Washing intermediate sample containers used in paddock surface water and stream monitoring

Wet Tropics Major Integrated Project Standard Operating Procedure WTMIP SOP 008

Version 1.0

Wet Tropics Major Integrated Project

11 March 2020

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WTMIP SOP 008 –Washing intermediate sample containers used for paddock surface water and stream monitoring

1. Purpose and scope

This document provides guidance on the process of washing intermediate sample containers (i.e. autosampler and rising stage sampler bottles) used in the Wet Tropics Major Integrated Project (WTMIP).

The method outlined in this document aims to quality assure the integrity of samples and data collected from bottles deployed in automatic samplers and rising stage samplers.

2. Training, competency and responsibilities

Program staff, contract staff, landholders and external stakeholders participating in water quality monitoring activities are provided with training in monitoring methods. Records of participant competency are maintained within the Terrain Natural Resource Management (NRM) file management system (SharePoint).

Table 1 Roles and responsibilities of WTMIP program participants

Position	Responsibilities
WTMIP Project Leader	Provide resources for the implementation and continued development of this method.
WTMIP Water Quality Project Officers (Leader/Technicians)	Ensure methods described in this method document are followed. Train new staff members in the methods. Continually review and develop the methods where appropriate.
WTMIP program staff, contract staff, landholders and external stakeholders	Follow the methods described in this document. Provide feedback to WTMIP Water Quality Project Officers for continued development of this method document.

3. Workplace health and safety

Laboratory based activities must adhere to Terrain NRM work health and safety (WHS) requirements. The following procedures and available equipment must be considered prior to undertaking laboratory work:

- Joint Corporate Nature, Terrain NRM, Cape York NRM and Northern Gulf Resource Management Group Health and Safety Policy and Procedures Manual.
- Terrain NRM Water Quality Monitoring Risk Assessment.
- Material safety data sheets, chemical spill and emergency response procedures.
- Communication methods required (e.g. signage on wash bins).
- First Aid Kit.
- Personal Protective Equipment, such as safety glasses, boots, pants, long sleeved shirt, gloves.

4. Equipment

The bottle washing space located in the MIP Tully office is set up so that risk of contamination during washing is reduced. Signage is established on all wash tubs to ensure there is no cross-contamination and for the health and safety of staff.

Equipment required for bottle washing can be found in Figures 1 to 9 below.

Figure 1 Tully office laboratory space

Figure 2 Flammable chemicals cabinet

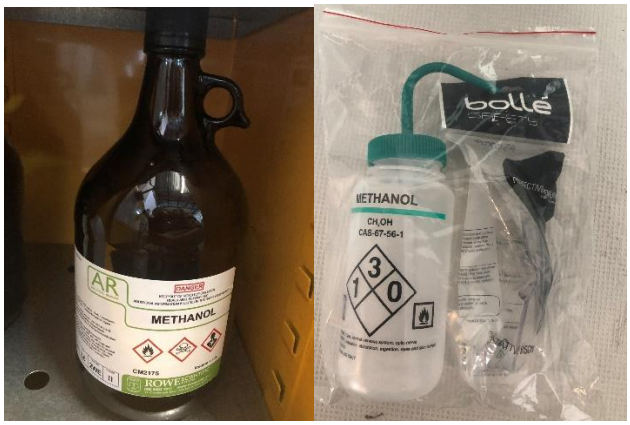


Figure 3 Methanol solution with labelled methanol bottle and safety glasses

Figure 4 Laboratory strength Decon Neutracon solution with re-usable safety gloves.

Figure 5 Reverse Osmosis (RO) water supplied by Cairns Regional Council Laboratory

Figure 6 Example signage on bottle wash tubs

Figure 7 Example of bottles soaking in bottle wash tub

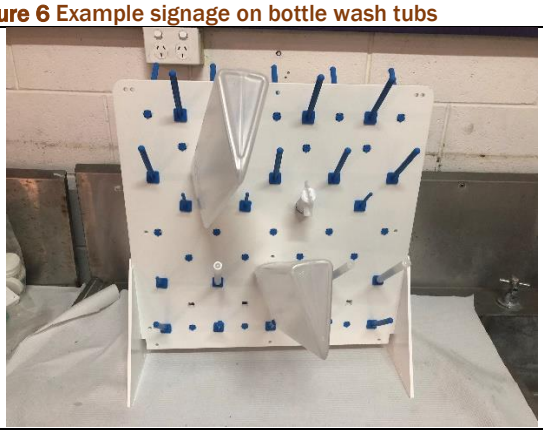


Figure 8 Bottle drying rack.

Figure 9 Disposable absorbent Labmat bench liners (photo source Fisher Scientific, fishersci.com)

5. Method

5.1 Pre-wash preparation

To ensure the Decon Neutracon detergent solution (Decon) is as effective as possible, it is important that all bottles and lids are cleaned thoroughly before they are submerged in the soak tub.

1. Collect together used bottles to be washed from the field (e.g. rising stage sampler bottles, rising stage sampler lids, rising stage sampler intake pipes, paddock sampler bottles and lids).
2. Using a clean wash cloth and soft bristled brush clean any dirt and debris from the bottles and lids using tab water in the 'laboratory' sink.

3. As each bottles/lid/pipe is cleaned, rinse three times with in RO water and place aside in a clean tub.
4. Repeat until all equipment has been washed.
5. Prepare the Decon wash in the designated tub following the Decon label instructions. Use 20 L of RO water as the base solution and ensure safety gloves are worn.

5.2 Decon detergent wash

1. Familiarise yourself with the Material Safety Data Sheet for Decon.
2. With safety gloves on, place each bottle/lid/pipe into the Decon wash. Make sure the whole bottle/lid/pipe is submerged in the solution, minimising air pockets within each item **(Figure 7)**.
3. Repeat until all equipment is submerged.
4. Replace the lid on the bottle wash tub with appropriate signage attached **(Figure 6)** and soak equipment for at least 24 hours.
5. After 24 hours, lay down fresh disposable lab mat **(Figures 1 and 9).** With safety gloves on, take out each piece of equipment one at a time and rinse 10 times with RO water. Ensure waste water is caught in the waste water tub **(Figure 1)**.
6. As each piece of equipment is rinsed place onto either the disposable lab mat (for lids and pipes) or the bottle drying rack **(Figure 8)**.
7. Leave equipment to completely dry (up to 24 hours), noting that the workspace is not a designated clean-lab area and the accumulation of dust greatly increases the chance of sample contamination.
8. For amber pesticide bottles, see **Section 5.3** for additional methanol washing steps.
9. Once equipment is dry, reassemble with lids on to prevent contamination after washing and store until required in the field. Ensure the box/shelf the equipment is stored in is clearly labelled as “Decon washed”.

5.3 Pesticide methanol wash

To ensure the removal of pesticides traces from the amber glass jars, an additional methanol wash is conducted after the Decon wash.

1. Familiarise yourself with the Material Safety Data Sheet for methanol which can be found in the flammable cupboard alongside the methanol container.
2. Take the methanol from the flammable chemical cabinet along with the methanol squirt bottle and safety glasses **(Figure 2 and 3)** and place onto a clear, clean work space. Ensure the workspace is lined with fresh disposable lab mat. Ensure the laboratory space is well ventilated by opening both garage doors and turning on the overhead fan.
3. Put on disposable rubber gloves and safety glasses. Fill the methanol squirt bottle (if required).
4. Take the amber glass pesticide sample bottles and one-by-one rinse the inside of the jar with methanol. To do this tip the jar at an angle and slowly squeeze the squirt bottle with the nose at the tip of the jar, without touching the tip of the squirt bottle on the glass. Slowly turn the amber glass jar in your hand to rinse the entire internal surface of the bottle with methanol.
5. Collect waste methanol in another glass bottle and store in the flammables cabinet until disposed of by a commercial hazardous waste contractor. Place the amber jar on the bottle rack to air dry.
6. Once completely dry (up to 24 hours) reassemble with lids on to prevent contamination and store until required in the field. Ensure the box/shelf the equipment is stored in is clearly labelled as “Decon & methanol washed”.

Please note: As the laboratory space is not a designated clean laboratory, the longer bottles are left out in the open the greater the risk of contamination via dust, fumes and other sources. It is important to reduce exposure time as much as possible and keep the wash space as clean as possible.

6. Quality control sampling

Quality control sampling for bottle washing follows the guidance of the stream event quality control requirements section 5.1 WTMIP Quality Assurance Framework 2019.

Bottle wash blanks were undertaken when first implementing the washing process to check the laboratory environment and wash procedure. Bottle wash blanks should be conducted anytime there is a change in wash conditions or laboratory environment. Three bottle wash blank checks had been performed at the time of SOP preparation. One to initially test the bottle wash method and the second and third in response to changes in environmental conditions with the arrival of a motorbike repair shop adjacent to the laboratory space and the addition of plastic autosampler bottles. The results of the bottle wash blank checks are described in **Appendix A**.

6.1 Filling washed bottles with blank samples

Using laboratory-grade ultrapure water (`MilliQ' water) a selection of washed bottles are filled and left to soak. This method is used to determine if there are any trace elements of nutrients or pesticides in the sample bottles after bottle washing.

The laboratory–grade ultrapure water ('MilliQ' water) used to prepare the sample blanks must be obtained from a NATA accredited laboratory. It is important that the MilliQ water used is fresh (i.e. no more than a week old from the date of preparation at the laboratory).

The number of bottles and soak time required is outlined in **Table 2**.

Table 2 Quantity and duration of blank bottle soak

Washed bottle type	Quantity of blanks	Soak duration & quantity		
		24hr	48hr	72hr
Rising stage samplers, plastic	3	1	1	1
Rising Stage samplers, glass	3	1	1	1
Automatic sampler, plastic	3	1	1	1
Automatic sampler, glass	3	1	1	1

Filling wash bottles with blank solution

1. Put on non-powdered disposable gloves. **These must be worn at all times during the handling of water quality samples.**
2. Fill each wash bottle with Milli-Q water and allocate a soak time. Ensure each bottle and associated soak time is clearly labelled.

6.2 Collection of blank samples

Before sampling

1. Ensure sampling packs assigned with a project number and fresh MilliQ water has been ordered through Cairns Water laboratory.
2. Put on non-powdered disposable gloves. **These must be worn at all times during the handling of water quality samples.**
3. Find the sample pack that corresponds to the correct washed bottles (e.g. MIP FS AS Blank 1, MIP – Project name, FS – Project area, AS – Program type (automatic sampler), Blank 1 – First soak (24hr)).

Filling the 500 mL suspended sediment bottle

1. Pour approximately 10 mL of sample water into the 500 mL laboratory bottle. Replace the lid on the 500 mL bottle and shake vigorously ensuring the entire inside of the bottle and lid comes into contact with the sample. Discard waste away from the work area. Repeat so the 500 mL bottle and lid are **rinsed twice** with the sample water.
2. To fill the 500 mL bottle, pour directly from the sample water container into the 500 mL bottle. Taking care not to touch the two bottles together.
3. Replace the lid of the 500 mL bottle tightly and keep refrigerated or on ice bricks in the dark at below 4°C – do not freeze 500 mL bottles.

Filling the 100 mL bottle

1. Pour approximately 10 mL of sample water into the 100 mL laboratory bottle. Replace the lid on the 100 mL bottle and shake vigorously ensuring the entire inside of the bottle and lid comes into contact with the sample. Discard waste away from the work area. Repeat so the 100 mL bottle and lid are **rinsed twice** with the sample water.

2. To fill the 100 mL bottle, pour directly from the sample water container into the 100 mL bottle. Taking care not to touch the two bottles together.
3. Replace the lid of the 100 mL bottle tightly and place into the designated water quality sample freezer to be frozen.

Filtering into the 50 mL bottle

The process to fill the 50 mL bottle requires sample water to be decanted into a syringe which is subsequently filtered into the 50 mL bottle. In undertaking this process, the syringe is considered an intermediate container and controls must be in place to mitigate the risk of sample contamination.

The following procedure should be followed to process all samples for analysis of filtered nutrients.

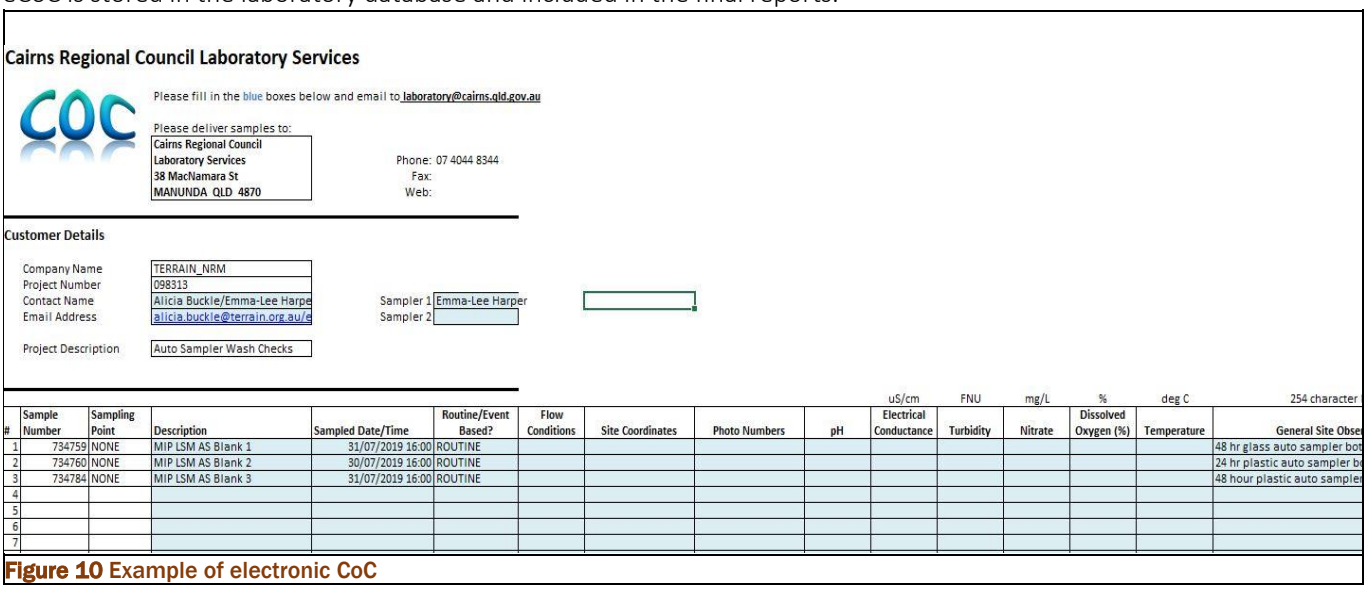
1. Attach a 0.8 µm pre-filter and 0.45 µm filter to the syringe. Connect filters without touching the end of the filter or the end of the syringe by using the plastic packaging.
2. Remove plunger from the syringe – avoid touching the internal surfaces of the barrel and plunger.
3. Fill the syringe with at least 60 mL of sample water and replace the plunger (if using a smaller 20 mL syringe, completely fill the syringe and repeat the filtering process to get the 20 mL minimum sample volume into the 50 mL bottle).
4. Discard the first 2 mL of sample water pushed through the filters as a filter rinse.
5. Remove the lid from the 50 mL bottle and filter approximately 5 mL of sample into the 50 mL bottle.
6. Replace the lid and shake vigorously ensuring all surfaces of the 50 mL bottle and lid come into contact with the water then discard waste water away from the work area in the lab/clean work space.
7. Repeat steps 5 and 6 to rinse the 50 mL bottle twice.
8. Filter the remaining Sample into the 50 mL bottle.
9. Replace the lid, ensuring a good seal and freeze the 50 mL bottle immediately.

Filling the amber glass bottle

1. To fill the amber glass bottle, pour directly from the sample water container into the glass bottle. Taking care not to touch the two bottles together. DO NOT rinse the amber glass bottle before sampling.
2. Replace the lid of the amber glass bottle tightly and keep refrigerated or on ice bricks in the dark at below 4°C – do not freeze amber glass bottles.

6.3 Record sheets

For each quality control sampling occasion there is a corresponding electronic CoC that needs to be filled out. The type of CoC and information required depends on the sampling method being used. Figure 10 shows an example electronic CoC. The electronic CoC is filled out as the samples are being collected. It is important that the information is complete and accurate as this is the link between the samples, field conditions, the laboratory and the final data set. The electronic CoC is emailed to the lab on the day of sample delivery. The information contained in the eCoC is stored in the laboratory database and included in the final reports.



Cairns Regional Council Laboratory Services

CoC

Please fill in the blue boxes below and email to laboratory@cairns.qld.gov.au

Please deliver samples to:
Cairns Regional Council
Laboratory Services
38 MacNamara St
MANUNDA QLD 4870

Phone: 07 4044 8344
Fax:
Web:

Customer Details

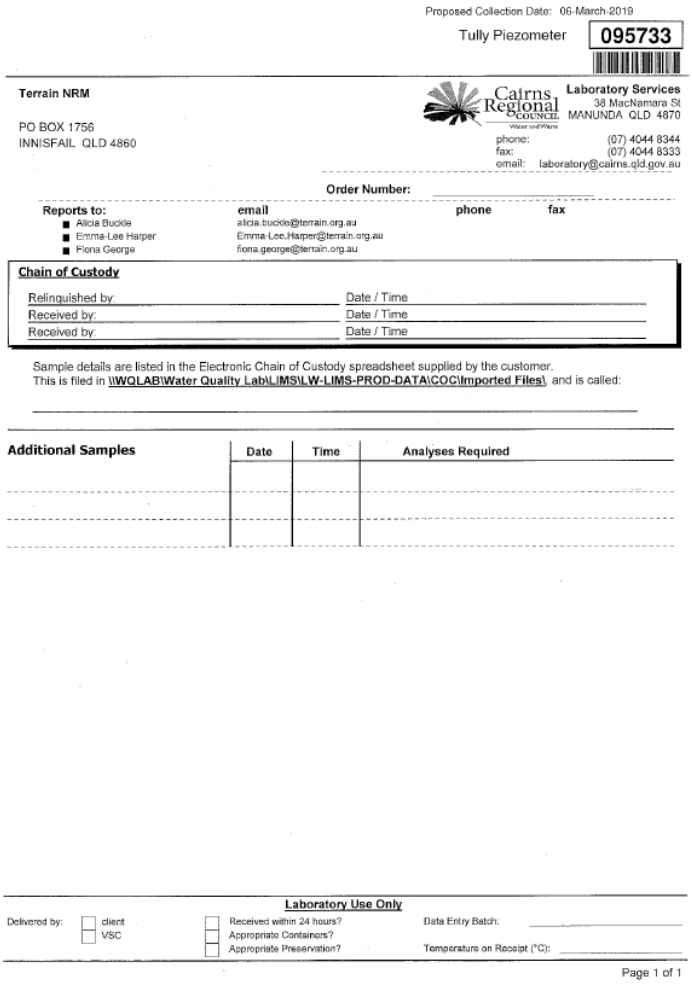
Company Name	TERRAIN_NRM
Project Number	098313
Contact Name	Alicia Buckle/Emma-Lee Harpe
Email Address	alicia.buckle@terrain.org.au/e
Project Description	Auto Sampler Wash Checks

Sampler 1 Emma-Lee Harper
Sampler 2

#	Sample Number	Sampling Point	Description	Sampled Date/Time	Routine/Event Based?	Flow Conditions	Site Coordinates	Photo Numbers	pH	uS/cm Electrical Conductance	FNU Turbidity	mg/L Nitrate	% Dissolved Oxygen (%)	deg C Temperature	254 character General Site Obse
1	734759	NONE	MIP LSM AS Blank 1	31/07/2019 16:00	ROUTINE										48 hr glass auto sampler bot
2	734760	NONE	MIP LSM AS Blank 2	30/07/2019 16:00	ROUTINE										24 hr plastic auto sampler bo
3	734784	NONE	MIP LSM AS Blank 3	31/07/2019 16:00	ROUTINE										48 hour plastic auto sampler
4															
5															
6															
7															

Figure 10 Example of electronic CoC

A paper relinquishment form is also required to be filled out and submitted along with the samples at delivery. The relinquishment form tracks the movement of samples between collection and delivery to the lab. Figure 11 shows an example of a paper relinquishment form.



Proposed Collection Date: 06-March-2019

Tully Piezometer **095733**

Terrain NRM

PO BOX 1756
INNISFAIL QLD 4860

Cairns Regional Council — Water and Waste

Laboratory Services
38 MacNamara St
MANUNDA QLD 4870
phone: (07) 4044 8344
fax: (07) 4044 8333
email: laboratory@cairns.qld.gov.au

Order Number: _____

Reports to:	email	phone	fax
■ Alicia Buckle	alicia.buckle@terrain.org.au		
■ Emma-Lee Harper	Emma-Lee.Harper@terrain.org.au		
■ Fiona George	fiona.george@terrain.org.au		

Chain of Custody

Relinquished by: _____ Date / Time _____
Received by: _____ Date / Time _____
Received by: _____ Date / Time _____

Sample details are listed in the Electronic Chain of Custody spreadsheet supplied by the customer.
This is filed in \\WQLAB\Water Quality Lab\LIMS\LW-LIMS-PROD-DATA\COC\Imported Files\ and is called:

_____

Additional Samples	Date	Time	Analyses Required

Laboratory Use Only

Delivered by: ☐ client ☐ VSC

☐ Received within 24 hours?
☐ Appropriate Containers?
☐ Appropriate Preservation?

Data Entry Batch: _____
Temperature on Receipt (°C): _____

Page 1 of 1

Figure 11 Paper relinquishment form

6.4 Sample transport

When sending samples to the laboratory, it is important to ensure an adequate number of ice bricks are placed in eskies with the samples. It is advised that chilled pesticide and/or unfiltered nutrient samples be transported in a separate esky to frozen dissolved nutrients bottles as this allows greater control over the preservation conditions. A higher ratio of ice bricks may be placed in eskies containing the frozen samples to prevent them from thawing.

To minimise contamination through leakage and sample bottles breaking, eskies should be clean, bottle lids must be on tight, glass bottles should be wrapped in padded sleeves and bottles should be packed upright. Bagged ice should only be used over ice bricks as an absolute last resort due to contamination risk from melt waters. Stocks of ice bricks are maintained in the designated sample refrigerators in the Tully, Innisfail and Cairns Terrain offices.

Given the proximity of CRC Water Laboratory to the Cairns Terrain office, and the number of WTMIP staff travelling between Innisfail/Tully and Cairns each week, it is often practical for a WTMIP staff member to deliver the samples to the laboratory in person. A courier can be used in cases when no WTMIP staff are available. Delivery by WTMIP staff is preferred over a courier to avoid the reliance on a third party and the risk that eskies may be left in a hot environment for an unacceptable period of time.

Each batch of samples shipped to the laboratory should contain the relinquishment form issued with each set of bottles prepared by CRC Water Laboratory. When packing samples, the CRC Water Laboratory project number (i.e. the 09XXXX number) on the sample bottles should be cross checked with that on the relinquishment forms accompanying the samples and on the CoC form emailed to the laboratory ahead of the sample delivery (see Section 6.3).

References

DSITI 2017. Standard Operating Procedure: FM010 Quality assurance and quality control procedures associated with the collection of water samples.
WTMIP 2019. A Quality Assurance Framework for Wet Tropics Major Integrated Project Water Quality Monitoring Programs. Prepared by Alluvium Consulting for Terrain NRM, October 2019.
WTMIP 2019. Standard Operating Procedure 007: Paddock surface water monitoring for nutrients and pesticides.

Appendix A

Rising Stage Sampler bottle wash check

A bottle wash check on rising stage samplers was conducted on the 13th March 2019 (CRC Water Laboratory project #095973) prior to the installation of rising stage samplers at event sampling sites. The wash was conducted following the procedure outlined in Section 6. Results from CRC Water Laboratory showed no trace pesticides (under limit of reporting, also known as <LOR) and three detections of nutrients. The detections of nutrients were TON and N_NO3 in blank 2 (6 hr Milli Q soak) and TON in blank 3 (1 hr 40 min Milli Q soak). All detects were at detection limit and would have negligible contribution to field results.

See the link below in SharePoint for the corresponding eCOC;

https://terrainnrm.sharepoint.com/:x:/r/projects/MIP/_layouts/15/Doc.aspx?sourcedoc=%7B1D55295C-D188-4202-8CF4-1383123379E5%7D&file=095973%20_COC_complete_MIP%20LSM_RSS%20wash%20blank_13Mar19.xlsx&action=default&mobileredirect=true&DefaultItemOpen=1

See the link below in SharePoint for the corresponding laboratory results;

https://terrainnrm.sharepoint.com/:x:/r/projects/MIP/_layouts/15/Doc.aspx?sourcedoc=%7BFCEC8B29-1F0A-47F7-9953-756D03E0CAA8%7D&file=095973_eresults_MIP%20LSM_RSS%20wash%20blank_13Mar19.csv&action=default&mobileredirect=true&DefaultItemOpen=1

Paddock surface water bottle wash check

A bottle wash check on paddock surface water auto-sampler bottles was conducted on three occasions between August 2019 and October 2019. The bottle wash check conducted on 31st August 2019 project #098313 was to verify the wash procedure outlined in Section 6. The results returned from Cairns Water Laboratory showed no detections of nutrients or pesticides (i.e. all results <LOR).

See the link below in SharePoint for the corresponding eCoC;

https://terrainnrm.sharepoint.com/:x:/r/projects/MIP/_layouts/15/Doc.aspx?sourcedoc=%7B945E9368-9E4E-4CA0-8C0A-05A4DBE968D7%7D&file=098313%20_COC_ASBottleWash_20190730.xlsx&action=default&mobileredirect=true&DefaultItemOpen=1

See the link below in SharePoint for the corresponding laboratory results;

https://terrainnrm.sharepoint.com/:x:/r/projects/MIP/_layouts/15/Doc.aspx?sourcedoc=%7B638D3936-9B67-4F7A-92B1-FC30362AF688%7D&file=098313_eresults.csv&action=default&mobileredirect=true&DefaultItemOpen=1

The following two wash tests conducted on the 18th September 2019 project #098678 and #098679 were in response to a motorcycle repair shop opening up adjacent to the laboratory area and also the addition of plastic auto-sampler bottles where previously only glass was used. The bottle wash checks on both glass and plastic bottles showed no detection of nutrients or pesticides (i.e all results <LOR).

See the link below in SharePoint for the corresponding eCOC;

https://terrainnrm.sharepoint.com/:x:/r/projects/MIP/_layouts/15/Doc.aspx?sourcedoc=%7B3E5C38C9-034B-4254-99E7-6EDAF75E227E%7D&file=098678_COC_complete.xlsx&action=default&mobileredirect=true&DefaultItemOpen=1

https://terrainnrm.sharepoint.com/:x:/r/projects/MIP/_layouts/15/Doc.aspx?sourcedoc=%7BFEA60055-B229-467E-A861-26D64D3B529C%7D&file=098679_COC_complete.xlsx&action=default&mobileredirect=true&DefaultItemOpen=1

See the link below in SharePoint for the corresponding laboratory results

https://terrainnrm.sharepoint.com/:x:/r/projects/MIP/_layouts/15/Doc.aspx?sourcedoc=%7B80B04330-1553-4FD6-9F44-824A557012C4%7D&file=098678_eresults.csv&action=default&mobileredirect=true&DefaultItemOpen=1

https://terrainnrm.sharepoint.com/:x:/r/projects/MIP/_layouts/15/Doc.aspx?sourcedoc=%7BC8FC8A70-2FFF-4B08-B36D-059C918E5D7E%7D&file=098679_eresults.csv&action=default&mobileredirect=true&DefaultItemOpen=1

Version Control

Document History	Date	Amendment:	Amended by:	Reviewed by:	Approved by:
Version 1.0	11/03/2020	SOP prepared from reference documents cited in above document.	Emma-Lee Harper (Terrain NRM)	Alicia Buckle (Terrain NRM) Romain Kobel (Terrain NRM) Aaron Davis (James Cook University – TropWater)	Charles Hammond WTMIP Manager 11/03/2020

Terrain NRM and WTMIP partner organisations acknowledge the Water Quality and Investigations team at Queensland Government Department of Environment and Science for providing SOPs from the Great Barrier Reef Catchment Loads Monitoring Program as model documents for the development of WTMIP SOPs.