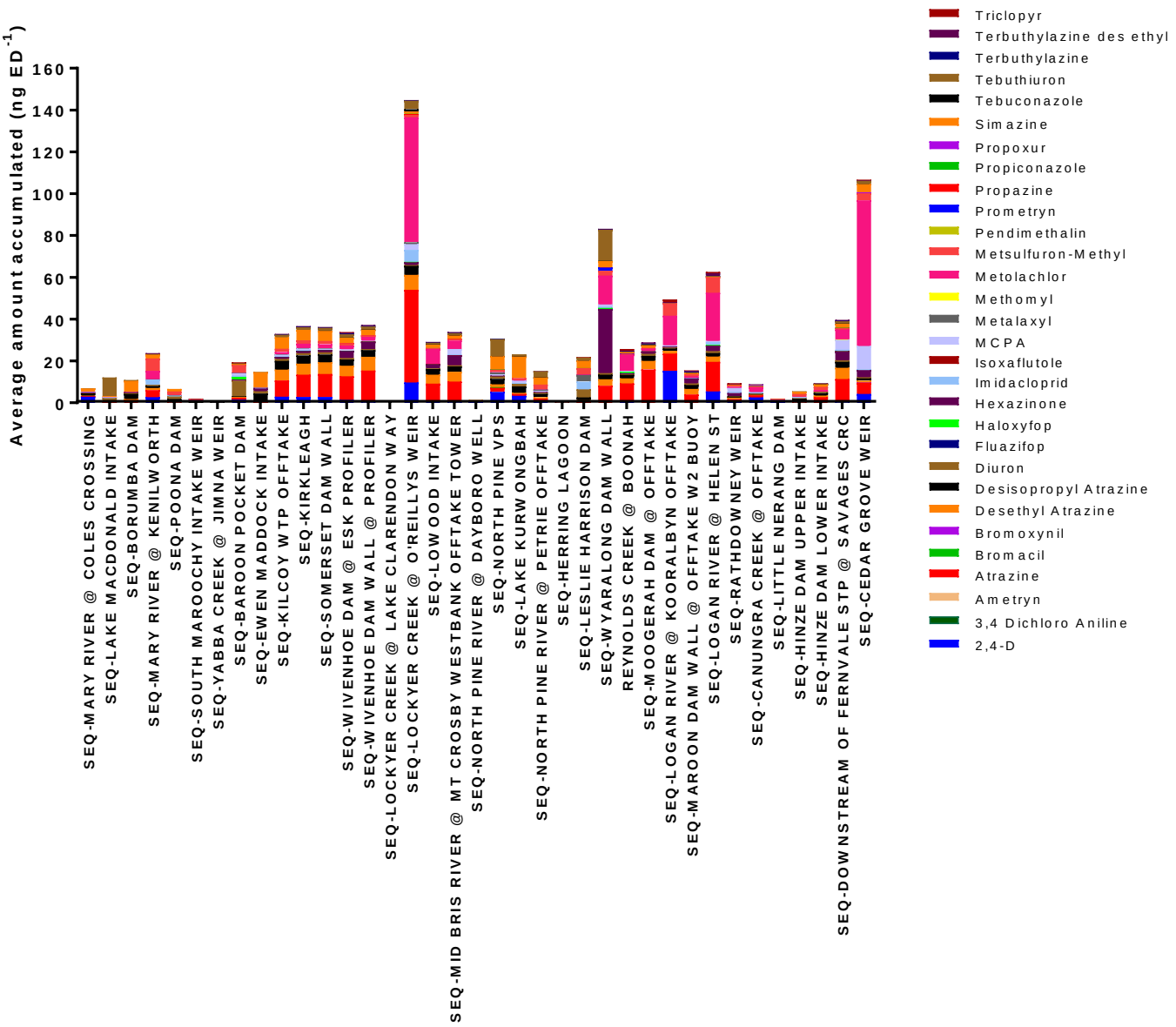

Figure 7 Total amounts of 30 Σherbicides and insecticides accumulated in ED passive samplers

Water concentrations were estimated for fifteen herbicides and insecticides with average total ∑concentrations ranging between 0.07 - 104 ng L^{-1} for sites 23 (Herring Lagoon) and 16 (Lockyer Creek @ O'reillys weir), respectively (Figure). The highest total ∑concentration across all sites was for atrazine (170 ng L^{-1}) followed by metolachlor (123 ng L^{-1}).

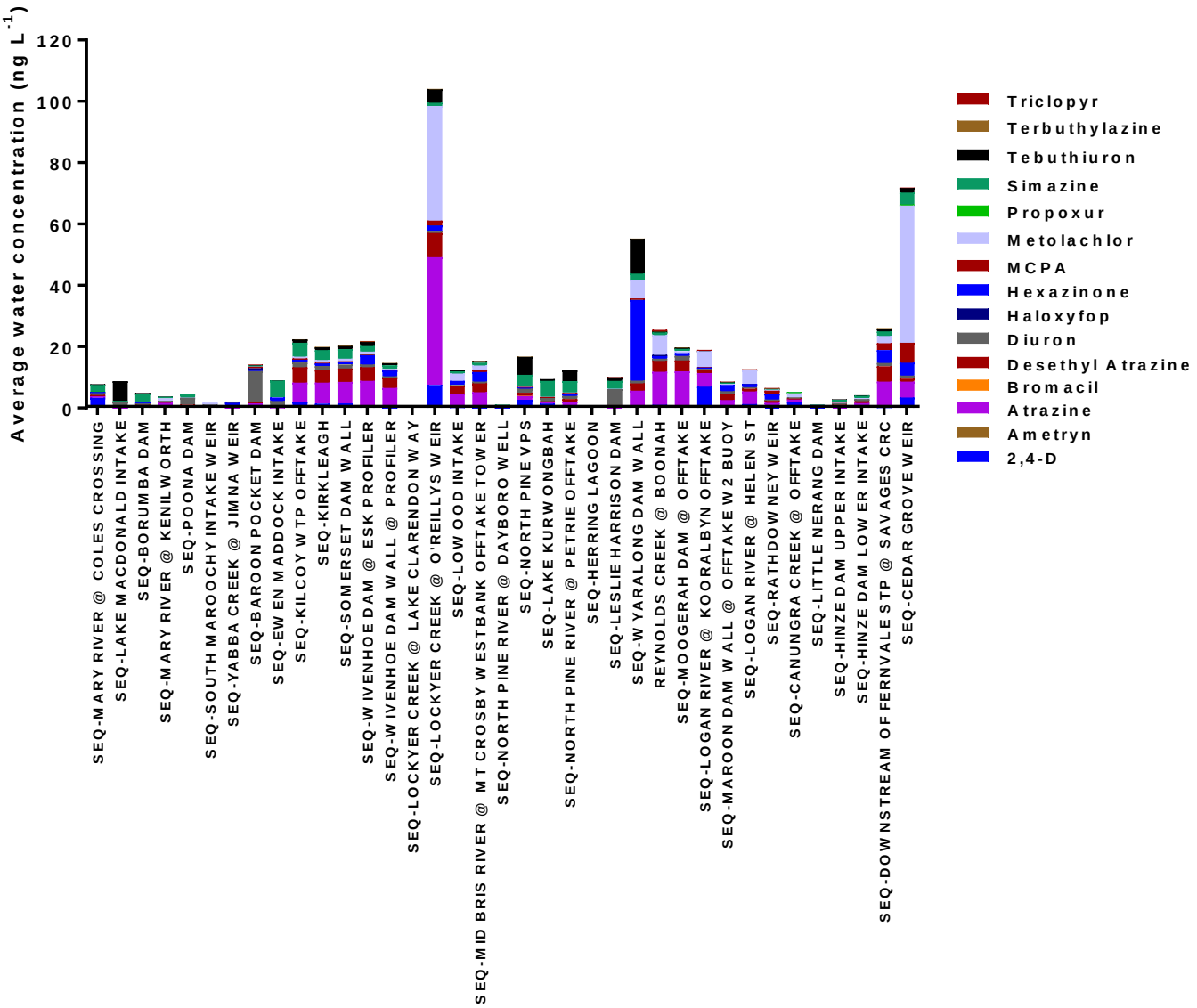

Figure 8 Total estimated water concentrations of 15 Σherbicides and insecticides

3.6 PPCPs

Fifteen PPCPs were detected with the average amount of ΣPPCPs accumulated ranging between 0.12 – 70 ng ED^{-1} at sites 35 (Hinze Dam lower intake) and 36 (Downstream of Fernvale STP @ savages CRC), respectively (

	Number of sites detected (n = 36)	% Detection	Min. Detected (ng $PDMS^{-1}$)	Max. detected (ng $PDMS^{-1}$)
OCPs				
endosulfan sulfate	35	97	0.1	3.1
pp-DDD	35	97	0.03	4.9
pp-DDE	31	86	0.12	10
heptachlor epoxide B	29	81	0.12	7
dacthal	26	72	1.3	660
a-HCH	25	69	0.03	0.3
op-DDD	18	50	0.1	3.4
dieldrin	17	47	3.8	33
op-DDT	15	42	0.01	0.14
pp-DDT	15	42	0.04	1.6
endrin	13	36	0.06	0.14
chlorpyrifos	10	28	22	380
op-DDE	9	25	0.015	0.2
cis-chlordane (a)	6	17	0.037	1.6
PeCB	5	14	0.9	1.4
trans-chlordane (r)	5	14	1	6.4
aldrin	4	11	0.56	1.1
endrin ketone	2	6	0.56	1.7
a-endosulfan	1	3	1.1	
b-HCH	1	3	0.11	
heptachlor	1	3	2.1	
heptachlor epoxide A	1	3	0.1	
PAHs				
Chrysene	20	56	3	14
Fluoranthene	17	47	12	180
Benzo (bjk) fluoranthene	16	44	0.6	3.6
Indeno (1,2,3-cd) pyrene	15	42	0.12	1
Benzo (a) anthrancene	11	31	0.76	10
Benzo (e) pyrene	11	31	1.1	4.3
Pyrene	9	25	23	170
Benzo (a) pyrene	8	22	0.51	1.4
Benzo (g,h,i) perylene	8	22	0.37	2.7

Table , Figure , Appendix 1). This is after the exclusion of site 22 (North Pine River @ Petrie Offtake) that had a total ΣPPCPs of 104 ng ED^{-1}. The elevated figure for this site is from unusually high levels of DEET detected (102.7 ng ED^{-1}), likely as a result of contamination from field. Most frequently detected were the insecticide DEET and salysilic acid with a detection frequency of 83% for both, followed by hydrochlorothiazide at 39% and carbamazepine detected at 28% of sites. Few PPCPs were detected at most sites with the exception of sites 36 (Fernvale STP @ Savages Crossing), 37 (Cedar Grove Weir) and 18 (Mid Brisbane River @ Mt Crosby) showing detects for 10, 8 and 7 PPCPs, respectively.

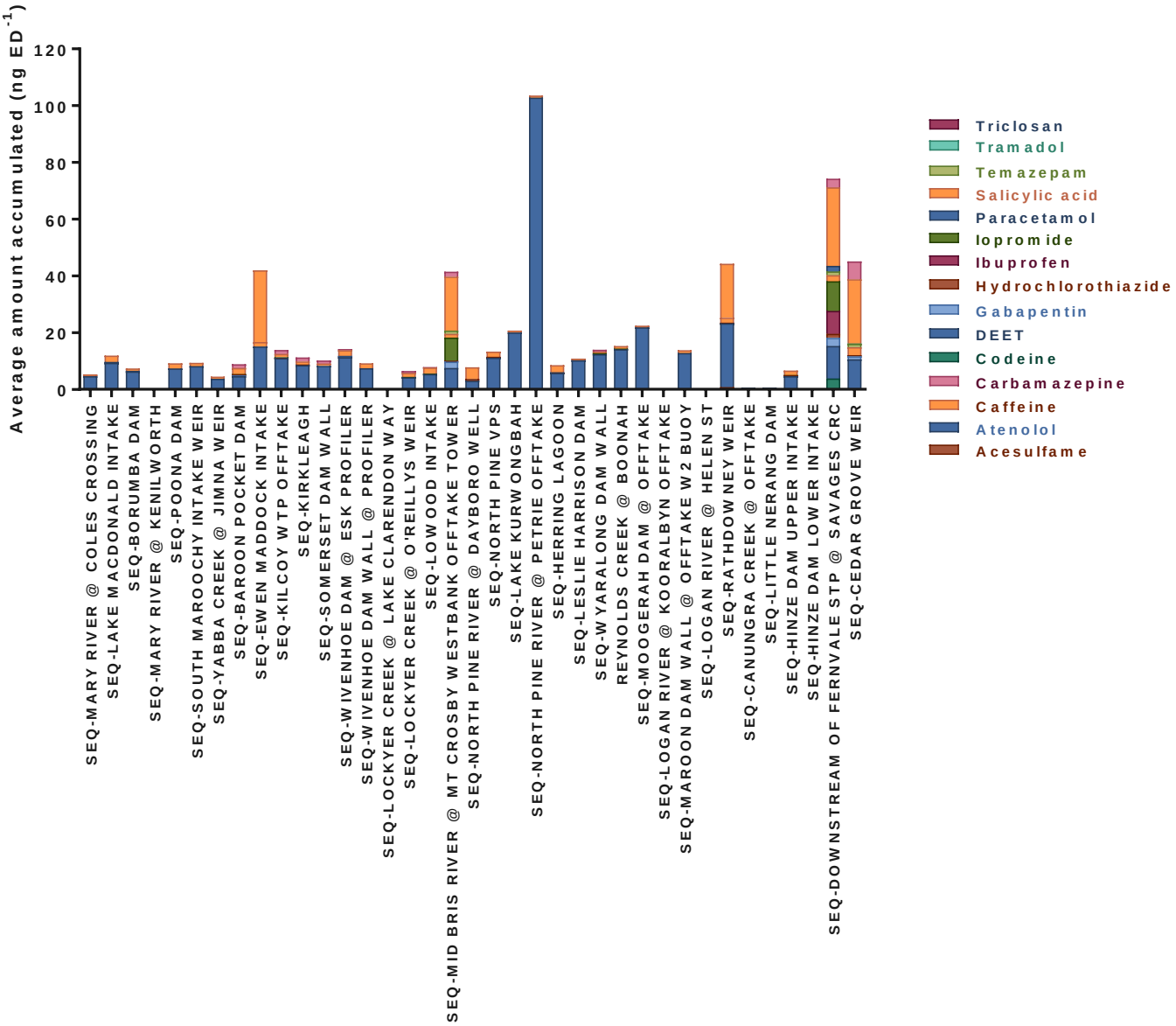

Figure 9 Average amounts of 16 PPCPs accumulated in ED passive samplers

When converting the masses of accumulated PPCPs in EDs to average water concentrations over the deployment period only caffeine, carbamazepine, codeine, DEET and hydrochlorothiazide could be quantified. For these PPCPs, average total ∑PPCP water concentrations ranged between 0.06 – 25.6 ng L^{-1} for site 4 (Mary River @ Kenilworth) and 36 (Downstream of Fernvale STP @ Savages CRC), respectively (Figure 2). DEET makes up the entire profile at 14 sites and was the most frequently detected PPCP.

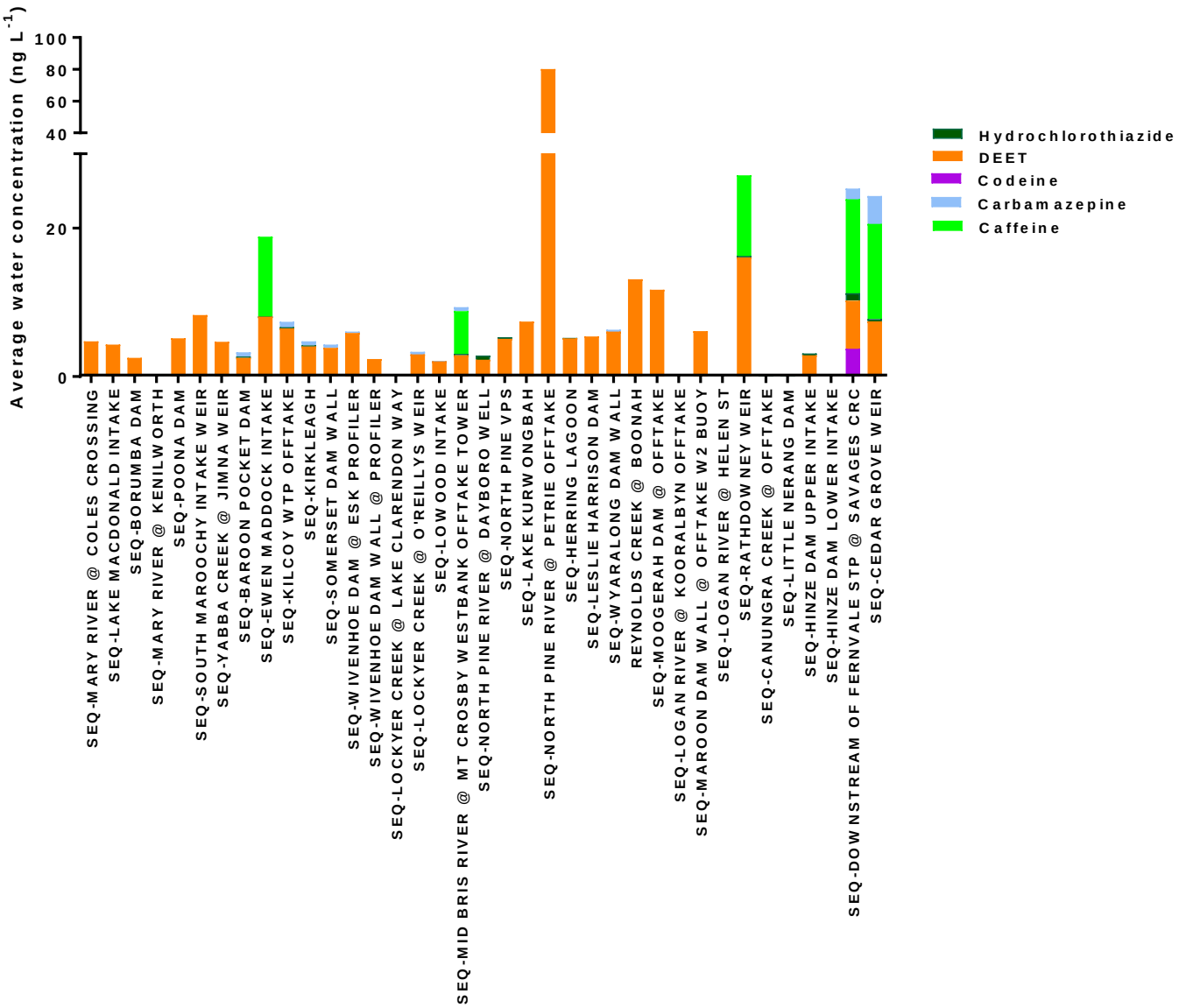

Figure 2 Average estimated water concentrations of 4 PPCPs

3.7 Analysis of non-target polar chemicals

Along with the target list of 75 polar chemicals identified for investigation, a screening for an additional 45 herbicides and PPCP chemicals that have the potential of transporting to waterways has been performed to investigate their presence in the water systems. During this sampling season four non-target chemicals were detected form this library: bendiocarb, carbaryl, carbendazim and sulphamethoxazole (Table 4). In addition to the suspect library search a broader scale non target search was performed on all ED sample extracts from this season (although this investigation does not form part of the deliverables for this project). The suspect search revealed an additional 7 compounds not previously targeted. These comprise mainly of insecticides, 2 fungicides and an antibiotic (Table 4). Any new chemicals tentatively identified here will be added to the non-target library list for investigation in future sampling campaigns. Performing full non-target suspect screening on all samples is an extremely time-consuming process and will only be conducted if/when time permits. It is possible that further investigations will be carried out on specific sites / samples of concern if/when time permits.

Table 4 List of tentatively identified non-target chemicals in EDs, and the sites in which they were detected. Chemicals were tentatively identified using suspect screening and library matching. ***Note: All chemicals listed here are only tentatively identified until full confirmation with relevant standards can be performed***

Chemicals name	Description	Sites with tentative detects
Bendiocarb	carbamate insecticide	35:HINZE DAM LOWER INTAKE, 30:LOGAN RIVER @ HELEN ST, 28:LOGAN RIVER @ KOORALBYN OFFTAKE, 29:MAROON DAM WALL @ OFFTAKE W2 BUOY, 18:BRIS RIVER @ MT CROSBY WESTBANK OFFTAKE TOWER, 36:DOWNSTREAM OF FERNVALE STP @ SAVAGES CRC
Carbaryl	Insecticide	21:LAKE KURWONGBAH, 16:LOCKYER CREEK @ O'REILLYS WEIR, 36:DOWNSTREAM OF FERNVALE STP @ SAVAGES CRC, 5:POONA DAM, 6:MAROOCHY INTAKE WEIR
Carbendazim	broad-spectrum benzimidazole fungicide	3:SEQ-BORUMBA DAM, 9:EWEN MADDOCK INTAKE, 37:CEDAR GROVE WIER, 28:LOGAN RIVER @ KOORALBYN OFFTAKE, 18:MID BRIS RIVER @ MT CROSBY WESTBANK OFFTAKE TOWER, 17:LOWOOD INTAKE.
Esfenvalerate	pyrethroid insecticide	17:LOWOOD INTAKE, 25:WYARALONG DAM WALL
Hexythiazox	acaricide pesticide	27:MOOGERAH DAM @ OFFTAKE
Omethoate	organophosphorous insecticide	29:MAROON DAM WALL @ OFFTAKE W2 BUOY
Parathion ethyl	organophosphate insecticide	24:LESLIE HARRISON DAM, 28:LOGAN RIVER @ KOORALBYN OFFTAKE
Phorate	organophosphate insecticide	1:RIVER @ COLES CROSSING, 2:-LAKE MACDONALD INTAKE, 3:BORUMBA DAM, 5:POONA DAM, 9:EWEN MADDOCK INTAKE, 10:KILCOY WTP OFFTAKE, 12:SOMERSET DAM WALL, 16:LOCKYER CREEK @ O'REILLYS WEIR, 17:LOWOOD INTAKE, 19:NORTH PINE RIVER @ DAYBORO WELL, 21:LAKE KURWONGBAH, 24:LESLIE HARRISON DAM, 27:MOOGERAH DAM @ OFFTAKE, 28:LOGAN RIVER @ KOORALBYN OFFTAKE, 29:MAROON DAM WALL @ OFFTAKE W2 BUOY, 30:LOGAN RIVER @ HELEN ST, 33:LITTLE NERANG DAM, 34:HINZE DAM UPPER INTAKE, 35:HINZE DAM LOWER INTAKE, 36:DOWNSTREAM OF FERNVALE STP @ SAVAGES CRC
Spirotetramat	insecticide	16:LOCKYER CREEK @ O'REILLYS WEIR,
Sulphamethoxazole	antibiotic	37:CEDAR GROVE WIER, 28:LOGAN RIVER @ KOORALBYN OFFTAKE, 36:DOWNSTREAM OF FERNVALE STP @ SAVAGES CRC
Trifloxystrobin	agricultural fungicide	33:LITTLE NERANG DAM

4. Summary

A wide range of organic micro-pollutants were detected at all thirty six sampling locations during the winter 2017 deployment period. 22 OCPs were detected in total with detects at all sites. Although a number of OCPs were detected at almost all monitoring sites, the majority of chemicals were present at very low levels (< 15 ng L^{-1} ΣOCPs) which may indicate residue background levels as a result of years of persistent use and subsequent deregulation. Most site profiles are dominated by dacthal, chlorpyrifos and endosulfan sulfate. Australia has set chlorpyrifos water guideline values of 0.04 and 10 ng L-1 for 99% and 95% species protection, respectively. Levels found at sampling sites have consistently been above 99% guideline value but below the 99% species protection limit.

PAHs were detected at 25 sites with a profiles dominated by fluoranthene, pyrene and chrysene. Nine PAHs were detected across sites, though overall maximum ΣPAHs were below 4 ng L^{-1}, indicating low background levels. PAHs are ubiquitous in the environment and are introduced via anthropogenic sources primarily as a result of incomplete combustion as well as via natural sources (i.e. forest fires and the transformation of biogenic precursors) (Nguyen *et al.* 2014). The hydrophobic nature of PAHs typically results in low concentrations in water as they generally associate with particles and sediment (Nguyen *et al.* 2014).

Herbicides and insecticides were detected at all sites with 14 out of 28 priority herbicides detected, with the highest total Σherbicides and insecticides detected < 104 ng L^{-1}. The triazine class herbicides (atrazine and its degradation products and simazine) were the most commonly detected with frequencies of detection of > 89%, followed by metolachlor with a frequently of detection at 89% of sites. Triazine herbicides can remain in soils for several months and can migrate from soil to groundwater or transport to waterways via runoff and flooding events. Atrazine and simazine have been widely used in Australia and are registered for 1600 uses including weed control in orchards and various crops (APVMA 2011a; ANZECC & ARMCANZ 2000).

PPCPs were found at all sites except site 28 (Logan River @ Kooralbyn offtake) with total concentrations ranging from 0.06 – 25.6 ng L^{-1}. The predominant PPCP was the insect repellent DEET, which was expected due to its widespread use. DEET was detected at 83% of sites with a maximum concentration of 80 ng L^{-1}. Hydrochlorothiazide and carbamazepine were detected at 39% and 28% of sites (at maximum concentrations of 0.39 and 6.3 ng L^{-1}, respectively). The contribution of pharmaceuticals and personal care products would generally be an indicator of systems which are used for human recreational activities or which receive some degree of treated effluent, however a number of PPCPs may be ubiquitous in many environments. Examples include DEET, caffeine and salysilic acid. Sites with a larger variety of PPCPs such as sites 36 (Fernvale STP @ Savages Crossing) and new site 37 (Cedar Grove weir) indicate higher anthropogenic waste input, likely due to their vicinity to STPs.

Pharmaceuticals and personal care products have emerged as a major group of environmental contaminants over the past decade. Some chemicals persist through wastewater treatment processes resulting in their continuous release into the aquatic environment (Kaserzon *et al.* 2014). While these chemicals are generally present at trace levels and present little risk of acute toxicity, some compounds can show chronic effects at these levels and the effects of mixture toxicities are unknown (Hughes *et al.* 2013).

4.1 Comparison to water quality guidelines values

A comparison with a selection of available water guideline values and species protection values are provided in Table
.

Table 5 Guidelines for Australian Drinking Water and Freshwater Aquatic Ecosystems

	ANZECC & ANCANZ (2000) Trigger values for freshwater		
	Australian Drinking Water Guidelines 6 (2011) (ng.L^{-1})	**99% species protection (ng.L^{-1})**	**95% species protection (ng.L^{-1})**
Herbicides & Insecticides			
Atrazine	20000	700	13000
Bromacil	400000	N/A	N/A
Diazinon	4000	0.03	10
Diuron	20000	N/A	N/A
Haloxyfop	1000	N/A	N/A
Hexazinone	400000	N/A	N/A
Metolachlor	300000	N/A	N/A
Metsulfuron methyl	40000	N/A	N/A
Simazine	20000	200	3200
Tebuthiuron	N/A	20	2200
Triclopyr	20000	N/A	N/A
2,4-D	30000	140000	280000
OCPs			
Chlordane	2000	30	800
Chlorpyrifos	10000	0.04	10
DDT	9000	6	10
Dieldrin and Aldrin	300		
Endosulfan	20000	30	200
Endrin	N/A	10	20
Heptachlor	300	10	90
r-HCH (lindane)	10000	70	200

No herbicides/insecticides or OCPs with an available ADWG value were detected at concentrations that exceeded their drinking water guideline value or the 99% freshwater species protection guideline. Chlorpyrifos exceeded the 99% species protection value at each of the sites it was detected at, although did not exceed the 95% species protection guideline. The highest estimated level for chlorpyrifos was 5.4 ng L^{-1} at site 30 (Logan river @ Helen St).

4.2 Future recommendations

Several recommendations for future work are suggested to build upon the preliminary findings in the current report.

- Continued temporal and seasonal comparisons will be further assessed as data from additional sampling campaigns is provided to assess if any trends emerge between sites / seasons.
- Sampling devices should be placed strategically at high rainfall sites to better measure and account for any higher water flow velocities and increased runoff activity.

- The screening for non-target chemicals will continue over the next sampling campaign, followed by a re-assessment of the need to continue with non-target screenings. This perhaps could be done at a reduced capacity for a handful of sites that have been identified to contain increased inputs of micro-pollutants.

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6. Appendix 1 –

See enclosed excel file 'SEQW results_Winter2017.xls'

Reporting sheet listing all micro-pollutants investigated, levels accumulated in PDMS and ED passive samplers (ng sampler^{-1}) and estimated average water concentrations over the deployment periods (28-29 days).