

**Shaping the Future** 



Privileged and Confidential Human Health Risk Assessment - Fiskville Community Fiskville Training College 4549 Geelong-Ballan Rd, Fiskville Victoria Ref No: 212163.18 Prepared for Ashurst March 2014



### **Shaping the Future**

## **DOCUMENT CONTROL**

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## HUMAN HEALTH RISK ASSESSMENT - FISKVILLE COMMUNITY

## 4549 Geelong-Ballan Rd, Fiskville Victoria

## **EXECUTIVE SUMMARY**

Cardno Lane Piper Pty Ltd was engaged by Ashurst ("the Client") on behalf of the Country Fire Authority (CFA), to undertake a range of investigations into environmental, health and safety aspects of the site and operations at the CFA Fiskville Training College ("The Site"). The Site is located at 4549 Geelong-Ballan Rd, Fiskville, Victoria. This report relates to an assessment of the risks to the health of people from the Fiskville Community potentially exposed to contaminants and is in response to recommendations in the Report of the Independent Fiskville Investigation Report (IFI Report).

### Scope of the HHRA

The IFI Report made recommendations indicating the need for assessments of risk to CFA personnel on site as well as "downstream users of water" potentially exposed to contamination associated with fire-fighting training activities. This Human Health Risk Assessment (HHRA) was prepared in addition to these studies to address risks for any people with access to Lake Fiskville for recreational activities, referred to herein as the Fiskville Community.

This HHRA assesses risk to the following groups of people on the Site who each have various levels of exposure to potentially contaminated water from Lake Fiskville:

- Any staff member who works on the site and who may have casually accessed Lake Fiskville in their spare time for recreational purposes (e.g. swimming, fishing etc.);
- Family members of staff who resided on-site and may have casually accessed Lake Fiskville for recreational purposes; and
- People from the local community who may have accessed the site (in particular Lake Fiskville) in the past for recreational purposes.

Recreational uses of the water bodies at CFA Fiskville are no longer permitted and signs have been erected and personnel were advised of these restrictions in June 2012.

The scope of the HHRA was expanded during the course of this investigation to assess risks to people from the Fiskville Community who are potentially exposed to wind-blown foams<sup>1</sup> and/or spray drift<sup>2</sup> from training areas. This does not include assessment of risks for potential occupational exposures in training areas which is addressed in a separate report "*Summary Report - Human Health Risk Assessment –CFA Fiskville Training College*" (Cardno Lane Piper 2014a)...

<sup>&</sup>lt;sup>2</sup> Spray drift results from the use of water in training exercises. Spray drift is unlikely to contain PFOS and PFOA since June 2012 as CFA ceased using recirculated dam water in training and switched to town mains water only for training purposes.



<sup>&</sup>lt;sup>1</sup> Wind-blown foam results from two sources: i) use of foam products in training and ii) foams generated in Dam 1 as a result of a mechanical aerator. The makeup of the foam is dependent on the source of foam.

#### HHRA Methodology

The methodology employed in this section is consistent with the guidelines of the Australian enHealth Council (enHealth 2012), and the Australian National Environmental Protection Measure for contaminated sites (NEPC 1999) and was conducted in the following four steps:

- 1. Issue identification
- 2. Exposure assessment
- 3. Dose Response
- 4. Risk characterisation.

The risk characterisation component of the HHRA includes a combination of methodologies for defining risk including:

- Quantitative analysis: This is based on blood serum data collected as part of a health surveillance program for people from the Fiskville Community.
- Qualitative Analysis: A general discussion of risks for all other types of exposure.

#### **Chemicals of Potential Concern and Exposure Pathways**

Perfluorinated Chemicals (PFCs) were identified as Chemicals of Potential Concern (CoPC) in water (Section 3.1). At the Site, various PFCs have been detected in soil, surface waters and sediments. This is due to the use of Class B fire-fighting foams used in the training of fire-fighters to fight liquid fuel fires. PFCs are assessed in this HHRA in three distinct groups which are represented by a surrogate chemical as follows:

- Perfluorinated alkyl sulfonates (PFAS) using pefluorooctane sulfonate (PFOS) as a surrogate,
- Perfluoroalkyl carboxylic acids (PFAA) using Perfluorooctane carboxylic acid (PFOA) as a surrogate, and
- Other Perfluorinated Chemicals (OPC) using 6:2 fluorotelomer sulfonate (6:2FTS) as a surrogate.

CoPC in this HHRA are restricted to those attributed to site related activities, i.e. fire-fighter training activities. This HHRA does not include assessment of risks of chemicals, including microbial pathogens, which may be present as a result of regional influences (e.g. thermotolerant coliforms sourced from animal faeces which are washed into waterways).

### **Exposure Scenarios**

A range of exposure pathways are assessed in this HHRA (see Section 3.2) including:

- Direct exposure pathways such as incidental consumption of water during recreational activities; and
- Secondary exposure pathways which include consumption of fish caught from the lake.

Potentially complete exposure pathways are identified and used as the basis for defining five different exposure scenarios.

The amount of exposure assumed for each scenario was ranked based on a qualitative assessment (Section 3.3). The amount of exposure assessed for each scenario is as follows:

 Scenario S1: exposure for people who have entered Lake Fiskville on a single or occasional basis is considered very low.



- Scenario S2: exposure for people previously involved in recreational activities in Lake Fiskville including swimming is considered low.
- Scenario S3: Exposure for people encountering spray drift leaving the training area is considered low.
- Scenario S4: Exposure for people consuming meat from wild rabbit caught on-site is considered medium. This is considered medium as the concentrations of PFOS found in rabbit meat are high but the rate of consumption of rabbit meat in Australia is low.
- Scenario S5: The potential for exposure of people who consume fish caught on-site is assumed to be high. This is based on the high PFOS concentrations in fish flesh.

The exposure assessment for each scenario has been made relative to that for people exposed by eating fish caught from Lake Fiskville (Scenario S5) which is the highest potential exposure in the Fiskville Community. Due to the very high concentrations of PFC in fish the exposure from this pathway is considered to be much higher (potentially orders of magnitude higher) than primary exposure pathways (e.g. due to direct contact with water) and also some secondary exposure pathways (e.g. consumption of local produce).

### Assessment of Chemical Risks

Risks characterisation was undertaken as follows:

- A quantitative assessment of chronic risks was performed by Dr Roger Drew<sup>3</sup> (ToxConsult 2014, Appendix G) for Scenario S5: Consumers of fish. This scenario represents the assumed highest exposure:
  - Compared against background exposures;
  - Compared against a safe blood serum concentrations; and
  - Used to calculate a Margin of Exposure (MOE);
- A qualitative assessment of risk for other assumed long-term scenarios S2 (past swimmers in the lake), S3 (exposure to spray drift) and S4 (consumers of rabbit meat); and
- A qualitative assessment of risk for single or occasional exposure scenarios (S1: casual entry in the lake)

It is important to note in the ToxConsult study that PFC blood serum data was collected from people in the Fiskville Community who volunteered to take part in a health surveillance program. This program included people who consumed fish from Lake Fiskville, some of which were also workers from the training area considered to be within the 'medium' and 'high' relative risk of exposure group identified in Chapter 7 of the IFI Report (Joy 2012).

The results of the risk characterisation for the five scenarios considered in this HHRA are that *risks are considered negligible* for people who:

- Casually entered the lake (Scenario S1);
- May have swum in the lake (Scenario S2);
- Were exposed to spray drift (Scenario S3);
- Consumed meat from rabbit hunted on-site (Scenario S4); and
- Consumed fish caught from Lake Fiskville (Scenario S5).

The finding of negligible risk for people who consume rabbit meat (Scenario S4) or fish (Scenario S5) is based on findings of negligible risk from a health impact assessment

<sup>&</sup>lt;sup>3</sup> Dr Roger Drew, PhD, DABT, Toxicologist and Risk Assessor, is one of Australia's leading toxicologists and has over 40 years of experience in his field of expertise.



conducted by Dr Roger Drew from ToxConsult (2014) for people who consumed fish from Lake Fiskville. Blood serum concentrations of PFC were measured for people who ate fish as part of a CFA health surveillance program which was initially offered to people considered to be within the 'medium' and 'high' relative risk of exposure group identified in chapter 7 of the IFI Report (Joy 2012), and was later extended to include people who may have eaten fish from the lake<sup>4</sup>.

PFC serum concentrations were used for the following reasons:

- Significant uncertainties in the data precluded assessing health risk from eating fish using a traditional tolerable daily intake (TDI) approach; and
- Toxicological effects of PFOS are directly related to blood serum concentrations.

Toxicologist Dr Roger Drew and the CFA medical officer (ToxConsult 2014) both concluded that they do not expect there to be any health implications arising from the concentrations of PFOS measured in the serum of the persons investigated. This is based on results indicating:

- A few individuals had PFOS concentrations at, or slightly above, the upper edge of the background range<sup>5</sup>; and
- None of the individuals examined had changes in their blood parameters characteristic of PFOS, or which correlated with their PFOS blood serum concentration<sup>6</sup>.

### Conclusions

The risks estimated for people from the Fiskville Community potentially exposed to PFCs present in water or fish and rabbits caught on-site are considered negligible.

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<sup>&</sup>lt;sup>6</sup> Some persons had blood parameters outside the reference ranges but these were associated with existing health conditions, medication or admitted lifestyle factors.



<sup>&</sup>lt;sup>4</sup> Twelve of the 22 participants in the 'fish consumption' study indicated that they had eaten fish or eel from the Lake in the past

<sup>&</sup>lt;sup>5</sup> These results are higher than what is expected for the majority (95%) of the general population. Nevertheless they were still markedly less than serum concentrations in factory workers making PFOS, and for whom there are no PFOS associated changes in blood parameters or demonstrable illness.

## HUMAN HEALTH RISK ASSESSMENT - FISKVILLE COMMUNITY

## 4549 Geelong-Ballan Rd, Fiskville Victoria

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## LIST OF ABBREVIATIONS AND UNITS

## **Chemical Names**

6:2 FTS	6:2 fluorotelomer sulphonic acid
BGA	Blue-green algae
BTEX	Benzene, Toluene, Ethylbenzene & Xylenes (subset of MAH)
MAH	Monocyclic Aromatic Hydrocarbons
OCP	OrganoChlorine Pesticides
OPC	Other perfluorinated chemicals
OPP	Organophosphate Pesticides
PAHs	Polycyclic Aromatic Hydrocarbons
PCBs	Polychlorinated Biphenyls
PFCs	Perfluorinated Chemicals
PFAA	Perfluorinated alkyl carboxylic acids
PFAS	Perfluorinated alkyl sulfonic acids
PFOA	Perfluorooctanoic acid
PFOS	Perfluorooctane sulphonic acid
SVOC	Semi-Volatile Organic Chemicals
TPH	Total Petroleum Hydrocarbons
TRH	Total Recoverable Hydrocarbons (= TPH)
VOC	Volatile Organic Chemicals

## **Technical Terms**

ANZECCAustralian and New Zealand Environment and Conservation CouncilASTAboveground Storage Tank



CoPC	Chemicals of Potential Concern
enHealth	Environmental Health Committee ( <i>enHealth</i> ), a subcommittee of the Australian Health Protection Committee (AHPC).
GME	Groundwater Monitoring Event
HHRA	Human Health Risk Assessment
HI	Hazard Index
HQ	Hazard Quotient
ME	Monitoring Event
NHMRC	National Health and Medical Research Council
N/A	Not Applicable
NEPM	National Environmental Protection Measure
OHS	Occupational Health and Safety
QMRA	Quantitative Microbial Risk Assessment
RME	Reasonable Maximum Exposure
TDI	Tolerable daily intake
ТІТ	Triple Interceptor Trap
TRV	Toxicity Reference Value
USEPA	United States Environment Protection Authority
UST	Underground Storage Tank
WHO	World Health Organisation

## Units

DALY	Disability Adjusted Life Year
EU/m <sup>3</sup>	Endotoxin units per cubic metre
LRV	Log Reduction Values
mg/kg	Milligram per Kilogram (approximately equivalent to ppm)
mg/L	Milligram per Litre
ML	Megalitres
ppb	Part per Billion
ppm	Parts per Million
µg/kg	Microgram per Kilogram (approximately equivalent to ppb)
µg/L	Microgram per Litre

## Site Specific

CFA	Country Fire Authority
FL PAD	Flammable Liquids PAD
IFI	Independent Fiskville Investigation
MMFB	Melbourne Metropolitan Fire Brigade



PAD	Practice Area for Drills
RTG	Regional Training Ground
WS Pit	Water Supply Pit



## HUMAN HEALTH RISK ASSESSMENT - FISKVILLE COMMUNITY. FISKVILLE TRAINING COLLEGE

## 4549 Geelong-Ballan Rd, Fiskville Victoria

## 1 INTRODUCTION

### 1.1 Background

Cardno Lane Piper Pty Ltd was engaged by Ashurst ("the Client") on behalf of the Country Fire Authority (CFA), to undertake a range of investigations into environmental, health and safety aspects of the site and operations at the CFA Fiskville Training College ("The Site"). The Site is located at 4549 Geelong-Ballan Rd, Fiskville, Victoria as shown in Appendix A, Figure A1).

The features at the site relevant to this assessment are shown in Figure 1-1 below. The fire training operations areas in the centre of the site include the Practical Area for Drills (PAD) incorporating the Flammable Liquids (FL) PAD. A more detailed site features plan is shown in Appendix A, Figure A2.

The Report of the Independent Fiskville Investigation (IFI Report, Joy 2012) prepared by Professor Rob Joy concluded, amongst other things, that there is a need to address risks posed by chemical contamination for downstream users and personnel working in the PAD (assessed in separate reports). This is evident from **IFI Recommendation 3** which states:

*"……that further investigation be undertaken into surface waters in and discharging from Lake Fiskville to:* 

- Better quantify the risk to downstream human health receptors, taking into account downstream dilution and environmental fate and transport mechanisms;
- Investigate potential sources of PFOA and PFOS discharges to Lake Fiskville and discharging off site, if the potential risk of adverse impact on downstream human health receptors is found to be unacceptable;
- Collect surface water samples at a representative location to assess whether the reported copper and zinc concentrations are consistent with background levels; and assess the ecological condition of Lake Fiskville."

The risk assessment was expanded to include any people who had access to water from Lake Fiskville, herein referred to as the Fiskville Community.



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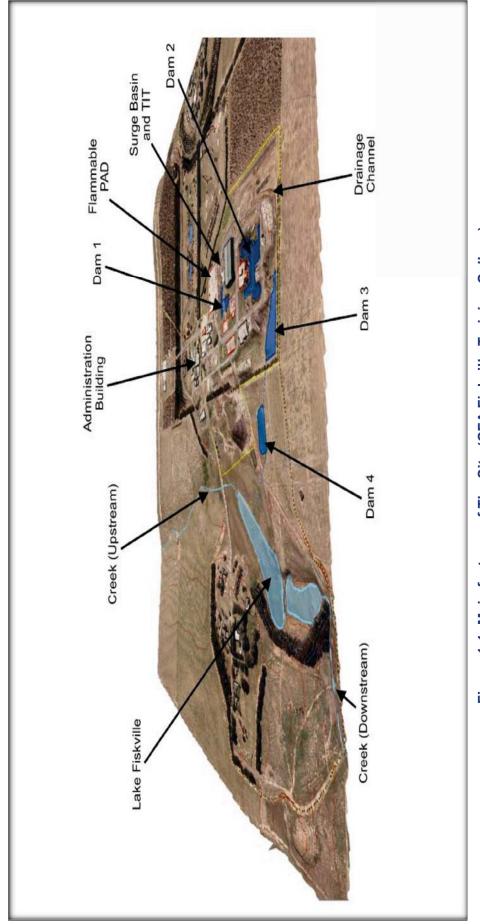


Figure 1-1: Main features of The Site (CFA Fiskville Training College)



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The scope of the HHRA was expanded further during the course of this investigation to assess risks to people from the Fiskville Community who are potentially exposed to chemical compounds<sup>7</sup> in fish caught in the lake and also rabbits potentially taken. Exposure to windblown foams<sup>8</sup> and spray drift<sup>9</sup> from the training area was also added to the assessment. This HHRA however does not include the assessment of risks for potential occupational exposures which is addressed in a separate report (Cardno Lane Piper 2014a).

In response to the IFI Report, Cardno Lane Piper has undertaken the following investigations (reported separately):

- The risks to human health for people downstream have been assessed in another HHRA in a document titled "Human Health Risk Assessment – Downstream Users, Fiskville Training College" (Cardno Lane Piper 2014b).;
- The potential sources of PFOS and PFOA and a range of other chemicals and metals has been documented in a report titled "*Surface Water and Sediment Contamination Assessment, CFA Fiskville Training College*" (Cardno Lane Piper 2014c); and
- The ecology of Lake Fiskville and the water bodies downstream has been assessed and documented in a report titled *"Aquatic Ecology Assessment, Fiskville Training College"* (Cardno 2014).

### **1.2** Other Related Recommendations from the IFI Report.

The IFI Report made a number of other recommendations relevant to risk assessment. These have been interpreted in the context of the overall understanding of the key risk issues for human health and to identify impacts on the aquatic ecology both on site and off site. The approach taken by Cardno Lane Piper to the assessment of risks and how this relates to the other IFI recommendations is summarised as follows:

#### IFI Report Recommendation 5:

"....that any subsequent study of possible linkages between exposure of persons during training at Fiskville to materials such as flammable liquids and health effects evaluate the usefulness of the qualitative assessment of relative risk of exposure of different groups developed in Chapter 7".

Cardno Lane Piper has not undertaken any work in relation to this as it relates to exposures of personnel to flammable chemicals in the past and is being addressed in a separate study being undertaken by Monash University.

#### IFI Report Recommendation 6:

*"…that procedures be put in place to protect the health of personnel potentially exposed to waters and sediments in Dams 1 and 2 of the firewater treatment system and, in* 

<sup>&</sup>lt;sup>8</sup> Wind-blown foam results from two sources: i) use of foam products in training and ii) foams generated in Dam 1 as a result of a mechanical aerator. The makeup of the foam is dependent on the source of foam <sup>9</sup> Spray drift results from the use of water in training exercises. Spray drift is unlikely to contain PFOS and PFOA since June 2012, as CFA ceased using recirculated dam water in training and switched to town mains water only for training purposes.



<sup>&</sup>lt;sup>7</sup> Compounds may be defined as a substance that is made of two elements chemically combined (i.e. a chemical or more broadly as a substance composed of multiple parts or ingredients (e.g. soap). The term chemical is used in this HHRA when referring to chemical compounds.

particular, to manage the risks to individuals who have the potential to come into contact with sediments in the dams during routine maintenance".

In response, Cardno Lane Piper has reviewed the potential exposures of current day CFA personnel including maintenance workers for the purpose of developing the current risk assessment and also preparing advice on upgrading the CFA Standard Operating Procedures for Health & Safety Management. The response to this recommendation has been extended to consider the health risk to personnel involved in hot-fire training drills using the Dam 2 water (and was reported in a document titled "*Summary report - Human Health Risk Assessment – CFA Training Personnel*" (Cardno Lane Piper 2014a). This HHRA is used to inform many of the decisions to be made in relation to upgrades to water systems and practices for future hot fire training. It will also provide the basis for development of a 'fit for purpose' non-potable and sustainable fire-training water supply into the future. This is documented in a report titled "*Fire Training Water Quality Criteria – CFA Training Grounds, Victoria*" (Cardno Lane Piper 2014d).

#### IFI Report Recommendation 8:

"....that historical landfill 1 which has been disturbed by the construction of a walking track needs to have its extent clearly identified, have an appropriate impermeable and properly drained cap constructed and be revegetated with shallow rooting species that will not compromise the integrity of the cap. This should ensure the safety of any people using the walking track".

In response, Cardno Lane Piper has undertaken an investigation into the landfill area to assess risk to people potentially exposed to the landfill area including those using the running track. This is documented in a separate report titled "*Investigation of Risks at Former Landfills, Fiskville Training College*" (Cardno Lane Piper 2014e). This includes a plan for on-going management of the landfills.

#### IFI Report Recommendation 10.

*"……that the site specific recommendations of the Golder Associates' Preliminary Site Assessment – CFA Regional Training Grounds be adopted including recommendations to:* 

- Undertake targeted soil and groundwater investigations at sites where possible sources of contamination have been identified;
- Assess fire fighting water quality for contaminants associated with flammable liquids and extinguisher foams;
- Assess water quality where discharges occur to the environment".

In response, Cardno Lane Piper has commenced a program of assessments of the Regional Training Grounds (RTGs). The findings of the human health and ecological assessments being prepared for the Fiskville Site are likely to be relevant to the future management of the RTGs including the use of fire training water.

### **1.3 Site Description**

The Site, shown above in Figure 1-1, is relatively flat in the central and eastern portions of the site. In the western part of the Site the land slopes towards the Beremboke Creek and Lake Fiskville. Site features relevant to recreational activities and shown in Appendix A, Figure A2 include:

• Lake Fiskville located on the south western portion of the site;



- Housing for CFA staff which gives people to the site 24-hour access. Housing for staff has been available since the early 1970s (expanded in 1987). It now comprises:
  - Four (4) cottages located near the eastern boundary of the Site<sup>10</sup> (Site Feature 10); and
  - Ten (10) houses located in the residential area west of Lake Fiskville (Site Feature 11a • and 11b).
- An accommodation and hospitality precinct on the Eastern boundary of the site (Site Feature 9):
- A 9 hole golf course (Site Feature 54); and
- A running track (see legend).

Water impacted with contaminants from hot-fire training activities on-site enters Lake Fiskville from a series of water retention dams (Dams 1 to 4) and drainage channels thereby providing a potential pathway for people to be exposed to contamination. A description of these surface water bodies which are related to CFA fire-fighter training activities and impact on Lake Fiskville at the site is provided in Appendix B. A more detailed description of site features can be found in the report on site history titled "Site History Review" (Cardno Lane Piper 2014f).

A number of management initiatives have been implemented by CFA at the Site since the release of the IFI Report. The following initiatives have been implemented to reduce potential exposures to people from the Fiskville Community:

- Banning of recreational activities (e.g. fishing, swimming) in water bodies at the site. Lake Fiskville and the dams have been signposted accordingly;
- Management authorisation required prior to hunting activities being conducted on The Site;
- Investigation into the feasible options for remediation of water bodies at The Site including • Lake Fiskville:
- Development of a water management strategy to provide clean water and treat • contaminated water generated during training;
- Altering the training program at the site to minimise the potential contaminant load in to • Lake Fiskville; and
- Construction of a bypass channel to divert Beremboke Creek around Lake Fiskville. This will prevent its flow through Lake Fiskville so as to minimise discharges from the lake.

#### 1.4 Purpose and Objectives of this HHRA

The purpose of this HHRA is to identify risks to individuals considered to be a part of the Fiskville Community.

The 'Fiskville Community' is defined for the purpose of this report as:

- Any staff member who works on the site and may casually access Lake Fiskville in their spare time<sup>11</sup> for recreational purposes (e.g. swimming, fishing etc.);
- Family members of staff who reside on-site and may casually access Lake in their spare time for recreational purposes; and
- People from the local community who may have accessed the site (in particular Lake Fiskville) in the past for recreational purposes.



<sup>&</sup>lt;sup>10</sup> The hospitality precinct at The Site includes a Main Dining Room, Lounge and recreational rooms.

<sup>&</sup>lt;sup>11</sup> CFA personnel are not required to enter any water body at the site as part of their training or employment, except

The specific objectives of the HHRA are to:

- 1. Conduct a Human Health Risk Assessment to estimate the potential for impacts upon people of the Fiskville Community from exposures to chemicals in water in Lake Fiskville.
- 2. Provide recommendations regarding actions required to eliminate or effectively manage risks identified.



## 2 RISK ASSESSMENT CONTEXT & METHOD

### 2.1 What is a risk assessment?

A HHRA is the process that estimates the potential for impact on specified human population(s) as a result of exposure to chemical hazards for a certain period of time (enHealth 2012). The impacts may be assessed as a result of the exposure of people to chemical contaminants in air, water, soil and/or food or pathogenic microbiological contaminants in food and water.

A risk assessment is a tool that gathers and organises information to ascertain whether further management action is necessary. This then allows the risk assessment to be used as a tool "to provide complete information to risk managers, specifically policymakers and regulators, so that the best possible decisions are made" (Paustenbach 1989).

Risk assessments may be performed in a "screening" manner in which the evaluation of risk is inherently conservative by use of conservative assumptions. This is termed a Tier 1 Risk Assessment and is considered a cautious approach. This approach is adopted if little is known about exposure, a quick assessment is being conducted and/or the level of uncertainty in the risk assessment is high. The level of risk identified in a screening assessment may necessitate that more site-specific data be acquired which escalates the assessment to a Tier 2 or Tier 3 Risk Assessment. The collection of more site specific data typically serves to decrease the level of uncertainty in an assessment of risk.

Most scenarios in this HHRA are assessed in a qualitative fashion which is consistent with a Tier 1 risk assessment. However, one scenario is assessed in a quantitative manner by ToxConsult (2014) and discussed in terms relative to other scenarios (where relevant).

### 2.2 HHRA Methodology

This HHRA is conducted to establish the risks associated with exposure to chemicals in the surface water of Lake Fiskville as briefly described in the objectives above (Section 1.3). Risk to human health was assessed for 'acute' (short one-off) and 'chronic' (prolonged and/or repeated) exposure types.

The steps used in conducting this HHRA are shown below in Figure 2-1. It includes the following four steps as per Australian guidelines for conducting risk assessments which are accepted by the Environmental Protection Authority (EPA) and the Department of Health (DoH), namely enHealth (2012):

- **Issue Identification:** Identifying the people who are exposed, where they are exposed and how they are exposed to the Chemicals of Potential Concern (CoPC) present in water and/or sediment.
- **Exposure assessment**: A description of assumed exposure for various risk scenarios being considered.
- **Hazard Assessment:** This includes a summary of the relationship between a dose of a CoPC and adverse health effect(s) based on latest toxicological information from published human and/or animal exposure studies.
- **Risk Characterisation**: This considers the significance of risks to people exposed to CoPC (issue identification) by comparing the level of exposure (exposure assessment) with a tolerable dose (hazard assessment).



#### **Privileged and Confidential** Human Health Risk Assessment - Fiskville Community 4549 Geelong-Ballan Rd, Fiskville Victoria



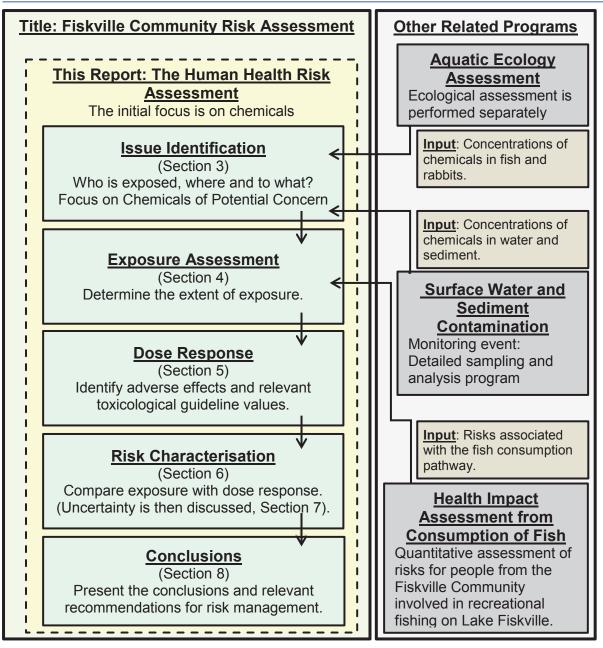


Figure 2-1: Outline of the Health Risk Assessment process and other related programs



A qualitative assessment of risk is primarily conducted for scenarios considered in this HHRA unless warranted and exposure assessment is well defined.

A quantitative assessment of risks has been performed by Dr Roger Drew<sup>12</sup> (ToxConsult 2014, Appendix G) for people who consumed fish caught recreationally from Lake Fiskville (see Section 3.3). Consumers of fish are considered to represent the assumed highest exposure group over the long-term in the Fiskville Community. The assessment performed by ToxConsult (2014) is based on highly site specific data, i.e. PFC blood serum data collected from people in the Fiskville Community who volunteered to take part in a health surveillance program. It includes people who consumed fish from Lake Fiskville as well as workers from the training area considered to be within the 'medium' and 'high' relative risk of exposure group as identified in Chapter seven of the IFI Report (Joy 2012). This PFC serum concentration was:

- Compared against background exposures; Background concentrations of PFC in the general communities was identified as <0.1mg/L. People with serum concentrations below this level were considered to have levels at background concentrations;
- Compared against a safe serum concentration: A human serum level considered without effect of 2 mg/L based on a number of methods (occupational epidemiological studies, no observable effect levels in animals and tolerable daily intakes); and
- Used to calculate a Margin of Exposure (MOE): "Calculation of margin of exposure is a standard risk characterisation method widely used by Australian Authorities" (ToxConsult 2014).

<sup>&</sup>lt;sup>12</sup> Dr Roger Drew, PhD, DABT, Toxicologist and Risk Assessor, is one of Australia's leading toxicologists and has over 40 years of experience in his field of expertise.



## **3** ISSUE IDENTIFICATION

This HHRA addresses potential exposures and risks to the Fiskville Community. The following is included as part of the issue identification process described in this section:

- The Chemicals of Potential Concern (CoPC), (Section 3.1);
- Identification of the potential exposure pathways (Section 3.2); and
- Detailing the Scenarios considered in this HHRA (Section 3.3).

### 3.1 Chemicals of Potential Concern

### 3.1.1 Water and Sediment Investigations

Three separate monitoring events have been conducted to characterise the extent of contamination in sediments and water at the Site, as summarised below:

- A monitoring event completed in 2012 as part of the IFI report (Golder 2012);
- The monitoring event conducted by Cardno Lane Piper in August 2012 (Cardno Lane Piper 2014c). The aim of this monitoring event was to further characterise the extent of contamination of water and sediment in surface water bodies at the Site and nearby downstream; and
- A further monitoring event by Cardno Lane Piper in June 2013 (Cardno Lane Piper 2014g) to further investigate concentrations of PFOS and PFOA (including extended PFC screen) in Lake Fiskville and at downstream sampling locations extending to the Moorabool River.

A summary of the results for these monitoring events is provided in Appendix C along with a summary of the data quality and relevant analytical results for the Cardno Lane Piper monitoring event (Cardno Lane Piper 2014c).

The following organic and inorganic chemicals were identified in the water and/or sediment of Lake Fiskville:

- Organic chemicals:
  - Perfluorinated chemicals;
    - Perfluorooctane sulphonic acid (PFOS) <sup>13</sup> including (but not limited to);
    - Perfluorooctanoic acid (PFOA); and
    - 6:2 fluorotelomer sulphonic acid (6:2 FTS)
  - Dioxins; and
  - BaP (Benzo(a)pyrene).
- Inorganic chemicals:
  - Metals including arsenic, chromium (total), copper, lead, nickel and zinc;
  - Ammonia (as nitrogen);
  - Fluoride;
  - Nitrate;

<sup>&</sup>lt;sup>13</sup> The following compounds were tested in both monitoring events; PFAS: Perfluorobutanesulfonic acid (PFBS), erfluorohexanesulfonic acid (PFHxS), Perfluorodecanesulfonic acid (PFDS), PFAS: Perfluorohexanoic acid (PFHxA), Perfluoroheptanoic acid (PFHpA),), Perfluorononanoic acid (PFNA), Perfluorodecanoic acid (PFDA), Perfluoroundecanoic acid (PFUnA), Perfluorododecanoic acid (PFDoA), Perfluorotridecanoic acid (PFTrA), Perfluorotetradecanoic acid (PFTeA), OPC: Perfluorooctanesulfonamide (PFOSA) and 1H,1H,2H,2Hperfluorodecanesulfonic acid (8:2 FTS).



- Nitrite; and
- Sulphate.

Note that the CoPC were selected in a screening process which considered the data for a large number of chemicals and indicators included in the analytical schedule such as:

- Inorganic chemicals: major anions/cations, BOD, COD, Ionic Balance, F-, Na, NH<sub>3</sub>, NO<sub>3</sub>, Nitrogen (Total), pH, Reactive Phosphorus as P, SO<sub>4</sub><sup>2-</sup>, TKN, Total F and TDS);
- Biological: Faecal Coliforms, Coliforms, E-coli, Total Coliforms (Colilert), Pseudomonas aeruginosa;
- Organics chemicals: perchlorate, TPHs, MAHs, PAHs/ Phenols, VOCs, SVOCs, BTEX, PFC (including PFOS, PFOA, 6:2 FtS), Amino Aliphatics, Amino Aromatics, Anilines, Chlorinated Hydrocarbons, Explosives, Halogenated Benzenes, Halogenated Hydrocarbons, Halogenated Phenols, Herbicides, Nitroaromatics, Organochlorine Pesticides, Organophosphorous Pesticides, Pesticides, Phthalates, PCB and Solvents.
- Metals: aluminium, antimony, arsenic, barium, beryllium, boron, cadmium, cobalt, chromium, copper, iron, lithium, magnesium, manganese, molybdenum, mercury, nickel, potassium, phosphorous, lead, selenium, silver, titanium, vanadium and zinc.

### 3.1.2 Chemicals Assessed in the HHRA

A screening assessment was performed to identify chemicals that are designated the term "Chemicals of Potential Concern (CoPC)" requiring further assessment (see Appendix C, Section 2). This was in order to discriminate these chemicals from a much larger number of chemicals that are typically identified in water and sediment studies. The following chemicals were identified as being of potential concern following screening and are assessed further in this HHRA:

- Perfluorinated chemicals:
  - Perfluorinated alkyl sulfonic acids (PFAS) assessed using PFOS as a surrogate<sup>14</sup>;
  - Perfluorinated alkyl carboxylic acids (PFAA) assessed using PFOA as a surrogate<sup>15</sup>; and
  - Other perfluorinated chemicals (OPC) assessed using 6:2 FTS as a surrogate<sup>16</sup>.

The reported concentrations of these CoPC adopted for the HHRA are shown in Table 3-1. The concentrations reported for the PFCs are from Lake Fiskville.

Chemicals	Screening value	Maximum Concentration
PFAS	0.2	47
PFAA	0.4	13
OPC	0.2	32
Note: Source of screening value = Provisional Health Advisories for drinking Water (USEPA 2009)		

### Table 3-1: Chemicals Identified in Water of Lake Fiskville (µg/L).

<sup>&</sup>lt;sup>16</sup> Very little toxicological data is available for the remaining PFCs. The basis of selecting the fluorotelomer, 6:2FTS, as the surrogate for this class is because it was identified in water and sediment in both monitoring events and is believed to be the PFC formulated in the class B foam product currently used by CFA.



<sup>&</sup>lt;sup>14</sup> The toxicological database for this chemical is large and complex (Appendix E). Other sulfonic acids are anticipated to have similar toxicity however toxicity is assumed to increase with length of the fluorinated alky chain present.

<sup>&</sup>lt;sup>15</sup> This was based on toxicity of PFOA for the same reason given for PFOS (see previous dot point).

*Escherica coli* was not selected as a CoPC even though it was detected in 6 samples out of 20 collected from Lake Fiskville (ranging from 72 to 330 organisms per 100mL). This is because E. coli is present as a result of regional activities and is typically identified in surface water bodies throughout Victoria. The mean *E. coli* levels reported in the literature for surface water bodies in regions with intense agriculture practice is 210 organisms per 100 mL whereas in urbanised areas it is 450 organisms per 100 mL. During rain events the mean level of *E. coli* in surface water bodies, in intense agricultural areas, increases by orders of magnitude (up to 17,700 organisms per 100 mL) (CRC 2004). E. coli is not present in water as a result of fire-fighter training activities at the site and it is beyond the scope of this HHRA to consider risks associated with this or other microbial pathogens.

### 3.1.3 Summary of Perfluorinated Chemicals in Various Media

A summary of data collected for PFCs in various media is provided here. This information is sourced from the reports prepared by Cardno Lane Piper including:

- Cardno Lane Piper (2014c). Surface Water and Sediment Contamination Assessment. Fiskville Training College, 4549 Geelong – Ballan Road, Fiskville, Victoria. Prepared for Ashurst.
- Cardno Lane Piper (2014g). Supplementary Surface Water and Sediment Sampling Downstream. Fiskville Training College, 4549 Geelong – Ballan Road, Fiskville, Victoria. Prepared for Ashurst.
- Cardno (2014). Aquatic Ecology Assessment, Fiskville Training College, Victoria, Country Fire Authority.

For some data, the sampling and/or data quality has not yet been reported (soil data away from training areas, fish data quality, and rabbit data). This information can be found for soil in Appendix D, fish in Appendix E and rabbit in Appendix F of this report. Refer to the source documents for other media such as the Surface Water and Sediment Contamination Assessment (Cardno Lane Piper 2014c). A summary of data quality is provided in Appendix C. References to data should be made to the source document.

A summary of PFC concentrations in various media is provided below in Table 3-2. See Appendix C for an extended summary and refer to Cardno Lane Piper reports for a more detailed analysis.

Media	Reported Concentrations of PFOS, PFOA and/or 6:2FTS
Water in Lake Fiskville	Maximum PFC concentrations in Lake Fiskville range from 32 $\mu$ g/L for OPC to 47 $\mu$ g/L for PFAS (Appendix C).
Water in spray drift	Water used in training was sourced from Dam 1 & 2 prior to June 2012. Therefore the maximum concentration of PFOS in spray drift prior to 2012 is anticipated to be approximately 200µg/L (Cardno Lane Piper 2014c).
Sediment	The maximum PFC level in sediment (<1mg/kg for the sum of PFAS, PFAA and OPC concentrations, Appendix C) is below the adopted human health screening criterion (6mg/kg for PFOS).
Soil away from training areas	PFOS was detected at very low levels in surface soil away from the training area on the Site. It is considered most likely that spray-drift from training on the FL PAD prior to June 2012 is the source (Appendix C and Appendix D).
Garden Produce	No data available for garden produce. However, PFOS was detected in 2 of 9 grass samples on an adjacent property (within 600m of the FL PAD) with a maximum concentration of 10 ng/g (Cardno Lane Piper 2014h). Note that PFOA and PFOS are

#### Table 3-2: PFCs in various media at the Site.



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Media	Reported Concentrations of PFOS, PFOA and/or 6:2FTS
	likely to be transferred from soil to the vegetative compartment of plants (e.g. leaves of plants) rather than the storage organs such as tubers (Stahl et al. 2009).
Livestock	No data collected by Cardno Lane Piper as part of this assessment. This data has been collected as part of an assessment being conducted by ToxConsult.
Fish	PFOS detected at levels ranging from 5,000 ng/g to 23,500 ng/g in fish from Lake Fiskville (Appendix C and Appendix E).
Rabbit	Rabbits were collected (10 samples, average of 224 ng/g, maximum of 600 ng/g) in the vicinity of dams in the training area (Appendix C and Appendix F).
	luorinated compounds, PFOS = pefluorooctane sulfonate, PFAS = pefluoroalkyl sulfonate, poctyl carboxylic acid and PFAA = Perfluoroalkyl carboxylic acid

PFOS impacts have been identified in surface soil away from training areas. The most likely cause of the impacts identified is either spray drift and/or wind-blown foam. An assessment was made during various site visits and included discussions with relevant CFA personnel at the PAD. Independent observations of training and operation of the aerator on Dam 1 have also been made. Spray from the "Fog Spray" used in training on the FL PAD is considered a potential source of spray-drift away from the PAD.

Wind-blown foams (aerated clumps) are considered highly visible and it would be clear if they were leaving the training area from the FL Pad or the aerator on Dam 1. It is considered most unlikely that 'clumps' of aerated foam contribute to contamination of surfaces including soil, hardstand or roof areas away from the PAD.

Table 3-3 below provides a summary of an assessment of the potential for fallout of foam and spray-drift from the PAD. This is also considered in the sensitivity analysis in Section 7.

At Boundary of Area	Wind-blown Foam 'Clumps'	Spray Drift
FL PAD	Possible on windy days	Possible
Training Area	Unlikely. Falls to ground quickly and gets caught in grass.	Possible on windy day.
Site	Highly unlikely.	Possible on a windy day.

### Table 3-3: Potential for Fall-out of Airbourne PFCs from the FL PAD.

### 3.2 Potential Exposure Pathways

A range of exposure pathways are assessed in this HHRA for people from the Fiskville Community. This includes direct exposure pathways (e.g. incidental consumption of water during recreational activities) and indirect or secondary exposure pathways<sup>17</sup> (e.g. consumption of fish caught from the lake). Incidental ingestion is considered to be the dominant pathway by which the CoPC enter the body. Dermal absorption is not considered relevant for PFCs as they are poorly absorbed through the skin (ATSDR 2009).

<sup>&</sup>lt;sup>17</sup> PFC have been shown to bioaccumulate and are considered highly persistent in the environment (ATSDR 2009, RIVM 2010) hence consideration of secondary exposure pathways is important. Bioaccumulation is a result of the uptake of a chemical from water and/or food by a species which is greater than the ability of these species to remove that chemical from the body (e.g. metabolism, elimination processes etc.).



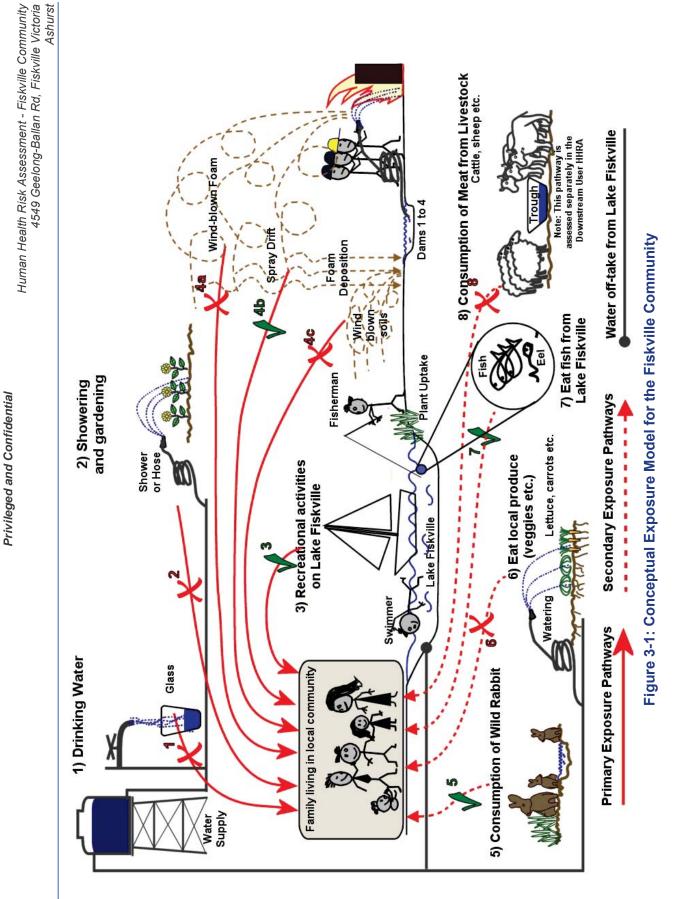
A list of the potential exposure pathways and their viability is discussed below in Table 3-4 and shown in Figure 3-1. Note that a viable pathway does not necessarily imply that the pathway is complete.



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	Potential huma	Potential human exposure pathways	Likel	Likelihood		Viable
#	Media	Pathway	Historic	Current	Comment on Viability of this pathway	Pathway?
Prim	Primary Exposure Pathways	thways				
~	Drinking Water from Lake Fiskville.	Potable water use.	Unlikely	Unlikely	Potable water is supplied by Central Highlands Water. Historical accounts during an internal workshop between CFA and Cardno Lane Piper indicate that water from Lake Fiskville was not used for drinking.	N
N	Incidental Ingestion of	Showering and/or watering the garden.	Unlikely	Unlikely	Lake Fiskville has not been used as a source of water for people residing in houses at CFA Fiskville Training College. Potable town water is currently available for this purpose. Stored rainwater was historically used for watering.	No
с	Water from Lake Fiskville.	Recreational activities on Lake Fiskville (e.g. swimming).	Likely	Unlikely	Recreational activities are currently not permitted on Lake Fiskville. The lake is large and deep enough to have possibly supported a range of recreational activities in the past.	Yes (historic exposures)
4a	Wind-blown foam clumps		Possible	Unlikely	Exposure to wind-blown foam clumps is possible in the training area only. The training area at Fiskville is a restricted area with access provided for authorised personnel only. People from the Fiskville Community may be escorted in the training area. Workplace management plans are in place to manage exposures to chemicals.	N
4b	Spray drift.	Incidental ingestion of and dermal exposure to.	Possible	Unlikely	Members of the Fiskville Community may have been exposed to spray drift from training exercises on the FL PAD. Spray drift is unlikely to contain PFOS and PFOA since CFA ceased using recirculated Dam 2 water in training in June 2012. Town mains water is now used. PFCs are not considered volatile (ATSDR 2009).	Yes (historic exposures)
4c	Contaminated Soils		Possible	Possible	Wind-blown foam chemicals may be deposited on soil in downwind areas around the PAD. PFC levels in soil are very low and well below screening criteria therefore this pathway is not considered further.	No
Sec	Secondary Exposure Pathways	Pathways				
Ŋ	Consumption of wild rabbit.	Consumption of wild rabbit meat hunted from the Site.	Likely	No	Wild rabbits are believed to have been hunted and consumed in the past. Rabbits have access to water and grass on the site which is potentially contaminated with CoPC. Hunting on site is not currently permitted.	Yes (historic exposures)
9	Consumption of Garden Produce.	Consumption of local produce grown for human consumption.	Unlikely	Unlikely	There is no history of water from Lake Fiskville being used to irrigate crops or vegetables grown on-site. PFC levels in grass (maximum of 10 ng/g, Table 3-2) indicate levels in garden produce would be negligible compared to fish and meat consumption pathways.	No
2	Consumption of aquatic life.	Consumption of fish and/or yabbies from Lake Fiskville.	Likely	No	Lake Fiskville currently has a population of edible fish species including Redfin Perch and yabbies. Eels have also been known to populate Lake Fiskville. Fishing is not currently permitted in Lake Fiskville.	Yes (historic exposures)
ω	Consumption of meat from livestock.	Consumption of meat from livestock (sheep) that graze on-site.	Unlikely	No (assessed separately)	Livestock have occasionally been held in one paddock separate from on-site training activities and Lake Fiskville. This exposure pathway is not complete for the Fiskville Community. It has been assessed separately as part of the Downstream HHRA.	No (Assessed separately)
		<b>Cardno</b> LanePiper			212163.18Report01.7 Page 15	





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### 3.3 Exposure Scenarios

Scenarios in this HHRA are based on whether exposure pathways are complete or active between the source of the CoPC and the receptor/person (as identified in Section 3.2) and on whether the CoPC is present in the exposure medium (e.g. water, food etc.) at sufficient concentrations.

The screening process, described in Appendix C, is applicable for screening CoPC as a result of exposure via primary exposure pathways. However it is not detailed enough in this instance to identify whether a viable secondary exposure pathway is complete because the adopted screening criteria do not take in to account the bioaccumulation potential of PFC. Therefore, the secondary exposure pathways considered viable (consumption of rabbit meat and fish) are also considered complete/active.

The following five scenarios outline the complete pathways and people (including adults and children) assessed in this HHRA:

- Scenario S1 (S1): People who have entered Lake Fiskville on a single or occasional basis. Please note this scenario does not include people involved in regular recreational activities on Lake Fiskville (Exposure Pathway 3).
- Scenario S2 (S2): People who were previously involved in recreational activities in Lake Fiskville including swimming (also Exposure Pathway 3). Recreational activities are currently not permitted in Lake Fiskville.
- Scenario S3 (S3): People who are exposed to spray drift (blown by the wind) from the PAD Areas (Exposure Pathway 4b).
- Scenario S4 (S4): People who consume meat from wild rabbits which drink water or eat grass that may contain PFC (Exposure Pathway 5).
- **Scenario S5 (S5):** People from the Fiskville Community who consume fish caught recreationally from Lake Fiskville (Exposure Pathway 7).



## 4 EXPOSURE ASSESSMENT

The exposure assessment defines the extent of intake of the CoPC for each scenario considered (S1 to S5). Exposure is estimated by accounting for the behavioural patterns of people from the Fiskville Community in each of the scenarios. The first four scenarios (S1 to S4) are assessed in this report in a qualitative fashion. The amount of the exposure assumed for S1 to S4 is discussed in comparison to the fifth scenario (S5) and therefore calculations of the amount of chemical intake are not necessary. Scenario S5 (Fish Consumption) has been quantitatively assessed separately in a report by ToxConsult (2014) presented in Appendix G. A summary of the assumptions used by ToxConsult (2014) is provided below.

### 4.1 Exposure from the Fish Consumption Pathway

The information in this section is paraphrased from the executive summary of the ToxConsult report. Readers are advised to read the ToxConsult (2014) report to gain a comprehensive understanding on how the exposure assessment was conducted for the fish consumption pathway.

Significant uncertainties in the data precluded assessing health risk from eating fish using a traditional tolerable daily intake (TDI) approach. Because the toxicological effects of PFOS are directly related to blood serum concentrations, persons who had eaten fish from the lake in the past, as well as the general Fiskville Community, were invited by CFA to participate in a health surveillance program as an extension of the health surveillance program already in place for CFA personnel. Participants also agreed to make their de-identified results available, via the CFA medical officer, to the consulting toxicologist and hence to the CFA via Cardno Lane Piper in the form of a statistical analysis for this report. Participants included people who may have had "*historical exposure to fire-fighting foams that contained PFOS*" (ToxConsult 2014). It is understood by Cardno Lane Piper that participants included people who worked in the training area and have a high potential for past exposures to PFCs. This would be PAD operators and PAD instructors who were identified in the IFI report (Joy 2012) as having 'medium' and 'high' relative risks from exposure to chemicals at the Site.

Serum PFC measurements were undertaken by a commercial laboratory that included appropriate quality controls and the data are considered reliable for assessment of potential health risk (ToxConsult 2014).

Twelve of the 22 participants in the 'fish consumption' study indicated that they had eaten fish or eel from the Lake in the past<sup>18</sup> (ToxConsult 2014). None of the persons tested had changes in blood clinical chemistry parameters that could be attributed to PFOS. While recognising the limitations of the study, statistical analysis of the data shows no association between blood parameters and serum PFOS levels. Nevertheless there were a number of individuals (fish eaters and non-fish eaters) that had clinical blood parameter measurements outside the population reference range. These occurrences were attributed to life style factors (e.g. alcohol consumption), body mass index, existing disease, and/or medication (including non-compliance).

Of the 10 PFC compounds tested for in human serum only two were present at measurable concentrations - PFOS and PFOA. All PFOA measurements were approximately an order of

<sup>&</sup>lt;sup>18</sup> No additional information has been provided to Cardno Lane Piper with respect to when and the regularity with which fish was eaten by these individuals except that Cardno Lane Piper understands that fish from Lake Fiskville had been eaten by some people from the Fiskville Community until recently.



magnitude less than the expected background concentrations for this compound. This indicates fish consumption has not contributed to human PFOA serum concentrations and does not need to be considered further (ToxConsult 2014).

Overall, it was concluded by ToxConsult (2014) that "existing serum PFOS concentrations or past theoretical concentrations are unlikely to give rise to adverse health effects".

### 4.2 Exposure from Other Pathways

The amount of exposure assumed for each scenario considered in this HHRA has been assigned a ranking based on a qualitative assessment of relative exposures, as shown in Table 4-1. Note that the <u>exposure ranking does not equate to a risk level</u> (risk characterisation is discussed later in Section 6). For comparative purposes the exposure amount via the fish consumption pathway is considered the highest of all complete pathways. Overall and in the case of a long term member of the Fiskville Community, the potential exposure amount ranges from potentially 'very low' for Scenario S1 (casual entry in the lake) to a 'high' exposure in Scenario S5 (consumers of fish).

<b>Scenario</b> (Exposure Pathway <sup>1</sup> )	Description of Scenario	Exposure Assessment
S1 (Exposure Pathway #3)	People who have entered Lake Fiskville on a single or occasional basis.	<ul> <li>People may have entered Lake Fiskville on an irregular basis either:</li> <li>As a result of carrying out work duties or (maintenance activities); and</li> <li>As a member of the public going in to retrieve an item during recreational activities on-site (no longer permitted).</li> <li>This type of exposure is considered an acute/short term exposure as it occurs on an irregular basis. It may include accidental ingestion of water if a high degree of body immersion occurs. PFC are poorly absorbed dermally (through skin) and accidental ingestion is assumed to be very low.</li> <li>Summary: The exposure of people to PFC in water from Lake Fiskville under Scenario S1 is considered very low.</li> </ul>
S2 (Exposure Pathway #3)	People previously involved in recreational activities in Lake Fiskville including swimming.	<ul> <li>Swimming is considered, on the basis of anecdotal information presented in the IFI Report, to have occurred in the past in Lake Fiskville however this cannot be confirmed. Swimming is considered the exposure pathway with the highest exposure for people involved in recreational activities on Lake Fiskville.</li> <li>Swimming is assumed to have: <ul> <li>Occurred on a weekly basis in the warmer months (mid-December to Mid-March), i.e. as many as 12 times per year. It is most likely swimming was less frequent than this or did not occur at all.</li> <li>Resulted in accidental ingestion of water which is likely to be low (assume 25 mL per event)</li> </ul> </li> <li>Summary: Exposure to PFC in water due to recreational activities under Scenario S2 is considered to be low compared to fish consumption.</li> </ul>

#### Table 4-1: Qualitative Assessment of Exposures Scenarios S1 to S5



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Scenario (Exposure	Description of Scenario	Exposure Assessment
Pathway <sup>1</sup> )		
<b>S3</b> (Exposure Pathway #4b)	People exposed to spray drift.	Spray drift is generated from the use of Fog Spray on the FL PAD which may be blown away from the PAD in the wind. Prior to June 2012, Dam 1 & 2 and town water was the source of water for training. Therefore spray drift prior to June 2012, when Dam water use ceased, would have contained PFCs. Consequently, exposure to spray drift prior to 2012 is considered a potential pathway. Exposure to spray drift was considered in occupational exposures on the FL PAD to be less than 0.1mL per hour (Cardno Lane Piper 2014a, Attachment 1). People from the Fiskville Community would be exposed to considerably less due to their distance from the FL PAD, reduced exposure time and variable wind direction.
	Deeple where	<b>Summary:</b> Exposure to PFC in spray drift under Scenario 3 is considered low.
S4 (Exposure Pathway #5)	People whom consume meat from wild rabbit caught on-site.	The number of individuals exposed to rabbit is considered very limited. Anecdotal information suggests at least two 2 individuals engaged in such hunting on-site. A number of PFCs were detected in muscle of wild rabbit collected from the site (Appendix C). PFOS levels, ranging from 44 ng/g to 600 ng/g (10 samples), are higher than background levels in European game animals (0.87 to 1.5ng/g, EFSA 2012). Other PFCs (PFPeA, PFHxS, PFDS and 8:2FTS) were also present but at levels 2 orders of magnitude lower than PFOS. The level of PFCs in rabbit is considerably lower than in fish (up to 23,000 ng/g in redfin) from Lake Fiskville. Consumption of rabbit meat in Australia is considered low (0.1 kg/person/year) however the rabbit consumption rates for a subset of the population (e.g. hunters) are likely to be higher but still considered low compared to fish consumption. <b>Summary</b> : The exposure of two individuals and potentially their family members is considered <b>medium</b> as exposure is considered to be less than for people who consumed fish.
S5	People who consume fish	Exposure as per Section 4.1. Consumption of fish meat (10 kg/person/year) is considered to be higher than for rabbit meat (0.1 kg/person/year).
(Exposure Pathway #7)	caught on-site.	<b>Summary</b> : The exposure of a limited number of individuals and potentially their family members is considered <b>high</b> as PFC levels in fish from Lake Fiskville is considered very high. However consumption of this fish is anticipated to form only a portion of the total fish consumption that makes up their diet.
8:2FTS = 1H,1	H,2H,2H-perfluorodecar	
1. Exposure pathways are described in Section 3.2		



## 5 DOSE RESPONSE

### 5.1 Hazard Identification

A short summary of hazards associated with exposure to the CoPC identified are presented here. For further information please refer to the toxicological summaries in Appendix H. The acute and chronic summaries, unless otherwise stated, for PFCs are based on information from the following review:

• Stahl, T., Mattern, D. and Brunn, H. (2011). Toxicology of perfluorinated compounds. Environmental Sciences Europe, Volume 23, Page 38.

### 5.1.1 Summary of Acute Risks of Compounds of Potential Concern (CoPC)

PFCs are not considered acutely toxic (HPA 2009, ATSDR 2009, Stahl 2011). There were no guidelines identified for acute exposure to PFCs.

There is no data available for humans and limited data for animals following acute exposure to PFCs via the oral, inhalation or dermal pathway. The data that is available is based on two of the surrogates used to represent the PFC classes in this HHRA: PFOS and PFOA. According to the ATSDR (2009) "*Acute- and intermediate-duration oral studies in animals have described primarily effects on the liver, body weight, developmental effects, and effects on the immuno/lymphoreticular system*". The acute toxicity in animals of these two surrogates is considered modest (Stahl 2011) as indicated by the acute toxicity ratings<sup>19</sup> shown in Table 5-1. These PFCs ranged from being practically non-toxic for PFOS following dermal exposure to moderate toxicity for PFOS following oral exposure (ATSDR 2009).

Douto	PFOS	PFOA
Route	Toxicity rating	Toxicity rating
Oral	Moderate	Slight to moderate
Dermal	Practically non-toxic	Slight
PFOS = perfluorina The toxicity rating i	ated octyl sulphonate, PFOA = perfl s based on acute effects as describ	uorinated octyl carboxylic acid ed by Stahl (2011).

### Table 5-1: Toxicity rating for PFOS and PFOA.

Irritation was not seen in rabbits<sup>20</sup> in toxicity studies following dermal exposure to PFOS (0.5 g), however it is considered mildly irritating to the eyes of rabbit following exposure of 0.1g (HPA 2009). Light skin irritation was observed following dermal application of PFOA to skin of rabbit (HPA 2009), however it is less in pronounced in rats (Stahl 2011). Gastrointestinal irritation has been observed in rats exposed to PFOA (higher than 680 mg/kg, HPA 2009). The lowest observed adverse effect level (LOAEL) following a single dose of PFOS was observed at 0.75 mg/kg for alterations in motor activity (ATSDR 2009).

### 5.1.2 Summary of Chronic Risks of CoPC

A summary of chronic risks is provided in Appendix H. Readers are directed there for more information. It is noted that for PFOS and PFOA, the critical effect in animal studies was

<sup>&</sup>lt;sup>20</sup> Albino New Zealand Rabbit



<sup>&</sup>lt;sup>19</sup> Classified according to the Hodge and Sterner scale

identified as being changes in liver weight or changes in biochemical parameters. A consistent correlation could not be shown between exposure to PFOS in the workplace and haematological or clinical chemistry parameters (HPA 2009). Epidemiological data for PFCs is limited.

### 5.2 Assessing Health Impacts from PFC Exposures

The selection of suitable toxicity guideline values is not performed in this report as the traditional TDI approach was precluded due to *"significant uncertainties regarding the extent and frequency that fish or eel were consumed, and lack of PFOS data in eels"* (ToxConsult 2014), see Appendix G. Therefore, the approach used by ToxConsult (2014) to assess health impacts from PFOS serum concentration (based on fish consumption) is:

- Compare measured PFOS levels in people from the Fiskville Community with:
  - "Background" serum concentrations; and
  - Human serum level concentrations considered to be without effects in humans; and
- Calculate a margin of exposure (MOE).

The qualitative assessment performed in this report is then based on the assessment used by ToxConsult (2014). A summary of the approach used by ToxConsult (2014) is taken from the executive summary of their report (italicised below). Readers are referred to ToxConsult (2014) for a detailed explanation of the approach adopted.

Many animal studies have shown toxicological effects of PFOS are directly related to serum concentrations. The potential health impact of serum PFOS concentrations measured in the health surveillance program has been assessed in a number of ways.

- Comparison with 'background' serum concentrations:
  - Review of many publications reporting PFOS serum concentration in general communities showed the majority of adults would be expected to a concentration of <0.1 mg/L.
- Comparison with a human serum level considered to be without effects in humans. Three different methods were used to establish a serum no observed effect level (serum NOEL) of 2 mg/L. These were:
  - Dose response analysis of a number of occupational epidemiology studies,
  - Derivation from monkey and rat serum NOELs using standard uncertainty factors, and
  - Conversion of the TDI set by the European Food Safety Authority into an equivalent steady state serum concentration.
- Calculation of margin of exposure (MOE) is a standard risk characterisation method widely used by Australian authorities. However instead of using experimental doses applied to animals and an uncertain estimated human intake in the calculation, the animal serum NOEL from toxicological studies and serum concentrations measured in program participants were used. While an acceptable MOE based on external dose is 100, that based on serum concentrations is 25. MOEs for four different endpoints (low birth weight, blood biomarkers, liver toxicity, and hepatic adenomas) were estimated.



## 6 **RISK CHARACTERISATION**

Risk characterisation describes the risk calculated or estimated for the selected exposure to CoPC by incorporating the exposure assessment (Section 3.3) and dose response (Section 5) sections. Risks were characterised separately for those based on:

- Long term exposures (Scenarios S2, S3b, S4 and S5, See Section 6.1); and
- Acute exposures (Scenarios S1 and S3a, See Section 6.2).

Note that risks related to occupational exposed workers at the Site are addressed as part of separate investigations (Cardno Lane Piper 2014a).

### 6.1 Quantitative Assessment of Risk (S2, S4 and S5)

Risks associated with consumption of fish (Scenario 5) were characterised by ToxConsult (2014). Risks from other scenarios with longer term exposures (Scenario S2: Past swimmers in the lake and Scenario S4: Consumers of rabbit meat) are characterised here in a qualitative fashion based on the findings of fish consumption. This is because the highest exposure to PFC is assumed to be associated with people from the Fiskville Community who ate fish from Lake Fiskville (Scenario S5) as discussed in Section 3.3.

This scenario has been assessed in a quantitative fashion by ToxConsult (2014). A summary of the risks characterised by ToxConsult (2014) for various assessment approaches is provided in Table 6-1 below. Only a summary of the risks conclusions provided by ToxConsult (2014) is presented here. The report prepared by ToxConsult (2014) is also available as a standalone document however it is provided as an appendix to this report (Appendix G) so that readers can access it readily.

Assessment Approach	Risk Conclusion Reached by ToxConsult (2014)
Comparison with 'background' serum concentrations	Four persons had serum PFOS concentrations above that identified as the higher end of the normal range expected from background (i.e. resulting from day to day living).
Comparison with a human serum level considered to be without effects in humans	All were below the serum NOEL, indicating low risk for adverse health effects.
Calculation of margin of exposure (MOE)	The Margin of Exposure (MOE) estimations calculated using current measured serum PFOS concentrations and serum NOELs identified in animal toxicity experiments also indicated very low risk for adverse health effects.
	When current serum concentrations were extrapolated back to theoretical levels that may have existed 5 or 10 years previously, and assuming no further fish consumption, both comparison with the human serum NOEL and the calculated MOEs indicate adverse health effects were unlikely to have arisen due to the hypothetical serum PFOS concentrations.
	Cardno note that ToxConsult (2014) considered susceptible populations when calculating the MOE. It is stated that <i>"in order that potential</i>

### Table 6-1: Risk Conclusions for Fish Consumption ToxConsult (2014).



Assessment Approach	Risk Conclusion Reached by ToxConsult (2014)
	reproductive risk (low birth weight) is addressed to the extent possible, females of reproductive age (≤ 45 years old) have been assessed as a separate group".
	Cardno note that ToxConsult (2014) considered susceptible populations when calculating the MOE. It is stated that <i>"in order that potential reproductive risk (low birth weight) is addressed to the extent possible, females of reproductive age (<math>\leq</math> 45 years old) have been assessed as a separate group".</i>
	ted in ToxConsult (2014), MOE = margin of exposure, NOEL = No observed Perfluorooctyl Sulphonic Acid.

Risks from the remaining scenarios (Scenario S2: past swimmers in the lake and Scenario S4: consumers of rabbit meat) are also considered negligible based on the findings from the quantitative assessment conducted for consumption of fish as discussed further in Table 6-1 above. This is because exposure to PFC from these other scenarios is considered to be less than PFC exposure from the consumption of fish (Section 3.3).

# 6.2 Qualitative Assessment of Risks for Other Exposure Scenarios (S1 and S3)

A qualitative assessment is performed here of the risks for people exposed to water from Lake Fiskville on a single or occasional basis (Scenario S1, Section 6.2.1) or exposed to spray drift (Scenario S3, Section 6.2.2).

### 6.2.1 People Involved in Recreational Activities on Lake Fiskville (Scenario S1).

People may have entered Lake Fiskville as part of recreational activities on a single occasion or on an occasional basis. This type of exposure is considered an acute exposure scenario due to the short duration. People may have been exposed to PFAS concentrations in Lake Fiskville approaching 47  $\mu$ g/L (Table 3-1).

Acute guideline values have not been set for PFOS and PFOA as acute toxicity is considered low via the oral and dermal routes of exposure. PFOS is considered practically non-toxic via the dermal route of exposure. Adverse health effects associated with acute exposure to PFCs (which potentially includes irritation) occur at concentrations that are much higher than levels seen in water at Lake Fiskville. Acute effects observed in rats include alterations in motor activity following consumption of PFOS at 0.75mg/kg. A 70kg adult would need to consume 2,000L of water in an acute exposure event with PFOS levels at 50µg/L to obtain a dose of 0.75mg/kg. This is clearly not achievable.

On this basis, acute risk associated with both incidental ingestion and dermal exposure to chemicals in water from Lake Fiskville is considered negligible.

### 6.2.2 Risks for People Exposed to Spray Drift (Scenario S3).

Acute and chronic risks are considered for people from the Fiskville Community assumed to be exposed to spray drift that originates from the FL PAD.

Spray drift is a result of training exercises on the FL PAD which involve spraying water on simulated training drills, most likely the Fog Spray exercise. Prior to June 2012 the water was



sourced from Dam 1 & 2 as well as potable town water. The use of Dam water meant that PFC, predominantly PFOS, would have been present in spray drift prior to June 2012. Dam 1 & 2 have PFOS levels of approximately 200  $\mu$ g/L. It is considered likely that spray drift has left the training area as is evident by PFOS impacts identified in surface soil (Soil impacts are discussed further in Appendix C).

Primary and secondary exposure pathways that could be impacted by spray drift have been assessed in this HHRA (e.g. consumption of rabbit meat). Spray drift and their potential impact on risk findings are most likely relevant to the following exposure pathways:

- Direct exposure to spray drift (primary exposure pathway): People exercising in areas outside of the training areas may potentially be directly exposed to spray drift, e.g. walking along the walking track to the South and East of the training area. This is an acute/short term exposure. PFC are practically non-toxic or exhibit only slight toxicity following dermal exposure and moderate toxicity via oral ingestion. PFC in water at 200µg/L is not considered toxic for an acute exposure. Spray drift may result in incidental exposure of up to 0.1mL of fine aerosols for CFA training personnel on the FL PAD (Cardno Lane Piper 2014a). For a person outside the training area the exposure is assumed to be considerably lower. No adverse effects would be expected as:
  - Localised adverse effects are not seen in the lung for PFC. Inhalation exposures in animal studies for PFOA did not result in localised effects in the lung (ATSDR 2009).
  - The first pass effect does not result in toxic metabolites of PFC<sup>21</sup>. PFCs are poorly metabolised (ATSDR 2009).
- Consumption of water (primary exposure pathway): Spray drift could make its way in to water tanks if fall-out occurred on nearby roof catchments. Potable water is available onsite therefore this pathway is not considered complete for people from the Fiskville Community; and
- Consumption of wild rabbit meat hunted from The Site (secondary exposure pathway): Rabbits were collected from the training area of the site may source PFC from grass that has taken up PFC from soil. The rabbits collected and assessed in this HHRA were collected in training areas with high water and soil concentrations (in the vicinity of Dams 1 and 2) therefore any impacts from spray drift would already have been taken in to account.

Chronic risks associated with potential exposure to spray drift are considered negligible.

<sup>&</sup>lt;sup>21</sup> The first pass effect results in a reduction of contaminant that is circulated throughout the body. The reduction is a result of metabolic processes in the liver that attempt to detoxify contaminants.



# 7 ASSUMPTIONS, UNCERTAINTIES AND DATA GAPS

# 7.1 Uncertainty Analysis

Uncertainty in the findings of any risk assessment is introduced due to limitations in data available and the range of assumptions made where site-specific data is not available. One method to account for uncertainty is to estimate risks using conservative assumptions. Although this HHRA is performed in a qualitative fashion there are assumptions made regarding the amount of exposure for people in each scenario. A summary of the uncertainties associated with these assumptions is provided below in Table 7-1. However, these uncertainties are unlikely to affect risk conclusions made in this HHRA as risk characterisation is based on measured PFOS blood levels in people from the Fiskville Community.

Assumption	Scenarios	Resultant risk	Comment
People from the Fiskville Community no longer engage in recreational activities on Lake Fiskville. This includes swimming, hunting and fishing	S1, S2, S4 and S5.	Reduced to nil.	Management decisions have resulted in a ban on recreational activities on Lake Fiskville and management authorisation is required prior to hunting on-site. It is envisaged that the bans will be in place at a minimum until contamination of Lake Fiskville is remediated.
Wind-blown foams (aerated clumps) do not leave the training area.	No Scenario (Exposure Pathway 4a )	Unlikely to pose a risk (foam from Dam 1 aerator) or exposure should not be permitted to occur (training exercise foam)	In the event that this assumption is not correct, i.e. wind-blown foam clumps do leave the training area, then consideration of wind-blown foams is discussed in 2 parts; foams from training exercises and foam from an aerator on Dam 1. <b>Dam 1 Aerator</b> . The aerator operates on a regular basis and generates clumps of wind- blown aerated foam. Based on the typical PFOS concentration range <sup>1</sup> in Dam 1 water (190 µg/L to 240 µg/L) then it is considered unlikely that exposure to this foam product would pose a risk to people exposed. <b>Training Exercise foam:</b> Training exercises with foam occur intermittently. Wind-blown foams from the FL PAD are likely to contain high levels of PFCs and a range of constituents <sup>2</sup> . Exposure of people from the Fiskville Community to this foam should not be permitted as they may not have training in the management of their use or be using appropriate personal protective gear.
PFC concentrations are higher than actually measured in various media (e.g. water, sediment, rabbit	S1, S2, S4 and S5.	No change	Concentrations of PFC in various media have not been used as a basis of risk characterisation in this HHRA. Instead, risk characterisation is based on measured PFOS blood serum concentrations. Risk conclusions are unlikely to change.

### Table 7-1: Uncertainty related to Exposure Assumptions.



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Assumption	Scenarios	Resultant risk	Comment
meat and fish muscle).			
Exposure pathway for the consumption of local produce irrigated with water from the lake is incomplete	<b>No Scenario</b> (Exposure Pathway 6)	No change	To exceed the TDI of 0.3 $\mu$ g/kg/day (Appendix H) a 70 kg person would need to consume 21 $\mu$ g of PFOS. Assuming that an average person eats approximately 280 g of vegetable products per day which is all irrigated with water from the lake then the vegetables would need PFOS levels at approximately 75 $\mu$ g/kg. Grass on a property adjacent to FTC has PFOS levels of 10 $\mu$ g/kg in grass from the paddock and 36 $\mu$ g/kg in grass from areas inundated with creek water near the lake overflow (PFOS levels approaching 20 $\mu$ g/L). Also, as shown in Section the relative intake from this pathway is considered small (1.4%) compared to the fish consumption pathway (97%) for which health implications arising from PFOS measured in blood serum are not expected.
People are exposed to PFC from multiple pathways.	<b>No Scenario</b> (Multiple Exposure Pathways)	No change	Assessment of multiple exposure pathways would not change the outcome of risks in this assessment as exposure from the consumption of fish is considered much higher than all other routes of exposure considered in this HHRA.

1. It is noted that the range of PFC concentrations in Dams 1 to 4 for PFOS (190  $\mu$ g/L to 240  $\mu$ g/L), PFOA (5  $\mu$ g/L to 8  $\mu$ g/L) and 6:2FTS (65  $\mu$ g/L to 95  $\mu$ g/L) is relatively consistent between sampling events.

2. Foam products used in training until 2007 contained PFOS and PFOA. This foam product was replaced with the PFC constituent changed to 6:2FTS. Foam products used for fire-fighter training are formulated with a range of constituents that are non-toxic (e.g. xanthum gum, polysaccharides, water etc.) or have low toxicity (e.g. glycol solvents). However, in some instances, constituents are classified as harmful substances, such as skin sensitisers (e.g. biocides in PFC-free foam products used by other agencies). It is noted that some foam products used by CFA are formulated with PFC but not PFOS or PFOA.

# 7.2 Data Gap Analysis

Overall, although data gaps have been identified (see Table 7-2 below) the quality of the data from all media (e.g. surface water and sediments) is considered suitable for use in a qualitative risk assessment.

Environmental Media	Data Gaps	Comment on Data Gaps
Surface Water and Sediment	Minimal temporal information available.	This is unlikely to affect the risk assessment as sediment and surface water data were used only to identify CoPC. They are not used in quantitation of risk and exposure is low for direct exposure pathways compared to other pathways. No additional data is required for the CoPC

## Table 7-2: Summary of Data Gaps and Comment



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Environmental Media	Data Gaps	Comment on Data Gaps
		assessed in this HHRA.
Soil and dust	This data is from one sampling event at any location. There is no temporal information for soil as short term changes in soil contamination are unlikely (compared with water quality changes) No information has been collected for PFCs other than PFOA, PFOS and 6:2FTS. No information has been collected for dust and it is assumed that PFC levels in dust would be similar to or less than levels in soils outside.	A correlation between soil impacts and distance from the FL PAD has been prepared (see Appendix C). Also, direct exposure to soil is considered a negligible pathway. No additional data is required for this HHRA.
Rabbit	There is a lack of temporal information (snapshot only) and there were no rabbits collected away from training areas.	It is highly unlikely that additional data for rabbits off-site would affect outcomes of the risks assumed in this HHRA as risks are not considered unacceptable. No further data is required.
Fish	There is no temporal data regarding PFC levels in aquatic species collected from Dams and Lake Fiskville from the site.	PFC levels in fish are unlikely to change in a significant way that would affect the outcomes of this HHRA unless there is a change in PFC levels in the lake. It is unlikely that PFC levels in the lake will increase as foams containing PFC are no longer used in training.



# 8 CONCLUSIONS

The potential for human health risks from exposure to water in Lake Fiskville has been assessed in this HHRA for people from the Fiskville Community. Perfluorinated Compounds (PFCs) were identified as the groups of compounds that are of potential concern at the Site.

Five human health scenarios were considered in this HHRA to address potential exposures to PFCs from the following exposure pathways considered complete:

- Dermal exposure and accidental ingestion of water during recreational activities in Lake Fiskville were considered in two scenarios:
  - Wading or casual entry in to Lake Fiskville (Scenario S1); and
  - Past swimmers in the lake (Scenario S2);
- Exposure to spray drift from the FL PAD (Scenario S3);
- The consumption of meat from wild rabbit hunted on-site (Scenario S4); and
- The consumption of fish caught recreationally from Lake Fiskville (Scenario S5). This pathway was assessed quantitatively by Dr Roger Drew<sup>22</sup> in an independent assessment of risk (ToxConsult 2014).

Risks for the human health scenarios are considered:

- **Negligible for Scenario S5** (consumers of fish): This finding is based on measured blood serum concentrations of PFC in people from the Fiskville community who have consumed fish caught from Lake Fiskville. This conclusion is based on an assessment that:
  - Only a few individuals had PFOS concentrations at, or slightly above, the upper edge of the background range<sup>23</sup>.
  - None of the individuals examined had changes in their blood parameters characteristic of PFOS, or which correlated with their PFOS serum concentration<sup>24</sup>.
  - Toxicologist Dr Roger Drew and the CFA medical doctor both conclude that they do not expect there to be any health implications arising from the concentrations of PFOS measured in the serum of the persons investigated.
- Negligible for Scenarios Scenario S1 (casual entry in the lake), Scenario S2 (past swimmers in the lake), Scenario S3 (exposure to spray-drift near the FL PAD) and Scenario S4 (consumers of rabbit meat): This conclusion is based on an assessment that:
  - Exposure to PFCs via consumption of fish is considered to be greater than other scenarios and was itself found to be associated with a negligible risk;
  - Accidental ingestion of water during recreational activities (Scenario S1 and Scenario S2) was considered minimal and the concentration of PFCs are relatively low compared to those in fish which was the highest exposure and therefore risk; and
  - The consumption of rabbit meat (Scenario S4) was rare and the concentrations in meat were relatively low.

There are no recommended actions given the conclusions of this HHRA and CFA have already implemented relevant management initiatives as listed in Section 1.3.

<sup>&</sup>lt;sup>24</sup> Some persons had blood parameters outside the reference ranges but these were associated with existing health conditions, medication or admitted lifestyle factors.



 <sup>&</sup>lt;sup>22</sup> Dr Roger Drew, PhD, DABT, Toxicologist and Risk Assessor, is one of Australia's leading toxicologists and has over 40 years of experience in his field of expertise.
 <sup>23</sup> These results are higher than what is expected for the majority (95%) of the general population. Nevertheless

<sup>&</sup>lt;sup>23</sup> These results are higher than what is expected for the majority (95%) of the general population. Nevertheless they were still markedly less than serum concentrations in factory workers making PFOS, and for whom there are no PFOS associated changes in blood parameters or demonstrable illness.

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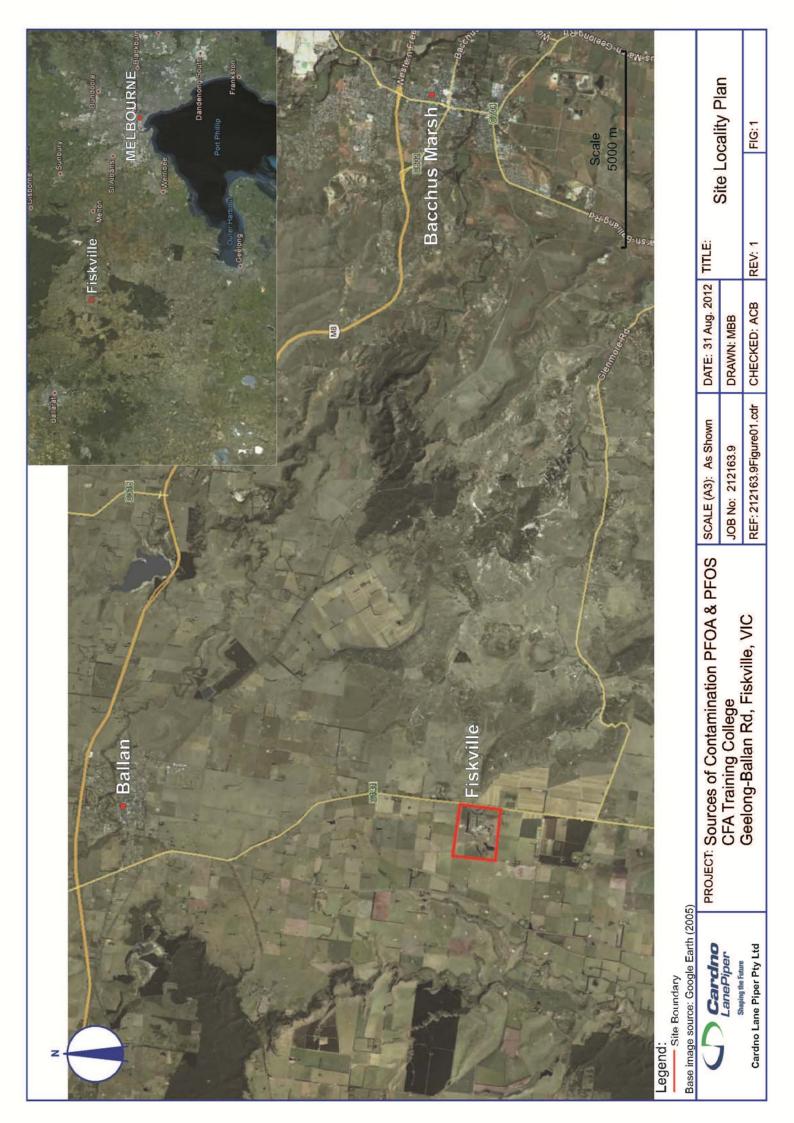
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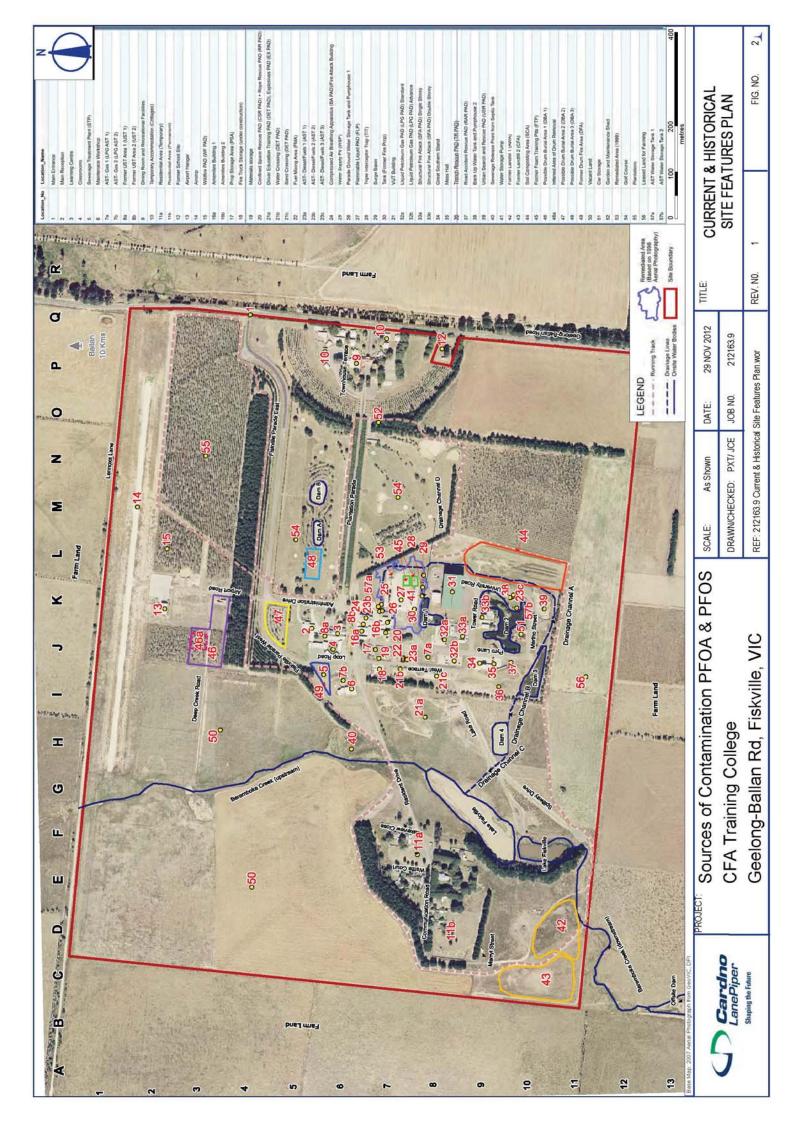


# Appendix A <sup>2 Pages</sup>

**Figures** Figure A1: Site location Figure A2: Site Features Plan







# Appendix B <sup>4 Pages</sup>

# **Summary Description of Surface Water Bodies**



# APPENDIX B - SUMMARISED SITE DESCRIPTION OF RELEVANT SURFACE WATER BODIES ON-SITE.

# Fiskville Training College, Geelong-Ballan Rd, Vic

The Site is relatively flat in the central and eastern portions, with the exception of western area (in which the land slopes down towards the Beremboke Creek and Lake Fiskville). The topography of the site is shown in Figure 1. The Beremboke Creek runs in a north to south direction across the western part of the site. The creek enters the site to the west of the airfield runway and then continues its course through the artificial Lake Fiskville before exiting the site following a southerly flow direction in to a tributary of the Beremboke Creek.

The CFA has installed a catchment and treatment system that includes a Surge Basin (or settling pond), triple interceptor trap (TIT) and various surface water bodies (Dams 1 to 4) to capture and treat water for re-use in training exercises. Water used on the flammable liquids PAD (FL PAD), the largest and most regularly used PAD at Fiskville, is directed to a Surge Basin and TIT to remove solid materials and excess liquid hydrocarbon fuel before release to Dam 1. Dam 1 is connected to Dam 2 via a damaged 300 mm pipe<sup>1</sup>. Dams 1 and 2 also collect an amount of surface water from the surrounding area<sup>2</sup>. Water then flows to Dam 3, Dam 4, and Lake Fiskville via open drain channels before release.

Water from this treatment system enters Lake Fiskville. The layout of the treatment facilities including surface water bodies at CFA Fiskville Training College is shown below in Figure 1. Images of Lake Fiskville and other on-site surface water bodies are shown below in Figure 2. A brief description of the surface water bodies at CFA Fiskville Training College is provided in Table 1.

<sup>&</sup>lt;sup>2</sup> Dam 1 and Dam 2 are connected hydraulically and have a limited catchment area. The land slopes away from these dams to the West, East and South.



<sup>&</sup>lt;sup>1</sup> It is believed the pipe connecting Dam 1 to Dam 2 was crushed during construction of a road. Water continues to flow as the crushed pipe is buried within a porous gravel layer.



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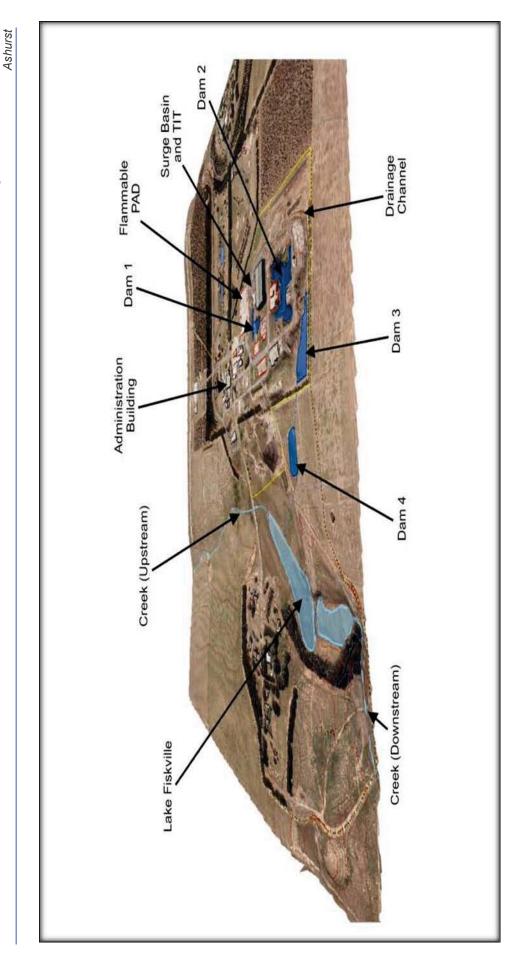


Figure 1: Main features of Fiskville Training College



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# Table 1: Description of Relevant Surface Water bodies including Lake Fiskville and the Beremboke Creek.

Area	Description
Dam 1	Dam 1 is located immediately south of the PAD. The approximate surface area is 1,500 $m^2$ and the average depth is 1.0 m (approximate volume is 1,400 $m^3$ ).
Dam 2	Dam2 is located south of Dam 1. The approximate surface area is 5,800 $m^2$ and the average depth is 1.0 m (approximate volume is 5,700 $m^3$ ).
Drainage Channel	The Drainage Channel is located on the north and eastern side of the drill operations area down from Dam 3. The Drainage Channel is approximately 530 m in length. For the purpose of this Assessment, the extent of the Drainage Channel was considered only up to the inflow into Dam 3.
Dam 3	The approximate surface area of Dam 3 of 2,900 $m^2$ and the average depth is 1.1 m (approximate volume is 3,290 $m^3$ ). Dam 3 receives surface excess spray and runoff from PAD area and is connected to Dam 2.
Dam 4	Dam 4 is located in the western portion of the site outside of the drill operations area near Lake Fiskville. The approximate surface area is $2,200 \text{ m}^2$ , the average depth is $1.4 \text{ m}$ (approximate volume is $3,190 \text{ m}^3$ ).
Lake Fiskville	Lake Fiskville is located on the south western portion of the site. The approximate surface area is 18,000 m <sup>2</sup> , with a depth ranging from 0.8 m on its northern portion to 4.7 m on its southern portion. It has an approximate volume of 45,900 m <sup>3</sup> . During dry periods, Lake Fiskville divides into two separate water bodies.
Beremboke Creek	The Creek runs in a north - south direction along the central to western portion of the site. Lake Fiskville receiving the inflow of the Creek on its northern end, which then continues to the southern end of the site (from the southern end of Lake Fiskville).



Appendix C 23 Pages

Description of Monitoring Events Analytical Results (from Cardno Lane Piper Monitoring Event) Screening for Compounds of Potential Concern Summary of Data Quality.



# HUMAN HEALTH RISK ASSESSMENT – FISKVILLE COMMUNITY

# 4549 GEELONG-BALLAN RD, FISKVILLE VICTORIA

# **APPENDIX C**

# SCREENING AND CHEMICALS OF POTENTIAL CONCERN (COPC).

# 1 SURFACE WATER AND SEDIMENT MONITORING ASSESSMENTS

Two (2) monitoring assessments (MA) have been conducted at the CFA Firefighting Training College, Fiskville Vic (the "Site") to collect water and sediment samples, these are described briefly below. Golder (2012) completed a surface water monitoring event in 2012 for surface water bodies at Fiskville (including Lake Fiskville). The second monitoring event was conducted by Cardno Lane Piper starting August 2012 (Cardno 2014a) which included multiple field events that were used to further characterise the extent of contamination of water and sediment in surface water bodies at CFA Fiskville Training College.

# 1.1 Surface Water Bodies Monitoring Assessment No 1, February 2012

Golder (2012) completed a surface water monitoring event in 2012. This was reported in their Preliminary Site Assessment (PSA) as part of the Investigation into Fiskville Inquiry (IFI), presented in Appendix C of the IFI report (Joy 2012).

A total of 10 sediment samples and 6 surface water samples were collected from Lake Fiskville and Dams 1 to 4. No samples were collected downstream from Lake Fiskville in this monitoring event (Golder 2012). Sample analysis was performed by ALS Environment Group (ALS), a NATA accredited laboratory. Samples were analysed for the following broad classes of compounds:

- Petroleum Hydrocarbons including Benzene, Toluene, Ethyl benzene and Xylenes (BTEX);
- Volatile Organic Compounds (VOC);
- Semi-volatile organic compounds (SVOC);
- Phenols;
- Perchlorates;
- Dioxins (PCDD and PCDF) in sediments only;
- Perfluorinated chemicals (PFC);
- Polychlorinated biphenyls (PCB);
- Pesticides;
- Metals (Arsenic, Cadmium, Chromium, Copper, Mercury, Nickel, Lead and Zinc); and
- Other inorganic compounds and nutrients.



# 1.2 Surface Water Bodies Monitoring Assessment No 2, August 2012, October 2012 and April 2013

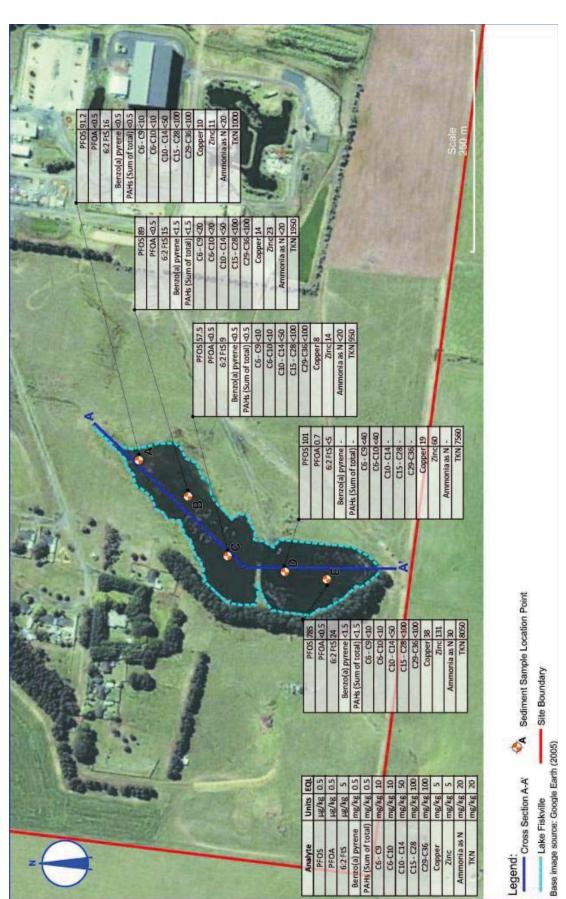
Cardno Lane Piper conducted a surface water monitoring event to further characterise the extent of contamination of water and sediment in surface water bodies at the Site. This assessment has been conducted over three field events (Field Events A to C). However, only Field Event A is relevant to Lake Fiskville. Information collected in this monitoring assessment which was combined with information from the previous monitoring assessment (Golder 2012, see Section 1.1). The combined dataset is used in this HHRA.

Field Event A occurred from the 1 to 21 August 2012 in multiple surface water bodies at the Site including Lake Fiskville (12 surface water samples at different depths from 5 locations, as shown in Figure 1-2). Off-site surface water samples was also collected downstream from Lake Fiskville at three different locations (1 surface water sample at each location) within 2 km of the Site's southern boundary.

The sampling locations for Lake Fiskville from Field Event A (August 2012) (including maximum concentrations identified at each location) and the corresponding sampling depths are shown in Figure 1-1 and Figure 1-2 respectively.

Only the analytical data from the August field event are shown in Figure 1-1 and Figure 1-2. The analytical data collected from all field events conducted by Cardno Lane Piper is reported in the *Surface Water and Sediment Contamination* report (2014a). The maximum concentration of PFC were higher from the October field event (e.g. PFOS =  $28.3 \mu g/L$ ).





C Cardno LanePiper

Figure 1-1: Lake Fiskville – Sediment Sampling Locations and Corresponding Concentrations for Select Compounds and PFCs

212163.18 Appendix C Monitoring Events and CoPC.docx



Human Health Risk Assessment - Fiskville Community 4549Geelong-Ballan Rd, Fiskville Victoria Ashurst

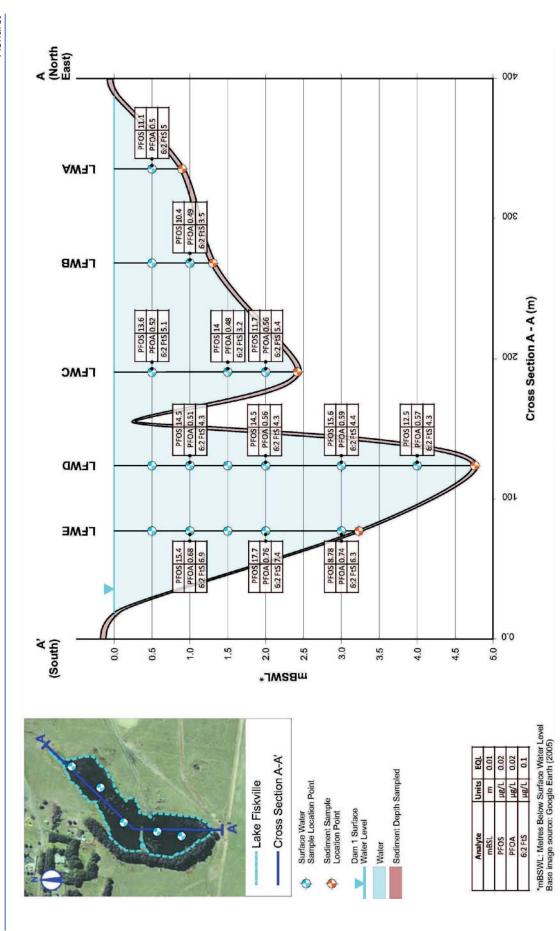


Figure 1-2: Lake Fiskville – Surface Water Sampling Locations and Depths, and Corresponding Concentrations for PFOS, PFOA and 6:2FTS.



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# **1.3 Summary of Compounds Identified in Surface Water and Sediments**

The following organic and inorganic compounds were identified in water and/or sediment in either or both of the Monitoring Events:

- Organic compounds:
  - Dioxins (Toxic equivalent at half the limit of detection)
  - BaP (Benzo(a)pyrene, toxic equivalent at half the limit of detection)
  - Perfluorinated Compounds
    - Perfluorooctane sulphonic acid (PFOS)
    - Perfluorooctanoic acid (PFOA)
    - 6:2 fluorotelomer sulphonic acid (6:2 FTS)
- Inorganic compounds:
  - Metals including arsenic, chromium (total), copper, lead, nickel and zinc
  - Ammonia (as nitrogen)
  - Fluoride
  - Nitrate
  - Nitrite
  - Sulphate

# **1.4** The Approach Required to Assess Perfluorinated Compounds (PFC)

There are potentially other PFC compounds present in the surface water and sediments other than PFOS, PFOA and 6:2 FtS. Therefore, to simplify the assessment of PFC identified in sediment and water, the following approach is adopted:

- Discuss sources of PFCs and identify other PFCs that may be present;
- Segregate PFCs identified into classes and identify a suitable surrogate<sup>1</sup> for use in the assessment; and
- Calculate the total concentration of PFCs in each class based on the surrogate.

Each PFC class is assessed in the HHRA rather than distinct PFC compounds. The assumption in this approach is that PFCs in the same class exhibit similar toxicity. This is considered a conservative approach which is necessary due to the large number of PFC potentially available and the limited data available for all these different PFC compounds.

### 1.4.1 Source of PFCs in Water and Sediment

PFCs are a key ingredient in Class B Aqueous Film Forming Foam (AFFF) products<sup>2</sup> used by the CFA in fighting fires that involve flammable liquids. Hence the source of the PFC in water and sediment at the Site is attributed to the use of foam products in CFA's hot fire training drills. Organisations other than the CFA also conduct hot fire training exercises at Fiskville. These exercises may include the use of Class B foams that may be different from those used by CFA, which may or may not be PFC free and may contain PFCs other than PFOS, PFOA or 6:2FtS. Hence, multiple PFCs are potentially present as contaminants in water and/or sediments at Fiskville.

<sup>&</sup>lt;sup>2</sup> AFFF = Aqueous Film Forming Foam. PFCs identified in the 2 Monitoring Events include PFOS, PFOA and 6:2 FTS which are commonly found alcohol resistant AFFFs (AR-AFFF).



<sup>&</sup>lt;sup>1</sup> A surrogate PFC is used to represent toxicity of other PFCs in its class

Class B foam products containing PFOS and PFOA were used by CFA at Fiskville from the 1990's until approximately 2007. PFOS and PFOA<sup>3</sup> have since been replaced in the use of different foam products currently used by CFA and other fire-fighting agencies. The PFCs present in the current foam products used by CFA include chemically similar compounds such as fluorotelomers (6:2 FTS) or analogues<sup>4</sup> of PFOS and PFOA<sup>5</sup>. Some analogues of PFOS and PFOA identified include perfluorinatedhexyl sulphonate (PFHxS), perfluorinatedhexyl octanoate (PFHxA) and perfluorinatedheptyl sulphonate (PFHpS).

## 1.4.2 Segregation of PFCs into Classes

The presence of other PFCs was taken into account by separating PFCs into different classes. Additional analysis was performed to identify and determine the concentration of other PFCs that may be present in water downstream from the Site. Additional analysis for 16 PFCs was performed from a water and a sediment sample taken from Lake Fiskville.

Due to the large number of compounds potentially present, PFCs are divided into 3 distinct classes and assigned a representative surrogate. The surrogate PFC is chosen for each class based on available chemistry and toxicity information. Currently, information on toxicity is limited for most PFC except for only a handful of compounds, most notably PFOS and PFOA<sup>6</sup>. Only 3 classes are chosen for this HHRA due to this database limitation. Therefore the PFC classes and representative surrogates selected in this HHRA are:

- **PFAS**: Perfluorinated alkyl sulfonic acids assessed using PFOS as a surrogate<sup>7</sup>;
- **PFAA**: Perfluorinated alkyl carboxylic acids assessed using PFOA as a surrogate<sup>8</sup>; and
- **OPC**: Other perfluorinated compounds assessed using 6:2 FTS as a surrogate<sup>9</sup>.

## 1.4.3 Calculating Concentration of each PFC Class in Water and Sediment

The concentrations of each PFC class are calculated using the following steps for sediment and water:

- 1. A representative media sample is selected from Dam 2.
- 2. Additional analysis is conducted on this sample for of a range of PFCs<sup>10</sup> as well as PFOS, PFOA and 6:2 FTS.

<sup>&</sup>lt;sup>10</sup> The following compounds were tested in both monitoring events; PFAS: Perfluorobutanesulfonic acid (PFBS), erfluorohexanesulfonic acid (PFHxS), Perfluorodecanesulfonic acid (PFDS), PFAS: Perfluorohexanoic acid (PFHxA), Perfluoroheptanoic acid (PFHpA), ), Perfluorononanoic acid (PFNA), Perfluorodecanoic acid (PFDA), Perfluoroundecanoic acid (PFUA), Perfluorododecanoic acid (PFDA), Perfluorotetradecanoic acid (PFTA), Perfluoroctanesulfonamide (PFOA), Perfluorotetradecanoic acid (PFTA), OPC: Perfluoroctanesulfonamide (PFOSA) and 1H,1H,2H,2H-perfluorodecanesulfonic acid (8:2 FTS). Golder (2012) also analysed for N-ethyl-perfluoroctanesulfonamidoacetic



<sup>&</sup>lt;sup>3</sup> The use of PFOS and PFOA in various products has been the subject of voluntary replacement by the international worldwide manufacturer since 2000 (NICNAS 2007). NICNAS (2009) recommends *"that these substances be restricted to only essential uses for which no suitable and less hazardous alternatives were available*".

<sup>&</sup>lt;sup>4</sup> Analogous are structurally similar compounds with a change in the alkyl chain length of the compound, e.g. perfluorinated hexyl sulphonate (C6 fluorinated aliphatic chain) and perfluorinated octyl sulphonate (C8 fluorinated aliphatic chain).

<sup>&</sup>lt;sup>5</sup> Unfortunately the precise makeup of PFC used in foam products is not divulged by manufacturers, suppliers or product MSDS sheets.

<sup>&</sup>lt;sup>6</sup> Further information on PFCs is provided in Appendix H (Toxicity Summary) which includes a summary of the broad range of compounds that belong to this class of chemical as well physical properties of select PFCs.

<sup>&</sup>lt;sup>7</sup> The toxicological database for this compound is relatively complete. Other sulfonic acids are anticipated to have similar toxicity however toxicity is assumed to increase with length of the fluorinated alky chain present.

<sup>&</sup>lt;sup>8</sup> This was based on toxicity of PFOA for the same reason given for PFOS (see previous dot point). PFAS and PFAA are not assessed as one class as PFOS has a lower tolerable daily intake than PFOA.

<sup>&</sup>lt;sup>9</sup> Very little toxicological data is available for the remaining PFCs. The basis of selecting the fluorotelomer, 6:2FTS, as the surrogate for this class is because it was identified in water and sediment in both monitoring events and is believed to be the PFC formulated in the class B foam product currently used by CFA.

- 3. The concentration of the surrogates, PFOS, PFOA and 6:FTS, is assigned to the parameter Conc<sub>SurrRep</sub>.
- 4. All PFCs measured in a media sampled are sorted into their respective classes and their concentrations are summed (ΣConc<sub>Class</sub>)
- 5. The percentage of the surrogate compounds (%Surr) is calculated using Equation 2.1.
- 6. The concentration of the PFC (Conc<sub>PFC</sub>) is then calculated for each media type using Equation 2-2 with data from each monitoring event.

$$\% Surr = \frac{Conc_{Surr \operatorname{Re} p}}{\sum Conc_{Class}} \times 100$$
 Equation 2.1

$$Conc_{PFC} = Conc_{Surr} \times \frac{100\%}{\% Surr}$$
 Equation 2.2

### Where

*****		
%Surr	=	Percentage surrogate contributes to the PFC class (PFAS, PFAA or OPC).
Conc <sub>SurrRep</sub>	=	Concentration of the representative surrogate PFC
Conc <sub>CLASS</sub>	=	The sum of all PFC in a particular class
Conc <sub>PFC</sub>	=	Maximum concentration of the PFC class, i.e. PFAS, PFAA or OPC
Conc <sub>Surr</sub>	=	Maximum concentration of the surrogate PFC, i.e. PFOS, PFOA or 6:2 FTS
		measured in Lake Fiskville.

A summary of the maximum concentration calculated of each PFC class is shown in Table 2-3 along with the maximum measured concentration of the surrogate (PFOS, PFOA or 6;2FTS) measured in water or sediment and the percentage of the surrogate calculated. The calculations for percentage surrogate are tabulated for both water and sediments in Appendix D, Table D1 and Table D2 respectively.

# Table 1-1: Calculated Maximum Concentration of PFC Classes calculated for Surface Water and Sediment using data from Lake Fiskville

Compound	Acronym	Surface Water (µg/L)		Sediment (mg/kg)	
		Cardno Lane Piper (2014a)	Golder (2012)	Cardno Lane Piper (2014a)	Golder (2012)
PFAS <sup>1</sup>	Conc <sub>PFC</sub>	32	47	0.79	0.34
%PFOS <sup>2</sup>	%Surr	74	%	99%	
PFOS <sup>3</sup>	Conc <sub>Surr</sub>	28.3	35	0.785	0.342
PFAA <sup>1</sup>	Conc <sub>PFC</sub>	12	13	n/a	0.0068
%PFOA <sup>2</sup>	%Surr	11	%	41% <sup>4</sup>	
PFOA <sup>3</sup>	Conc <sub>Surr</sub>	1.27	1.46	0.0007	0.0028
OPC <sup>1</sup>	Conc <sub>PFC</sub>	27	32	0.024	0.028
%6:2 FTS <sup>2</sup>	%Surr	77%		100%5	

acid (NEtFOSAA) N-methyl-perfluorooctanesulfonamidoacetic acid (NMeFOSAA) in ME1 whereas Cardno (2013) analysed for N-ethyl-perfluorooctanesulfonamide (NEtFOSA), N-methyl-perfluorooctanesulfonamide (NMeFOSA), N-ethyl-perfluorooctanesulfonamidothanol (NEtFOSE), N-methyl-perfluorooctanesulfonamidoethanol (NMeFOSE), 1H,1H,2H,2H-perfluorohexanesulfonic acid (4:2 FTS)



Compound	Acronym	Surface Water (µg/L)		Sedimen	t (mg/kg)
6:2 FTS <sup>3</sup>	Conc <sub>Surr</sub>	20.7 24.6		0.024	0.028
1.       PFAS concent         2.       Percentage su         3.       Maximum mea         4.       Based on the 0	ration calculated us rrogate (%Surr), ca asured concentratio %PFOA from Dam	2 as calculated in Hum	cPFC) n 2.1. an Health Risk Assess	ment – CFA Training P ore no adjustment is	



# 2 SELECTION OF COMPOUNDS OF POTENTIAL CONCERN (COPC)

The HHRA process includes a step to identify Compounds of Potential Concern (CoPC). This step is undertaken to identify those chemicals that are most likely to contribute to overall risk. These compounds are then carried forward in the risk assessment process for further assessment. The selection of CoPC in this HHRA is performed as follows:

- identify compounds detected in water and sediment
- select suitable health based screening criteria for the compounds identified
- collate the maximum concentrations for these compounds
- compare maximum concentrations identified with the selected health based screening criteria
- CoPC are those compounds with maximum concentrations that exceed the selected health based screening criteria.

Maximum concentrations detected in surface waters and sediment data are chosen from the 2 monitoring events described earlier (Sections 1.1 and 1.2).

The Screening is performed in two parts:

- Screening for Primary Exposure Pathways in water and sediment:
- Secondary Exposure Pathways from consumption of meat products, etc.

QAQC and data gaps for data presented is also summarised below.

## 2.1 Screening for Primary Exposure Pathways

The screening process for primary exposure pathways is suitable for direct contact exposure such as ingestion of water and dermal contact. The exposure pathway is considered complete where PFCs are detected in water or sediment above relevant screening values.

## 2.2 Compounds of Potential Concern in Water

The maximum reported surface water concentrations of compounds detected in the water samples from Lake Fiskville and the Beremboke Creek (on-site sampling locations only) are provided in Table 2-1. Drinking water guidelines (DWG) from NHMRC (2011) are used as screening values, where available, to identify those compounds that require further assessment. DWG from NHMRC (2011) were not available for TPH fractions and PFC. Screening values for these compounds are selected from DWG derived by the WHO (2005) and USEPA (2011) respectively. The compounds with concentrations that exceed the screening values are bolded and those compounds selected as CoPC.

Compound	Screening	Courses	Lake Fiskville			
Compound	Value	Source	Мах	CoPC		
Nutrients and others (Inorganics)						
Ammonia as N	60,000 <sup>1</sup>	See table note 1	110	No		

#### Table 2-1: Screening of CoPC (using max concentration) in Water (µg/L).



#### Privileged and Confidential

		-	
250,000 <sup>2</sup>	NHMRC (2011)	24	No
1,500	NHMRC (2011)	0.2	No
50,000	NHMRC (2011)	0.87	No
50,000	NHMRC (2011)	0.03	No
180,000 <sup>2</sup>	NHMRC (2011)	31	No
500,000	NHMRC (2011)	21	No
	·		
7	NHMRC (2011)	1	No
2	NHMRC (2011)	0.1	No
50	NHMRC (2011)	5	No
2,000	NHMRC (2011)	30	No
10	NHMRC (2011)	4	No
20	NHMRC (2011)	9	No
3,000 <sup>2</sup>	NHMRC (2011)	97	No
	·		
0.2	USEPA (2011b)	47	Yes
0.4	USEPA (2011b)	13	Yes
0.2	USEPA (2011b)	32	Yes
600	USEPA (2011b)	114	No
90	WHO (2005)	240	No <sup>4</sup>
	1,500 50,000 50,000 180,000 <sup>2</sup> 500,000 7 2 2 50 2,000 10 20 3,000 <sup>2</sup> 0.2 0.2 0.4 0.2 0.4 0.2 600	1,500       NHMRC (2011)         50,000       NHMRC (2011)         50,000       NHMRC (2011)         180,000 <sup>2</sup> NHMRC (2011)         500,000       NHMRC (2011)         500,000       NHMRC (2011)         7       NHMRC (2011)         2       NHMRC (2011)         50       NHMRC (2011)         2       NHMRC (2011)         2,000       NHMRC (2011)         2,000       NHMRC (2011)         10       NHMRC (2011)         20       NHMRC (2011)         3,000 <sup>2</sup> NHMRC (2011)         0.2       USEPA (2011b)         0.4       USEPA (2011b)         0.2       USEPA (2011b)         0.2       USEPA (2011b)	1,500NHMRC (2011)0.250,000NHMRC (2011)0.8750,000NHMRC (2011)0.03180,0002NHMRC (2011)31500,000NHMRC (2011)217NHMRC (2011)217NHMRC (2011)12NHMRC (2011)150NHMRC (2011)0.150NHMRC (2011)52,000NHMRC (2011)3010NHMRC (2011)3010NHMRC (2011)93,0002NHMRC (2011)970.2USEPA (2011b)470.3USEPA (2011b)130.2USEPA (2011b)32600USEPA (2011b)114

Notes: Bolded values have exceeded adopted screening criteria

 Based on the WHO (2006) assessment that "toxicological effects are observed at exposures above 200mg/kg of body weight". Therefore the screening value for ammonia is 6mg/L (i.e. screening value = 0.01 x 200mg/kg/day x 0.1 x 60Kg ÷ 2L/day where 0.01 is an uncertainty factor, 60kg is an average body weight, 2L is the assumed daily drinking water consumption and 0.1 assumes that only 10% intake is permissible from drinking water.

2. Selected screening values based on aesthetic guidelines are in red text.

3. The approach used to calculate the maximum concentrations for PFAS, PFAA, and OPC is presented in Section 1.4.

 Note that there were 2 detects for this fraction from 14 results. The average concentration from all results is 71µg/L assuming non-detects are present at half of the LOR (LOR = 100µg/L).

A suitable health based screening value for Ammonia was not available. The NHMRC (2011) and WHO (2008) drinking water guidelines only provide an aesthetic guideline value of 0.5mg/L. No health based guideline was derived as the presence of ammonia in water is not considered of immediate health relevance. Therefore a screening value was derived by Cardno Lane Piper for this compound (60 mg/L) in water for use as a screening value<sup>11</sup>.

The PFC classes have multiple detects in water from Lake Fiskville that exceed the selected screening criteria and are therefore selected as CoPC in water. There were 2 detects for the

<sup>&</sup>lt;sup>11</sup>. The health-based screening value used for ammonia is based on a statement in WHO (2006) "*toxicological effects are observed at exposures above 200mg/kg of body weight*"; thus this dose is considered a NOAEL. Using 200mg/kg as a point of departure and applying an uncertainty factor of 0.1 (intraspecies variability) a provisional drinking water guideline of 60mg/L was. This assumes 10% background from drinking water, 2L water consumed per day and a body weight of 60kg, consistent with NHMRC (2011) processes, i.e. screening value =0.1 x 200mg/kg/day x 0.1 x 60Kg ÷ 2L/day.



TPH >C<sub>16</sub> to C<sub>34</sub> fraction (from a total of 14 results) that exceeded the screening criteria (90 $\mu$ g/L). An average concentration from all results is 71 $\mu$ g/L (assuming non-detects are present at half of the LOR or 50 $\mu$ g/L)<sup>12</sup> therefore this fraction is not considered a CoPC.

# 2.3 Compounds of Potential Concern in Sediment

The maximum reported concentrations of compounds in sediment Lake Fiskville are provided in Table 2-2. Human health screening values for sediment are not widely available and it is common practice for soil screening values to be adopted. The screening values selected to identify those compounds that require further investigation are:

- Soil investigation levels (HIL-A) from NEPM (1999) where available.
- Regional screening levels (RSL, USEPA 2012) for "nutrients" and Bis(2-ethylhexyl) phthalate in soil.
- Soil screening values from USEPA for PFCs.

There are no compounds in sediment that have exceeded the adopted screening levels therefore no CoPC are identified in sediment. Analysis for PFC conducted in sediment samples from the Lake Fiskville are below selected screening value with a maximum PFAS concentration reported of 0.79mg/kg.

A SSV was not identified for ammonia (as nitrogen) in soils however it is not considered a CoPC. Background concentrations of ammonia in soils typically range from 1 to 5mg/kg (ATSDR 2004) and after application of fertilisers may increase up to 3000mg/kg with a drop to 890mg/kg within 5 days. The maximum concentrations of ammonia observed (30mg/L) is well below levels anticipated in soils within a farming community.

Compound	Screening Value	Source	Maximum Concentration	CoPC				
Nutrients and others (Inorganics)								
Ammonia	-	nil	30	No <sup>1</sup>				
Fluoride	3100	USEPA (2012)	180	No				
Nitrate (as N)	130000	USEPA (2012)	21.1	No				
Nitrite (as N)	7800	USEPA (2012)	4.8	No				
Metals (Inorganics)	Metals (Inorganics)							
Arsenic	100	NEPC (1999)	16	No				
Chromium (III+VI)	100	NEPC (1999)	98	No				
Copper	6000	NEPC (1999)	38	No				
Lead	300	NEPC (1999)	31	No				
Mercury	15	NEPC (1999)	0.1	No				
Nickel	600	NEPC (1999)	38	No				
Zinc	7000	NEPC (1999)	131	No				

## Table 2-2: Screening of CoPC (using max concentration) in Sediment (mg/kg).

<sup>&</sup>lt;sup>12</sup> Note that the limit of reporting (LOR <100  $\mu$ g/L) for the TPH >C<sub>16</sub> to C<sub>34</sub> fraction is greater than the selected screening criteria (<90 $\mu$ g/L)



Hydrocarbons (Organics)							
PFAS <sup>2,3</sup>	6	USEPA (2009)	0.79	No			
PFAA <sup>2,3</sup>	16	USEPA (2009)	0.0068	No			
OPC <sup>2, 3, 4</sup> 6 See PFAS <sup>3</sup> 0.024 No							
<ol> <li>Considered within background levels in a farming community</li> <li>The approach used to calculate the maximum concentrations for PFAS, PFAA, and OPC is presented in Section 1.5.</li> <li>The approach used to calculate the minimum and maximum concentrations for PFAS, PFAA, and OPC is presented in Section 1.5.</li> </ol>							

4. Screening value for PFAS used for OPC.

A total of 18 primary soil samples from the site were collected on-site and away from training areas. The data is considered acceptable based on the agreement achieved in the interlaboratory and intralaboratory samples. Refer to Appendix E of the main report for a more detailed discussion. A summary of QA/QC results is as follows:

- Intralaboratory Samples (2 samples): The intra-laboratory assessment showed acceptable reproducibility with %RPD less than 50%; and
- Interlaboratory Samples (1 sample): The %RPD for PFOS shows an acceptable correlation between the two laboratories.

A total of 97 primary soil samples from the paddock and floodplain of adjacent land were collected on the adjacent land. The intra- and interlaboratory assessment of QC showed %RPD of up to 50% and some exceedences. This is not considered ideal; however, it is considered suitable for a qualitative risk assessment. Refer to Cardno Lane Piper 2014b. A summary of QA/QC results is as follows:

- Intralaboratory Samples (4 samples): RPD ranged from 14% up to 64%; however, there
  were some higher exceedances due to the PFC being present below levels of reporting in
  some duplicate samples. The exceedances are most likely related to low analyte
  concentrations; and
- Interlaboratory Samples (5 samples): PFC were below levels of reporting in most of the secondary laboratory samples. PFC was only detected in one sample (QC14) with RPD ranging from 56.7% to 65.2%.

A single sampling event has occurred for soil data on-site and away from training areas. This data is considered a snapshot demonstrating the potential for soil impacts away from training areas and on adjacent land. This data is currently considered sufficient for this HHRA as a correlation can be shown with distance (see Figure 2-4 above) and the majority of samples taken downwind (southeast) of the FLPAD (the prevailing wind is considered to be from the northwest) also match the correlation. All data collected is below screening levels for soil data therefore direct contact exposures are considered negligible.

## 2.3.1 QAQC and Data Gaps Discussion - Primary Exposure Pathways

### **Surface Water and Sediment**

Sampling of surface water and sediment has occurred at various times of the year spanning from February 2012 (Golder 2012) to March 2013 (Cardno 2014a). The temporal variability of the data is limited (refer to Table 2-5 below) in that they provide a snapshot of conditions in various surface water bodies upstream, on and downstream, of the site. Water and sediment from Lake Fiskville and immediately downstream of the site have been sampled on two occasions.



Surface Water Body		Month	Sample		Deferrence
			Number <sup>1</sup>	Location	Reference
Beremboke Creek	Upstream	August 2012	1	А.	Cardno Lane Piper 2014a
		October 2012	2	A1, M.	
Lake Fiskville		February 2012	2	Inlet, Outlet.	Golder 2012
		August 2012	12 (5)	LFA to LFE.	
Beremboke Creek	Downstream	August 2012	4	B ,C, D, E.	Cardno Lane Piper 2014a
		June 2013	8	B ,C, D, E, S, T, U, V.	
Drainage Channel		No access obtained	nil	Not applicable	Not applicable
Eclipse Creek <sup>2</sup>		October 2012	2 (3)	F, G, I.	Cardno Lane
Moorabool River	Downstream	October 2012	3	J, K, L.	Piper 2014a
	Upstream	March 2013	4	O, P, Q, R.	

### Table 2-3: Summary of Sampling Events for Sediment and Water.

taken. When different, the number of sediment samples is indicated in brackets.

2. No water was present in sample location G at the time of sampling.

Data from both monitoring assessments (Golder 2012, Cardno 2014a) achieved completeness of greater than the target of 95%. An assessment of data quality, chain of custody and analytical reports for the monitoring assessment conducted by Cardno is provided in Cardno Lane Piper (2014a). In the PSA prepared by Golder (2012) it is stated that the quality of data collected during the water monitoring program is "*of acceptable quality upon which to base decisions for this assessment*". This was based on the laboratory QA/QC program achieving a completeness of 98.2% which is greater than the target of 95%. Non-conformances were discussed and appropriately justified.

The temporal nature of the data, i.e. it is considered a snapshot in time, is considered an uncertainty in this HHRA. Two field events have been conducted in Lake Fiskville. It is noted that creeks downstream of the site were flowing in August 2012 (Cardno's 1<sup>st</sup> field event) but it is not known if they were flowing during Golder (2012) monitoring event. The maximum PFOS concentration recorded in water and sediment from Lake Fiskville between both events are similar in magnitude although higher in the Golder (2012) monitoring event. Lack of temporal information for sediment and water is considered a data gap.

## Soil data

A total of 18 primary soil samples from the site were collected on-site and away from training areas. The data is considered acceptable based on the agreement achieved in the interlaboratory and intralaboratory samples. Refer to Appendix E of the main report for a more detailed discussion. A summary of QA/QC results is as follows:

- Intralaboratory Samples (2 samples): The intra-laboratory assessment showed acceptable reproducibility with %RPD less than 50%; and
- Interlaboratory Samples (1 sample): The %RPD for PFOS shows an acceptable correlation between the two laboratories.



A total of 97 primary soil samples from the paddock and floodplain of adjacent land were collected on the adjacent land. The intra- and interlaboratory assessment of QC showed %RPD of up to 50% and some exceedences. This is not considered ideal; however, it is considered suitable for a qualitative risk assessment. Refer to Cardno Lane Piper (2014b). A summary of QA/QC results is as follows:

- Intralaboratory Samples (4 samples): RPD ranged from 14% up to 64%; however, there
  were some higher exceedances due to the PFC being present below levels of reporting in
  some duplicate samples. The exceedances are most likely related to low analyte
  concentrations; and
- Interlaboratory Samples (5 samples): PFC were below levels of reporting in most of the secondary laboratory samples. PFC was only detected in one sample (QC14) with RPD ranging from 56.7% to 65.2%.

A single sampling event has occurred for soil data on-site and away from training areas. This data is considered a snapshot demonstrating the potential for soil impacts away from training areas and on adjacent land. This data is currently considered sufficient for this HHRA as a correlation can be shown with distance (see Figure 2-4 above) and the majority of samples taken downwind (southeast) of the FLPAD (the prevailing wind is considered to be from the northwest) also match the correlation. All data collected is below screening levels for soil data therefore direct contact exposures are considered negligible.

# 2.4 Screening For Secondary Exposure Pathways

The screening process described in Section 2.1 is applicable for screening CoPC as a result of exposure via primary or direct contact exposure pathways. However it is not sensitive enough to identify whether a viable secondary exposure pathway is complete for bioaccumulative compounds such as PFCs<sup>13</sup>. A screening process was also conducted for secondary exposure pathways (e.g. consumption of rabbit meat and fish) by identifying whether PFCs have been detected in various media (soils, fish muscle and rabbit muscle). The exposure pathway is considered complete where PFC are detected in these media.

## 2.4.1 Perfluorinated Compounds in soil (consumption of meats)

It was assumed that wind-blown foams and/or spray drift from the FL PAD and dam 1 could potentially impact on soils away from training areas. Discussions with CFA personnel (07/02/2014) from the FTC indicates that is highly unlikely that either leave the training area and highly unlikely that they leave the site. Cardno is of the view that it would be difficult to see if spray drift was leaving the site and is the most likely cause of impacts in soil detected away from the training area (on-site and off-site)

There is potential for PFC in soil to be taken up in to plants and consumed by grazing animals (rabbit, livestock etc.). The presence of PFC in soil would represent 2 possible pathways for PFC to enter the human food chain

- Through grazing animals which eat grass that have taken PFC up from soil; and
- Wind-blown soils could be blown on to a catchment area and washed ion to tanks used for drinking water.

<sup>&</sup>lt;sup>13</sup> PFC have been shown to bioaccumulate and are considered highly persistent in the environment (ATSDR 2009, RIVM 2010). Bioaccumulation is a result of the uptake of a compound from water and/or food by a species which is greater than the ability of these species to remove that compound from the body (e.g. metabolism, elimination processes etc.).



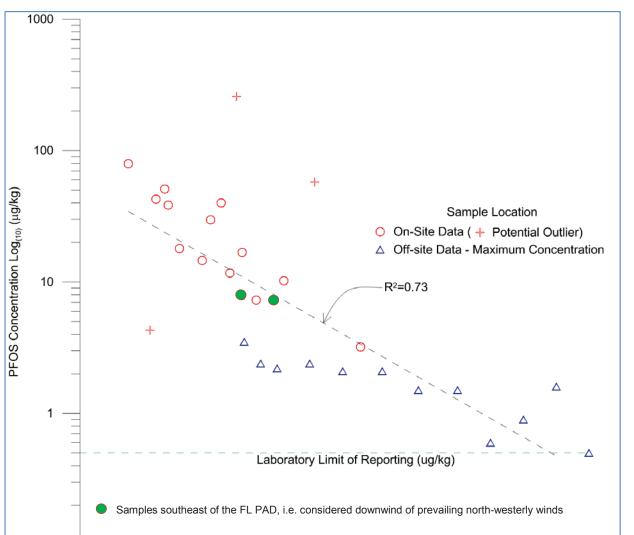
The impact of spray drift is indicated by PFOS levels detected in surface soil on the site away from training areas (on-site and off-site, see section 2.1.3). PFOS is used to demonstrate the trend as other PFC identified in soil (PFOA and 6:2FTS) were present at lower concentrations and often below limits of detection, i.e. PFOS contributes the majority of total PFC concentration detected in soil. PFOS concentrations in soil on-site and away from training areas (shown in Figure A6) ranged from 3.2 to 258  $\mu$ g/kg (Cardno Lane Piper 2014a)<sup>14</sup>. Note that units previously discussed for soil were in mg/kg.

Soil data has also been collected on adjacent land (Cardno lane Piper 2014b) which shares a boundary with the site. These soil results are shown in a Figure A7. Multiple samples were collected in various rows with increasing distance from the training area. The maximum PFOS value from each row has been selected and matched to their relative distance to the FL PAD. The on-site data and off-site data for soil are plotted against distance from the centre of the training area (FL PAD) as shown in Figure 2-4 (note that the concentration scale on the 'y axis' is logarithmic). PFOS soil concentrations are decreasing in a logarithmic fashion with distance from the FL PAD as shown by the line of best fit. PFOS concentrations in soil reduce by more than an order of magnitude between 100 and 600 m of the FL PAD and by a further order magnitude by 1500m away. This decreasing trend is represented by the line of best fit which is not influenced by outliers except that the goodness of fit when outliers are included (Coefficient of  $R^2$ =0.58) is lower than when the outliers are excluded (Coefficient of  $R^2$ =0.73). 3 soil sample results (marked by a red plus sign) are considered potential outliers. The results demonstrate that there is potential for impacts in soil away from the site.

<sup>&</sup>lt;sup>14</sup> This concentration range is well below the soil PFOS screening criterion of 6,000 μg/kg for direct exposure pathways (accidental ingestion and dermal exposures with soil) from USEPA (2012).



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## Figure 2-1: PFOS Concentration in Soil with Distance from the Flammable Liquid PAD.

Wind direction (indicated for each sample point shown in Figure 2-4) could not be visually correlated with PFOS concentration and distance (not shown). It is noted that there is a relatively uniform wind distribution pattern at the site as shown below in Table 2-5 however the prevailing wind tends to come from the northwest albeit only 20% of the time. Properties with water tanks closest to the site (from 650m away) are southeast of the site, i.e. predominantly downwind. Soil on the adjacent site are south of the FL PAD in the direction of northerly winds which are only registered 10% of the time. Irrespective of wind direction, on-site data to the southeast of the site and south of the site both fit the correlations shown in Figure 2-4. Hence, the correlation shown is considered robust enough to be used as a predictor of soil impacts away from training areas irrespective of direction.



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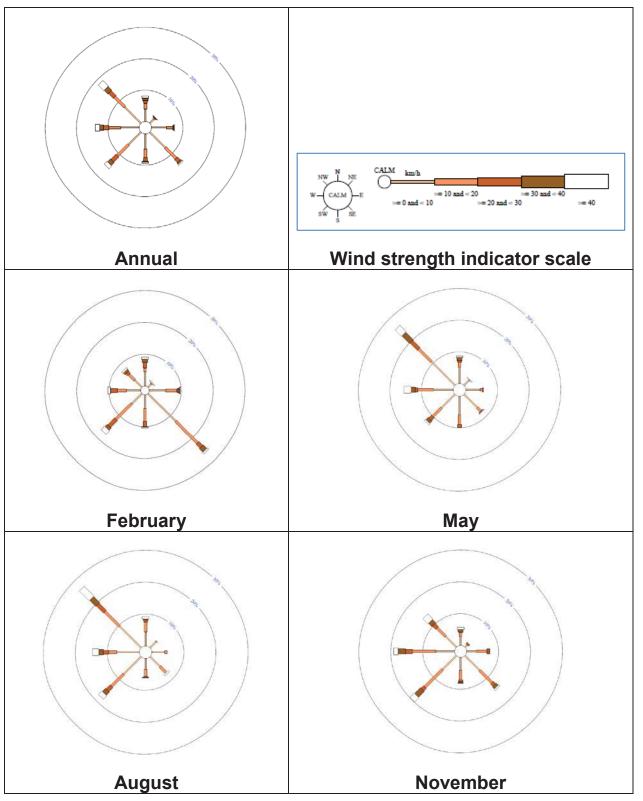


Figure 2-2: Rose of Wind direction versus wind speed<sup>15</sup> – Ballan (Fiskville).

<sup>&</sup>lt;sup>15</sup> Wind rose taken Bureau of Meteorology (Australian Government) site and was last accessed on 05 March 2014 at http://www.bom.gov.au/clim\_data/cdio/tables/pdf/windrose/IDCJCM0021.087005.3pm.pdf



It is noted that the level of PFOA and 6:2FTS was of a similar magnitude to FPOS in one of the three potential outliers identified (in the on-site data) which is different to other samples where total PFC concentration was contributed to mainly by PFOS. This outlier was approximately 480m away from the FL PAD with the maximum PFOS concentration detected in soil (away from the training area) of 258µg/kg. This indicates that the source of PFC for this outlier is potentially different to PFC impacts identified in soil (e.g. ad-hoc training activities conducted away from training areas).

These results indicate that:

- The surface soil contamination by PFOS has occurred away from training areas;
- The surface soil concentrations of PFOS are very low compared with relevant assessment criteria for direct contact exposures (6,000µg/kg);
- The concentrations in surface soil diminish rapidly away from the training areas.

Exposure pathways are potentially complete where exposure to soil (and plants grown in this soil) is considered possible (e.g. consumption of meat from grazing animals).

### 2.4.2 Perfluorinated Compounds in Fish

Sampling of aquatic biota in on-site surface water bodies has been conducted (Cardno 2014). Aquatic species collected from Lake Fiskville include; Redfin perch (Perca fluviatalis), freshwater shrimp, mosquite fish (Gambusia holbrooki) and pondweed. 5,400 to 22,300 ng/g of PFOS was detected in fish muscle (average = 9,139 ng/g in 21 samples) collected from Lake Fiskville: These are levels considerably higher than would be expected and is indicative of bioaccumulation of PFC in fish as a result of on-site activities.

Summary: Exposure to PFOS in fish caught from Lake Fiskville is considered a potentially complete exposure pathway.

### 2.4.3 Perfluorinated Compounds in Rabbit (on-site)

Rabbits were collected from the site. PFC were detected in muscle with PFOS levels ranging from 44 ng/g to 600 ng/g (10 samples) with an average of 224 ng/g (Cardno 2014). Perfluoropentanoic Acid (PFPeA) was also detected in muscle samples (Average of 1.3 ng/g with a range of 0.25 ng/g to 3.7 ng/g). These rabbits were collected in the vicinity of on-site dams with PFOS concentrations in water (approximately 200µg/L) that are an order of magnitude higher than PFOC concentrations in water in Lake Fiskville and dams immediately downstream of the site. Concentration of PFOS in water continues to drop by a couple of orders of magnitude in the waterway downstream of the site. This suggests that PFOS levels in rabbits away from the training area will be considerably lower than in rabbits caught on-site in the training area.

Summary: Exposure to PFOS in rabbit caught on-site is considered a potentially complete exposure pathway.

### 2.4.4 QAQC and Data Gaps Analysis for Secondary Exposure Pathways

#### Rabbit data

The data quality is considered acceptable primarily based on the agreement achieved in the interlaboratory samples. Refer to Appendix D of the main report for a more detailed discussion. A summary of QA/QC results is as follows:



- Intralaboratory Samples (2 samples): The intra-laboratory assessment showed acceptable reproducibility with only one sample exceeding an acceptable %RPD of 50%; (57.4% for PFPeA in sample RA6-1D for);
- *Interlaboratory Samples (3 samples)*: The %RPD for PFOS shows an acceptable correlation between the two laboratories (i.e. within 15%); and
- *Spiked Samples (2 samples)*: The first batch could have overestimated the concentration for some analytes with the average spiked concentrations reported at 136%, while the second batch may have underestimated the concentration for some analytes with the average spiked concentrations reported at 84%.

The rabbits (10) were sampled from the site in May 2013 (Appendix D). These rabbits were collected from a part of the site where the concentration and PFC in water and air are greatest (within the training area). Rabbits have not been collected from areas of the site away from training areas and off-site. The assumption was made that PFC levels would be considerably lower than rabbits collected off-site than those collected near the training area. However, this data has not been collected and is considered a data gap.

### Grass (adjacent land)

Grass was collected in October 2013. A total of 9 grass samples (with 2 detects) were collected in the paddock of the adjacent land and 6 samples (with 5 detects) from areas near surface water bodies and assumed to be inundated wih water during high rainfall events.

The maximum detect in samples from the paddock was 10  $\mu$ g/kg whereas it was 36  $\mu$ g/kg in samples from potentially inundated areas. There are no RPD exceedances for grass samples. Refer to Cardno Lane Piper (2014b) for more information. The data is considered suitable for use in a risk assessment.

The data is limited, based on a single event and is only considered indicative of potential PFC levels in leafy parts of vegetation.

## Aquatic Ecology Data

A total of 60 samples were analysed for PFCs and metals (38 from Lake Fiskville). Refer to Appendix F of the main report for a more detailed discussion. Overall it was concluded that data such as the %RPD, spike recovery and the frequency of QC samples conducted is considered to be sufficient and provides a reliable set of results. It is noted that the:

- %RPD between two laboratories (interlaboratory, National Measurement Institute NMI Sydney and Assure Quality, Wellington, NZ) is within recommended guidelines (i.e. < 50%) for PFOS (8.5 to 27%) and PFDA (9.5 to 42%). The %RPD ranged from 6.1 to 100% for 6:2 FtS. Note that %RPD was not calculated for samples which reported less than the LOR.; and
- The surrogate recovery is low for PFOS in data reported by the primary laboratory (NMI) however this does not impact on the reproducibility of the results from the two laboratories.

A number of laboratory surrogate recovery exceeded 400% which are mainly related to the first batch of samples analysed and believed to be related to laboratory handling procedures. When only redfin perch muscle data is considered the average surrogate recovery % were predominantly between 50% and 80% and considered acceptable. The surrogate recovery for PFOS for the primary laboratory (NMI) was consistently lower than the secondary laboratory (Assure Quality).



In summary, the fish results reported are considered to be accurate and can be relied upon for use in risk assessments. A large amount of data has been collected in fish and other aquatic species from surface water bodies on the site and from the Moorabool River. No aquatic species were collected from the creeks as aquatic life was not evident and is unlikely to support fish species that could be eaten. This is expected to some extent as the creek is ephemeral. It is noted however that investigations of aquatic life along the creeks were restricted to areas that could be accessed and that there is a possibility that during high water flow events that aquatic life from Lake Fiskville could be washed downstream and in to the creeks.



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Samula Matrix		Golder			Cardno			Cardno	
Laboratory reference		107033-2		EW	EM1209107-6		EM	EM 1208979-4	
Sample ID	Fe0	Fe06440 Water	-	D2WD	D2WD1.0/09082012	012	LFW E	LFW E2.0/06082012	012
Location					Dam 2		Lak	Lake Fiskville	
Units	Conc (µg/L)	% Total	% PFC	Conc (ng/L)	% Total	% PFC	Conc (µg/L)	% Total	% PFC
Total PFCs	463	100		504.16	100	T	40.1	100	-
Perfluoroalkylsulfonic acids	81.9	18%	%PFAS	326.4	65%	%PFAS	23.82	%69	%PFAS
Perfluorobutanesulfonic acid (PFBS)	9.9	2.14%	12%	9.58	2%	3%	1.62	4%	7%
Perfluorohexanesulfonic acid (PFHxS)	43	9.30%	53%	41.8	8%	13%	4.48	11%	19%
Perfluorooctanesulfonic acid (PFOS)	29	6.27%	35%	275	22%	84%	17.7	44%	74%
Perfluorodecanesulfonic acid (PFDS)	Not	Not quantifiable	Ð	0.02	%00.0	0.01%	0.02	0.05%	0.08%
Perfluoroalkylcarboxylic acids	82.3	18%	%PFAA	63.54	13%	%PFAA	69.9	17%	%PFAA
Perfluorohexanoic acid (PFHxA)	59	12.76%	72%	46.6	%6	73%	4.96	12%	74%
Perfluoroheptanoic acid (PFHpA)	13	2.81%	16%	2.48	0.5%	4%	0.66	2%	10%
Perfluorooctanoic acid (PFOA)	10	2.16%	12%	13.1	3%	21%	0.76	2%	11%
Perfluorononanoic acid (PFNA)	0.21	0.05%	0%	1.1	0.2%	2%	0.05	0.1%	1%
Perfluorodecanoic acid (PFDA)	0.065	0.01%	%0	0.02	0.004%	%0	0.02	0.05%	0.3%
Perfluoroundecanoic acid (PFUnA)	0.017	0.00%	0%	0.02	0.004%	%0	0.02	0.05%	0.3%
Perfluorododecanoic acid (PFDoA)	0.017	0.00%	%0	0.1	0.02%	%0	0.1	0.2%	1%
Perfluorotridecanoic acid (PFTrA)	Not in	Not in analytical suite	uite	0.02	%00.0	%0	0.02	0.05%	0.3%
Perfluorotetradecanoic acid (PFTeA)	Not in	Not in analytical suite	uite	0.1	0.02%	%0	0.1	0.2%	1%
Other PFCs	298	64%	%OPFC	114.22	23%	%OPFC	9.62	24%	%OPFC
Perfluorooctanesulfonamide (PFOSA)	Not	Not quantifiable	e	0.02	0.004%	%0.0	0.02	0.050%	0.2%
N-ethyl-perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	0.013	0.003%	0.004%	Not in a	Not in analytical suite	uite	Not in a	Not in analytical suite	uite
N-methyl-perfluorooctanesulfonamidoacetic acid (NMeFOSAA)	0.019	0.004%	0.006%	Not in a	Not in analytical suite	uite	Not in a	Not in analytical s	suite
N-ethyl-perfluorooctanesulfonamide (NEtFOSA)	Not in	Not in analytical suite	uite	0.1	0.02%	0.1%	0.1	0.25%	1%
N-methyl-perfiluorooctanesulfonamide (NMeFOSA)	Not in	Not in analytical suite	uite	0.1	0.02%	0.1%	0.1	0.25%	1%
N-ethyl-perfluorooctanesulfonamidothanol (NEtFOSE)	Not in	Not in analytical suite	uite	1	0.2%	%6.0	٢	2.5%	10%
N-methyl-perfluorooctanesulfonamidoethanol (NMeFOSE)	Not in	Not in analytical suite	uite	-	0.2%	0.9%	٦	2.5%	10%
1H,1H,2H,2H-perfluorohexanesulfonic acid (4:2 FTS)	Not	Not quantifiable	е	Not in a	Not in analytical suite	uite	Not in a	Not in analytical suite	uite
1H,1H,2H,2H,2H-perfluorooctanesulfonic acid (6:2 FTS)	290	63%	97%	112	22%	<mark>98%</mark>	7.4	18.44%	77%
1H,1H,2H,2H-perfluorodecanesulfonic acid (8:2 FTS)	8.3	1.8%	3%	Not in a	Not in analytical suite	uite	Not in a	Not in analytical suite	uite

# Table D1: PFC surrogate percentages for surface waters



Sample Matrix		Golder			Cardno			Cardno	
Laboratory reference		107033-1		EM	EM1209107-2	5	EM1	EM1208900-1	1
Sample ID	Fe	Fe06439 Soil	_	D2SF(	D2SF0.1/08082012	012	LFSE0	LFSE0.1/02082012	012
Location					Dam 2		Lak	Lake Fiskville	e
	Conc (µg/g)	% Total	% PFC	Conc (ng/L)	% Total	% PFC	Conc (µg/g)	% Total	% PFC
Total PFCs	223	100		2284	100		946	100	
Perfluoroalkylsulfonic acids	212	95%	%PFAS	1971.04	86%	%PFAS	793	84%	%PFAS
Perfluorobutanesulfonic acid (PFBS)	0.033	0.01%	0.02%	3.14	0.1%	0.2%	0.5	0.05%	0.06%
Perfluorohexanesulfonic acid (PFHxS)	0.51	0.23%	0.24%	17.4	0.8%	0.9%	7.39	0.8%	0.9%
Perfluorooctanesulfonic acid (PFOS)	210	94%	% <mark>66</mark>	1950	85%	<mark>66</mark> %	785	83%	<mark>%66</mark>
Perfluorodecanesulfonic acid (PFDS)	1.5	0.67%	0.71%	0.5	0.02%	0.03%	0.5	0.05%	0.06%
Perfluoroalkylcarboxylic acids	2.3	1%	%PFAA	44	2%	%PFAA	18	2%	%PFAA
Perfluorohexanoic acid (PFHxA)	0.77	0.35%	33%	5	0.2%	11%	5	%9.0	28%
Perfluoroheptanoic acid (PFHpA)	0.2	0.09%	8.7%	1.69	0.07%	4%	0.5	0.05%	3%
Perfluorooctanoic acid (PFOA)	0.94	0.42%	41%	25.5	1%	58%	0.50	0.05%	3%
Perfluorononanoic acid (PFNA)	0.034	0.02%	1.5%	0.5	0.02%	1%	0.5	0.05%	3%
Perfluorodecanoic acid (PFDA)	0.19	0.09%	8.2%	0.5	0.02%	1%	0.5	0.05%	3%
Perfluoroundecanoic acid (PFUnA)	0.1	0.04%	4.3%	0.5	0.02%	1%	0.5	0.05%	3%
Perfluorododecanoic acid (PFDoA)	0.073	0.03%	3.2%	5	0.22%	11%	5	0.5%	28%
Perfluorotridecanoic acid (PFTrA)	Not in	Not in analytical suite	suite	0.5	0.02%	1%	0.5	0.05%	3%
Perfluorotetradecanoic acid (PFTeA)	Not in	Not in analytical suite	suite	5	0.22%	11%	5	0.5%	28%
Other PFCs	8.3	4%	%OPFC	269	12%	%OPFC	135	14%	%OPFC
Perfluorooctanesulfonamide (PFOSA)	0.54	0.2%	6.5%	0.5	0.02%	0.2%	0.5	0.05%	0.4%
N-ethyl-perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	0.011	0.005%	0.13%	Not in a	Not in analytical suite	suite	Not in a	Not in analytical suite	suite
N-methyl-perfluorooctanesulfonamidoacetic acid (NMeFOSAA)	0.044	0.02%	0.53%	Not in a	Not in analytical suite	suite	Not in a	Not in analytical	suite
N-ethyl-perfluorooctanesulfonamide (NEtFOSA)	Not in	Not in analytical suite	suite	5	0.2%	2%	5	0.5%	4%
N-methyl-perfluorooctanesulfonamide (NMeFOSA)	Not in	Not in analytical suite	suite	5	0.2%	2%	5	0.5%	4%
N-ethyl-perfluorooctanesulfonamidothanol (NEtFOSE)	Not in	Not in analytical suite	suite	50	2%	19%	50	5%	37%
N-methyl-perfluorooctanesulfonamidoethanol (NMeFOSE)	Not in	Not in analytical suite	suite	50	2%	19%	50	5%	37%
1H,1H,2H,2H-perfluorohexanesulfonic acid (4:2 FTS)	Not	Not quantifiable	le	Not in a	Not in analytical suite	suite	Not in a	Not in analytical suite	suite
1H,1H,2H,2H-perfluorooctanesulfonic acid (6:2 FTS)	-	0.4%	12%	158	7%	59%	24	3%	18%
1H,1H,2H,2H-perfluorodecanesulfonic acid (8:2 FTS)	6.7	3%	81%	Not in a	Not in analytical suite	suite	Not in a	Not in analytical suite	suite

# Table D2: PFC surrogate percentages for sediments



### Appendix D 42 Pages

### Soil Sampling and QA/QC



### HUMAN HEALTH RISK ASSESSMENT – FISKVILLE COMMUNITY

### 4549 GEELONG-BALLAN RD, FISKVILLE VICTORIA

### **APPENDIX D**

### SOIL SAMPLING AWAY FROM TRAINING AREAS

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### APPENDIX D - SOIL SAMPLING AWAY FROM TRAINING AREAS

### **1** INTRODUCTION

This summary is intended to provide a description for the additional soil sampling conducted by Cardno Lane Piper in areas away from the training area at the CFA Fiskville Training College, Fiskville Vic (the "Site"). The work was conducted as per proposal reference 212163.18Proposal01.2, dated 18 April 2013. This summary does not have nor provides any discussions with regards to results or corresponding criteria.

### 1.1 Sampling Event and Sample Locations

The field event was conducted on 29 April 2013. A total of 18 near surface soil samples were collected at the Site at depth of 0.05 to 0.1 m Below Ground Level (mBGL). Sample locations are shown in Figure 1-1. The corresponding sample identification number and approximate georeferenced locations are provided in Table 1-1. Field notes including sample description log are provided in Attachment A



Figure 1-1: Sample Locations



Sample ID	Easting <sup>1</sup> (m)	Northing (m)
SS65	254242	5826272
SS66	254184	5825945
SS67	254381	5825884
SS68	254624	5825900
SS69	254556	5826095
SS70	254597	5826254
SS71	254399	5825669
SS72	254452	5825373
SS73	254626	5825372
SS74	254813	5825340
SS76	255121	5825270
SS77	255305	5825267
SS78	255303	5825456
SS79	255289	5825567
SS80	255117	5825638
SS81	255141	5825787
SS82	254993	5825790
Notes: 1. UTM Zone 55 (MGA94) reports an error or ± 10		ed to metre. The GPS system

### Table 1-1: Sample Location ID and Georeferenced Positions

### 1.2 Objective

The additional soil sampling effort was to assess the extent of soil contamination and potential for impact of spray drift from the firefighting training due to the presence of Perfluoro Compounds (PFCs) in the water. The data obtained from this assessment is incorporated in the Human Health Risk Assessments prepared for the Fiskville Community and Downstream Users (reported separately).

### 2 SOIL SAMPLING

### 2.1 Sample Strategy & Methodology

The scope and method of the sampling event is summarised in Table 2-1. Locations, shown in Table 1-1, were chosen to provide even coverage of the site away from the main PAD area and target areas with potential deposition of windblown foam.



Activity	Details				
Dates of Field Activity	29 April 2014				
Sample Collection	Soil samples were collected using a shovel or hand trowel at a depth of 0.05 to 0.1 mBGL.				
Soil Logging	Soils encountered during sampling were described and logged, and the corresponding soil descriptions are presented in Attachment A.				
Soil Sampling	Soil samples were collected into sample containers provided by the laboratory.				
Decontamination Procedure	Reusable soil sampling equipment was rinsed with Decon 90 and deionised water prior to the collection of subsequent samples.				
Soil Screening	PID screening did not report any evidence of hydrocarbons.				
Sample Preservation and Transport	Samples were stored on ice, in an esky while on-site and in transit to the laboratory under Chain of Custody documentation presented in Attachment B.				
*It is noted that the logs in	ndicate a depth of 0.2-0.3 mBGL, however this is incorrect. A review of photos of sample				

\*It is noted that the logs indicate a depth of 0.2-0.3 mBGL, however this is incorrect. A review of photos of sample locations (refer Attachment C) show near surface sample locations that are less than 0.1m in depth. This is further supported by the fact that the scope of work and the JSA for the work have shown "near surface samples" are to be collected.

### 2.2 Laboratory Analysis

All near-surface samples were submitted for laboratory testing and analysed for PFOS, PFOA and 6:2 FtS. Copies of the NATA accredited laboratory reports and sample receipt records are included in Attachment B. The Quality Assurance and Quality Control (QA/QC) of the soil sampling program is discussed in Section 3.

### 3 QUALITY ASSURANCE AND QUALITY CONTROL REVIEW

The following sections provide a summary review of QC.

### 3.1 Intra-Laboratory Analysis (ALS)

Two blind samples, QC1 and QC3, were submitted to ALS to assess the intra-laboratory reproducibility of the analysis. The Relative Percentage Difference (RPD) calculated from the parent samples (i.e. SS69 and SS78 respectively) are provided in Table 3-1.

Chemical Name	Units	LOR	SS69	QC1	RPD	SS78	QC3	RPD
PFOS <sup>1</sup>		0.0005	0.0168	0.0152	10	0.0399	0.018	35
PFOA	mg/kg	0.0005	0.0006	0.0005	18	0.0007	0.0006	15
6:2 FtS		0.005	<0.005	<0.005	0	<0.005	<0.005	0
Note: 1. PFOS is reported i	in units of µg/k	g in the analy	ytical reports.					

### Table 3-1: %RPD Calculation for Intra-laboratory Assessment



The %RPD calculation shows that there is no systematic error in the laboratory assessment and the results calculated are within the acceptable range of < 50%.

### 3.2 Inter-Laboratory Analysis (ALS and Eurofins-MGT)

One blind sample, QC2, was submitted to Eurofins-MGT to assess the intra-laboratory reproducibility of the analysis. The RPD calculated from the parent sample (i.e. SS69) for the corresponding analysis are provided in Table 3-2.

Chemical Name	Units	LOR	SS69	QC2	RPD
PFOS <sup>1</sup>		0.0005	0.0168	0.018	14
PFOA	mg/kg	0.0005	0.0006	<0.0022	-
6:2 FtS		0.005	<0.005	<0.0033	-
Note: 1. PFOS is reported ir 2. "-" indicates %RPD				t less than lab	oratory LOR.

 Table 3-2: %RPD Calculation for Inter-laboratory Assessment

The %RPD for PFOS shows an acceptable correlation between the two laboratories. The high %RPD for PFOA and 6:2 FtS are outside the acceptable range; however, this is due to calculating an RPD for data which reported less than laboratory LOR and differences in the laboratories LOR. The data is considered acceptable since the results for PFOS has a %RPD less that 20% and it is the main chemical indicator for the current assessment.

### 3.3 Field Blank – Rinsate

One field rinsate was collected for the field event. The results from the analysis, shown in Table 3-3, for the contaminants of concern demonstrates that the field decontamination that was put in place did provide adequate quality control between sample locations.

Chemical Name	Units	LOR	Reported
PFOS	mg/L	0.00002	<0.00002
PFOA	mg/L	0.00002	<0.00002
6:2 FtS	mg/L	0.0001	<0.0001

### Table 3-3: Field Rinsate

### 3.4 Soil Results

A summary of the soil results is provided in Table 3-4. The extended soil sampling away from the PAD training showed detectable levels of PFCs suggesting potential for windblown dispersion.



	Number of Analysis	Number of Detects	Minimum Detect	Maximum Detect	Detection (%)
PFOS	18	18	0.0032	0.258	100
PFOA	18	10	0.0005	0.0204	56
6:2 FtS	18	2	0.027	0.144	11

### Table 3-4: Summary of Soil Analysis

### 4 ATTACHMENTS

### Attachment A

Field Notes

### **Attachment B**

Laboratory Reports and Chain of Custody

### **Attachment C**

Photos

### Cardno Lane Piper March 2014





### QF3.01 - Fieldwork Daily Report

Project Detail	S S S S S S S S S S S S S S S S S S S
Project Name: Soil Assessment	Job Number: 212163 . 18
Site Address: Fiskiville	PP/PM: ADL/LMR
Client Company/Contact: CFA	Date: 29 4
Persons Present: SD	Notes By: 56

Site Activities	Yes	Comment/Details
PESA Site Inspection / Interview personnel	~	
Inspect or supervise bores/test pits/ observe sampling/ remediation works	-	2
Audit fieldwork methods QA/QC	- ,	
Soil sampling - test pit / soil bore (soil grab		
Soil gas / LFG investigation	-	
Groundwater bore construction / GME / Groundwater levels / sampling	-	
Geotechnical Investigation		
Compaction Control Tests	- ,	
Field consumables used? (if so what?)	, ·	These must be charged via timesheet
Photographs (Digital)		
Supplementary notes attached		
Weather Conditions & Temperature	T:  { °C	Windy, party sunny

Notes / Sketch Plan:

Arrival on site - 8:150m Arrival of Shocter - 8:35am - JSA & sign in \$\$\$ site \$ inspection, delay in CFA induction (JJ/Martyon preoccupied) > induction by Martyn at 10.15

Sheder left @ 10:30am Stort seil sampling @ 10:45 Lunch - 12:00 - 12:30 Finish - Left site - 3:30pm

( Condrag	DAUVTOOL	DOV CAFETV	ACCTINIC
Cardno LanePiper		BOX SAFETY	2
Date:4 13		Car Car	dno Job No.: 212163-18
Client: CFA	Site ID:	Fiskentle	
Site Address: 4545 Specific Location:	Gectang Ballan	Kol	
Type of Work:	assessment	Chemicals Used:	
	assessment		
SAFETY TOPICS PRESENTED			
Protective Clothing / Equipment		-	
Hard Hat	Steel-Toed Boots	Long Sleeve Protectior	Air Monitoring
Safety Glasses	Gloves	Hearing Protection	Respirator
Safety Goggles	Reflective Traffic Vest	Tyvek Suit	Other:
Biological Hazards			
Bees / Wasps Chemical Hazards	Spiders	Snakes	Other:
	its in Soil / Groundwater	X Other: $PFCs$	
Physical Hazards			
Drilling Equipment	Vehicle Traffic	Material Handling	Overhead/Buried Utilities
Earth-moving Equip.	🔀 Pedestrian Traffic	Pinch Points	Inclement Weather
Crane(s)	Slips, Trips & Falls	Elec./Shock Hazards	Other:
Special Equipment			
Traffic Control Safety Documents	Exclusion Zone	Barricades	Other:
White Card	LPS Training	JSA/SWP Reviewed	Safety Alerts
Required Permits			
Hot Work Permit	Well Const/Dest Permit	Other	Other:
Additional / Other Safety Topics	Presented:	2	
			р) -
EMERGENCY PROCEDURES	Call 0-0-0 (cell phone) 1-1-2	Apply First Aid	Emergency Rally Point
HOSPITAL/CLINIC INFORMATION	1	DIRECTIONS TO HOSPI	
	tospital		
Phone No:			
Address:			
City, State:			
ATTENDEES			
NAME PRINTED	S	IGNATURE	COMPANY
ROD MASONI	A	9 20	17
PHIL SAN SOM	the		JENSAN FARM
		8	SERVICES
5			
MEETING CONDUCTED BY:			
0	Amon	ula are	UP
DRISEETA DE NAME PRINTED		IGNATURE	COMPANY

C Cardno LanePiper

Integrated Management System Procedure Manual QF3.03 – Soil Sample Descriptions

## QF3.03 – Soil Sample Descriptions

Project Details	
Project Name: Community Risk - Soil Assessment	Job Number: 212163 - 18
Site Address: Fiskunik V	PP/PM: APL LUNE
Client Company/Contact:	Date: 29/4/13
Persons Present: SD	Notes By: 'So'

Sample No.	Depth Interval	Soil Type	Description (Include fill/natural, texture, moisture, plasticity, colour, odours noted, inclusions)	PID (ppm) (Headspace)
SS 65	0.2-0.3	Sandy Cory	Brawn, O'ange matting, L-Mp, dry, med grained	0.0
SS 66		Sitty Clay	0.2-0.5 Sitty Clay Braun/Grey, Lp, dry, fine grained	0-0
F9 22	SS 67 0.2-2.3 SHY Cloy	Sity Clary	te v V	0.0
SS 68	0.2 - 0.3	0.2 - 0.3 Shundy Clay	" Np, diry fine to wed preined.	0-0
SS69	0.2 - 0.3	SS69 0.2-0.3 Sitty day	Grey/Breven, LP, dry, free are	0 - 0
of SS	SS 70 0.2 - 0.3	23		0-0
Itss	SS71 0.2-0.3	N	N N N	1.0
St SS	SS 72 0.2 - 0.3	М	11 M K 11	0.0
SF2S	0.2-0.3	Sandy Gogy	SS73 0.2-0.3 Soundy Goy As SS 65 without but wet	0-0
Revision: 1			QF3.03 Soil Sample Descriptions dotx	Page 1 of 32

Approved: 4 January 2013

Printed: 24/04/2013

This document is current for 24 hours after print date QF3.03 Soil Sample Descriptions.dotx

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Integrated Management System Procedure Manual QF3.03 – Soil Sample Descriptions

Sample No.	Depth Interval	Soil Type	Description (Include fill/natural, texture, moisture, plasticity, colour, odours noted, inclusions)	PID (ppm) (Headspace)
SS74	0.2-0.3	Sandy Clay	AS SS 68	0.0
SFSS	М	Sundy Clary	As de SS68	0:0
9£SS	N	Sity Clay	AS SS 66	0-0
ttss	M	SHA Clay	61	0-1
StSS	М	Silty Clay	13	0.0
bt SS	J1	Sandy Clay	Brewn, orange red, metting, MP, dry, nedgrained	0 · 0
SS 80	<u>J</u>	-		0.0
SS&1	Þć	М	1/ 1/ 1/ 1/ 1/	0.0
BCI	у		45 SS 69	0.0
QC2	И			0.0
gc3	Ņ		AS SS 76	0 . ()
Qc4	h		VI.	0-0
Revision: 1 Approved: 4 January 2013	lanuary 2013		QF3.03 Soil Sample Descriptions.dotx This document is current for 24 hours after print date	Page 2 مۇچ ك



### QF3.01 – Quality Control Sample Register

Project Detai	ls
Project Name: 2 Soil Assessment	Job Number: 212163 18
Site Address: Fiskville	PP/PM: APULLMR
Client Company/Contact: CFA	Date: 29/4
Persons Present: AD	Notes By: So

### **Quality Control Requirements**

### Standard QC Sample Requirements (see Proposal for project specific details)

Rinsate Blank:	1/day (even if only placed on hold)
DI Water Blank:	1/day (even if only placed on hold)
Trip Blank:	1/day or 1/esky (if volatiles are suspected or present at site)
Blind Replicate (Primary List):	1 in 20 primary samples
Split Replicate (Secondary List):	1 in 20 primary samples

### Labelling

Samples to be labelled **QC##\_date** where "##" is a numerical sequence commencing at **01** for each field event and **date** is the date of sampling in ddmmyyyy format (**e.g. QC01\_03112010**)

	Qualit	y Control Sample Register	
<b>QC Sample</b> e.g. QC01_03112010	Primary Sample	Description	DI Water Batch Number
0.01	SS 69	Que	
QCZ	5569	Trip	
QCS	SS78	Dup	
QCA	SS78	Trip	
3			
QCA	Rinsafe		
QCB	Trip. B		
	,		

Environmental Division	) Group			Sciences
		CERTIFICATE	E OF ANALYSIS	
Work Order	: EM1304402			: 1 of 7
Client	CARDNO LANE PIPER PTY LTD	ΤΥ LTD	Laboratory	: Environmental Division Melbourne
Contact	: MS SRIJEETA DE		Contact	: Carol Walsh
Address	: 154 HIGHBURY ROAD		Address	: 4 Westall Rd Springvale VIC Australia 3171
	BURWOUD VIC, AUSTRALIA 3123	ALIA 3123		محمد المامانية فالمعالمة المستعدية
с-maii Telephone	: srijeeta.de@cardno.com.au · +61 03 98880100	au	E-mail Telephone	: caroi.walsn@aisgiopai.com · +61-3-8549 9608
Facsimile	: +61 03 98083511		Facsimile	: +61-3-8549 9601
Project	: 212163 18		QC Level	: NEPM 1999 Schedule B(3) and ALS QCS3 requirement
Order number				
C-O-C number			Date Samples Received	: 30-APR-2013
Sampler	: SDe		Issue Date	: 07-MAY-2013
Site	: CFA			
			No. of samples received	: 23
Quote number	: MEBQ/115/12		No. of samples analysed	: 21
This Certificate of General	This Certificate of Analysis contains the following information:	ormation:		
NATA	NATA Accredited Laboratory 825 Accredited for compliance with ISO/IEC 17025.	Signatories This document has been electroni carried out in compliance with procedure Signatories	been electronically signed by the authorized se with procedures specified in 21 CFR Part 11. <i>Position</i>	orized signatories indicated below. Electronic signing has been Accreditation Category
WORLD RECOGNISED		Phalak Inthaksone Phalak Inthaksone	Laboratory Manager - Organics Laboratory Manager - Organics	Organics Sydney Inorganics Organics Sydney Organics
		Addrass 4 Westall Rd Springvale VIC Australia 3171   PHONE +61-3-8549 9600   Facsimile +61-3-8549 9601 Environmental Division Melbourne ABN 84 009 936 029 Part of the ALS Group Ad IS i imited Commany	gyale VIC Australia 3171   PHONE +61-3-8549 9600   Facstimile +61-3-8549 9601 leibourine ABN 84 009 936 029 Part of the ALS Groun An ALS Limited Commany	1-3-8549 9601 vited Pownanov
Environmental 🔊	ental 🔏		www.alsolobal.com	
			0	

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: 2 of 7	: EM1304402	: CARDNO LANE PIPER PTY LTD	: 212163 18
Page	Work Order	Client	Project



### **General Comments**

The analytical procedures used by the Environmental Division have been developed from established internationally recognized procedures such as those published by the USEPA, APHA, AS and NEPM. In house developed procedures are employed in the absence of documented standards or by client request.

Where moisture determination has been performed, results are reported on a dry weight basis.

Where a reported less than (<) result is higher than the LOR, this may be due to primary sample extract/digestate dilution and/or insufficient sample for analysis.

Where the LOR of a reported result differs from standard LOR, this may be due to high moisture content, insufficient sample (reduced weight employed) or matrix interference.

When sampling time information is not provided by the client, sampling dates are shown without a time component. In these instances, the time component has been assumed by the laboratory for processing purposes.

CAS Number = CAS registry number from database maintained by Chemical Abstracts Services. The Chemical Abstracts Service is a division of the American Chemical Society. LOR = Limit of reporting Key :

A = This result is computed from individual analyte detections at or above the level of reporting

- EP231: PFOA & PFOS results are reported as an aggregate of linear and branched isomers.
  - PFOS/PFOA conducted by ALS Sydney, NATA accreditation no. 825, site no 10911.

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### Analytical Results

Sub-Matrix: SOIL (Matrix: SOIL)		Clier	Client sample ID	SS65	SS66	SS67	SS68	SS69
	Cli	ent samplin <sub>t</sub>	Client sampling date / time	29-APR-2013 10:45				
Compound	CAS Number LOR	LOR	Unit	EM1304402-001	EM1304402-002	EM1304402-003	EM1304402-004	EM1304402-005
EA055: Moisture Content								
Moisture Content (dried @ 103°C)		1.0	%	19.3	10.9	7.2	10.2	8.8
EP231: Perfluorinated Compounds								
PFOS	1763-23-1	0.0005	mg/kg	0.0032	0.0575	0.0117	0.0180	0.0168
PFOA	335-67-1 0.0005	0.0005	mg/kg	<0.0005	0.0024	0.0005	0.0005	0.0006
6:2 Fluorotelomer sulfonate (6:2	27619-97-2	0.005	mg/kg	<0.005	<0.005	<0.005	<0.005	<0.005
FtS)								

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Sub-Matrix: SOIL (Matrix: SOIL)		Clie	Client sample ID	SS70	SS71	SS72	SS73	SS74	
	CI	ient samplir	Client sampling date / time	29-APR-2013 10:45					
Compound	CAS Number LOR	LOR	Unit	EM1304402-006	EM1304402-007	EM1304402-008	EM1304402-009	EM1304402-010	
EA055: Moisture Content									
Moisture Content (dried @ 103°C)		1.0	%	12.8	4.6	10.0	23.5	11.7	
EP231: Perfluorinated Compounds									
PFOS	1763-23-1 0.0005	0.0005	mg/kg	0.0102	0.0043	0.0073	0.0146	0.0298	
PFOA	335-67-1 0.0005	0.0005	mg/kg	<0.0005	<0.0005	<0.0005	<0.0005	0.0011	
6:2 Fluorotelomer sulfonate (6:2	27619-97-2 0.005	0.005	mg/kg	<0.005	<0.005	<0.005	<0.005	<0.005	
FtS)									

: 5 of 7	: EM1304402	: CARDNO LANE PIPER PTY LTD	: 212163 18
Page	Work Order	Client	Project



Analytical Results								
Sub-Matrix: SOIL (Matrix: SOIL)		Clier	Client sample ID	SS75	SS76	SS77	SS78	SS79
	Cli	ent sampling	Client sampling date / time	29-APR-2013 10:45	29-APR-2013 10:45	29-APR-2013 10:45	29-APR-2013 10:45	29-APR-2013 14:45
Compound	CAS Number LOR	LOR	Unit	EM1304402-011	EM1304402-012	EM1304402-013	EM1304402-014	EM1304402-015
EA055: Moisture Content								
Moisture Content (dried @ 103°C)		1.0	%	15.5	6.7	9.2	10.6	8.4
EP231: Perfluorinated Compounds								
PFOS	1763-23-1 0.0005	0.0005	mg/kg	0.258	0.0073	0.0079	0.0399	0.0427

**0.0023** <0.005 0.0427

**0.0399 0.0007** <0.005

**0.0079** <0.0005 <0.005

**0.0073** <0.0005 <0.005

0.258 0.0204 0.144

mg/kg mg/kg mg/kg

 1763-23-1
 0.0005

 335-67-1
 0.0005

 27619-97-2
 0.005

6:2 Fluorotelomer sulfonate (6:2 FtS)

PFOA



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	•••	•••	
Page	Work Order	Client	Project



sub-Matrix: SOIL (Matrix: SOIL) Clie	Client samplir	CAS Number LOR	A055: Moisture Content	Moisture Content (dried @ 103°C) 1.0
Client sample ID	sampling date / time	Unit		%
SS80	29-APR-2013 15:00	EM1304402-016		10.0
SS81	29-APR-2013 15:00	EM1304402-017		8.7
SS82	29-APR-2013 15:00	EM1304402-018		9.5
QC1	29-APR-2013 15:00	EM1304402-021		8.8



29-APR-2013 15:00 EM1304402-022

10.6

QC3

0.0279 0.0006 <0.005

**0.0152 0.0005** <0.005

**0.0510** <0.0005 <0.005

0.0794 0.0062 0.027

**0.0384 0.0011** <0.005

mg/kg mg/kg mg/kg

 1763-23-1
 0.0005

 335-67-1
 0.0005

EP231: Perfluorinated Compounds PFOS 27619-97-2 0.005

6:2 Fluorotelomer sulfonate (6:2 FtS)

PFOA

: 7 of 7 : EM1304402 : CARDNO LANE PIPER PTY LTD	; 212163 18
Page Work Order Client	Project



### Analytical Results

QCA	
Client sample ID	
Sub-Matrix: WATER (Matrix: WATER)	

	Clie	ent samplin	Client sampling date / time	29-APR-2013 15:00	 	 
Compound	CAS Number LOR Unit	LOR	Unit	EM1304402-019	 	 
EP231: Perfluorooctyl Acids and Sulfonates.						
PFOS	1763-23-1 0.02	0.02	hg/L	<0.02	 	 
PFOA	335-67-1	0.02	µg/L	<0.02	 	 
6:2 Fluorotelomer sulfonate (6:2 FtS)	27619-97-2 0.1	0.1	hg/L	<0.1	 	 
	-					

Work Order Client Contact	EM1304402 CARDNO LANE PIPER PTY LTD	QUALITY CON	TROL REPORT		
nt ntact	<b>CARDNO LANE PIPER PT</b>		Page	: 1 of 5	
Address	: MS SRIJEETA DE : 154 HIGHBURY ROAD BURWOOD VIC, AUSTRALIA 3125	<b>Y LTD</b> ALIA 3125	Laboratory Contact Address	: Environmental Division Melbourne : Carol Walsh : 4 Westall Rd Springvale VIC Australia 3171	3171
E-mail Telephone Facsimile	: srijeeta.de@cardno.com.au : +61 03 98880100 : +61 03 98083511	Ξ	E-mail Telephone Facsimile	: carol.walsh@alsglobal.com : +61-3-8549 9608 : +61-3-8549 9601	
Project Site	: 212163 18 · CEA		QC Level	: NEPM 1999 Schedule B(3) and ALS QCS3 requirement	QCS3 requirement
C-O-C number Sampler Order number	SDe		Date Samples Received Issue Date	: 30-APR-2013 : 07-MAY-2013	
Quote number	 . MEBQ/115/12		No. of samples received No. of samples analysed	: 23	
This report supersedes release. This Quality Control Repoi Laboratory Duplicat Method Blank (MB) Matrix Spike (MS) F	<ul> <li>This report supersedes any previous report(s) with this release.</li> <li>This Quality Control Report contains the following information: <ul> <li>Laboratory Duplicate (DUP) Report; Relative Percentage Dif</li> <li>Method Blank (MB) and Laboratory Control Spike (LCS) Rep</li> <li>Matrix Spike (MS) Report; Recovery and Acceptance Limits</li> </ul> </li> </ul>	port supersedes any previous report(s) with this reference. Results apply to the ality Control Report contains the following information: Laboratory Duplicate (DUP) Report; Relative Percentage Difference (RPD) and Acceptance Limits Method Blank (MB) and Laboratory Control Spike (LCS) Report; Recovery and Acceptance Limits Matrix Spike (MS) Report; Recovery and Acceptance Limits	sample(s) as submitted.	All pages of this report have been	checked and approved for
	NATA Accredited Laboratory 825 Accredited for compliance with ISO/IEC 17025.	<i>Signatories</i> This document has been electronically signed by the autho carried out in compliance with procedures specified in 21 CFR Part 11. <i>Signatories</i>	electronically signed by the authorized procedures specified in 21 CFR Part 11. <i>Position</i>	signatories indicated below. Accreditation Ca	Electronic signing has been tegory
WORLD RECOGNISED		Phalak Inthaksone Phalak Inthaksone	Laboratory Manager - Organics Laboratory Manager - Organics	nics Sydney Inorganics Sydney Organics	

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### **General Comments**

The analytical procedures used by the Environmental Division have been developed from established internationally recognized procedures such as those published by the USEPA, APHA, AS and NEPM. In house developed procedures are employed in the absence of documented standards or by client request.

Where moisture determination has been performed, results are reported on a dry weight basis.

Where a reported less than (<) result is higher than the LOR, this may be due to primary sample extract/digestate dilution and/or insufficient sample for analysis.

Where the LOR of a reported result differs from standard LOR, this may be due to high moisture content, insufficient sample (reduced weight employed) or matrix interference.

CAS Number = CAS registry number from database maintained by Chemical Abstracts Services. The Chemical Abstracts Service is a division of the American Chemical Society. Anonymous = Refers to samples which are not specifically part of this work order but formed part of the QC process lot RPD = Relative Percentage Difference LOR = Limit of reporting Key :

# = Indicates failed QC

: 3 of 5	; EM1304402	: CARDNO LANE PIPER PTY LTD	: 212163 18
Page	Work Order	Client	Project



## Laboratory Duplicate (DUP) Report

The quality control term Laboratory Duplicate refers to a randomly selected intralaboratory split. Laboratory duplicates provide information regarding method precision and sample heterogeneity. The permitted ranges for the Relative Percent Deviation (RPD) of Laboratory Duplicates are specified in ALS Method QWI-EN/38 and are dependent on the magnitude of results in comparison to the level of reporting: Result < 10 times LOR:-No Limit, Result between 10 and 20 times LOR:-0% - 50%; Result > 20 times LOR:-0% - 20%.

			l						
Sub-Matrix: SOIL						Laboratory L	Laboratory Duplicate (DUP) Report		
Laboratory sample ID	Client sample ID	Method: Compound	CAS Number	LOR	Unit	<b>Original Result</b>	Duplicate Result	RPD (%)	Recovery Limits (%)
EA055: Moisture Co	EA055: Moisture Content (QC Lot: 2853239)								
EM1304402-003	SS67	EA055-103: Moisture Content (dried @ 103°C)		1.0	%	7.2	6.6	9.5	No Limit
EM1304402-014	SS78	EA055-103: Moisture Content (dried @ 103°C)		1.0	%	10.6	11.3	6.7	0% - 50%
EP231: Perfluorinate	EP231: Perfluorinated Compounds (QC Lot: 2846523)	46523)							
EM1304402-001	SS65	EP231: PFOS	1763-23-1	0.0005	mg/kg	0.0032	0.0019	50.2	No Limit
		EP231: PFOA	335-67-1	0.0005	mg/kg	<0.0005	<0.0005	0.0	No Limit
		EP231: 6:2 Fluorotelomer sulfonate (6:2 FtS)	27619-97-2	0.005	mg/kg	<0.005	<0.005	0.0	No Limit
EM1304402-011	SS75	EP231: PFOS	1763-23-1	0.0005	mg/kg	0.258	0.229	11.9	0% - 20%
		EP231: PFOA	335-67-1	0.0005	mg/kg	0.0204	0.0179	13.0	0% - 20%
		EP231: 6:2 Fluorotelomer sulfonate (6:2 FtS)	27619-97-2	0.005	mg/kg	0.144	0.119	19.5	0% - 20%
Sub-Matrix: WATER						Laboratory L	Laboratory Duplicate (DUP) Report		
Laboratory sample ID	Client sample ID	Method: Compound	CAS Number	LOR	Unit	<b>Original Result</b>	Duplicate Result	RPD (%)	Recovery Limits (%)
EP231: Perfluorinate	EP231: Perfluorinated Compounds (QC Lot: 2847277)	47277)							
EM1304402-019	QCA	EP231: PFOS	1763-23-1	0.02	hg/L	<0.02	<0.02	0.0	No Limit
		EP231: PFOA	335-67-1	0.02	hg/L	<0.02	<0.02	0.0	No Limit
		EP231: 6:2 Fluorotelomer sulfonate (6:2 FtS)	27619-97-2	0.1	hg/L	<0.1	<0.1	0.0	No Limit

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# Method Blank (MB) and Laboratory Control Spike (LCS) Report

parameter is to monitor potential laboratory contamination. The quality control term Laboratory Control Sample (LCS) refers to a certified reference material, or a known interference free matrix spiked with target The quality control term Method / Laboratory Blank refers to an analyte free matrix to which all reagents are added in the same volumes or proportions as used in standard sample preparation. The purpose of this QC analytes. The purpose of this QC parameter is to monitor method precision and accuracy independent of sample matrix. Dynamic Recovery Limits are based on statistical evaluation of processed LCS.

Method Blank (MB)

Laboratory Control Spike (LCS) Report

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				Report	Spike	Spike Recovery (%)	Recovery Limits (%)	Limits (%)
Method: Compound	CAS Number	LOR	Unit	Result	Concentration	SDT	Том	High
EP231: Perfluorinated Compounds (QCLot: 2846523)								
EP231: PFOS	1763-23-1	0.0005	mg/kg	<0.0005	.0025 mg/kg	85.8	54	146
EP231: PFOA	335-67-1	0.0005	mg/kg	<0.0005	.0025 mg/kg	74.8	54	134
EP231: 6:2 Fluorotelomer Sulfonate (6:2 FtS)	27619-97-2	0.005	mg/kg	<0.005	.0125 mg/kg	72.8	56	138
Sub-Matrix: WATER				Method Blank (MB)		Laboratory Control Spike (LCS) Report	S) Report	
				Report	Spike	Spike Recovery (%)	Recovery Limits (%)	Limits (%)
Method: Compound	CAS Number	LOR	Unit	Result	Concentration	7CS	Том	High
EP231: Perfluorinated Compounds (QCLot: 2847277)								
EP231: PFOS	1763-23-1	0.02	hg/L	<0.02	0.25 µg/L	111	20	136
EP231: PFOA	335-67-1	0.02	hg/L	<0.02	0.25 µg/L	94.6	72	134
EP231: 6:2 Fluorotelomer Sulfonate (6:2 FtS)	27619-97-2	0.1	hg/L	<0.1	1.25 µg/L	7.77	61	145

## Matrix Spike (MS) Report

Б parameter is to monitor potential matrix effects The quality control term Matrix Spike (MS) refers to an intralaboratory split sample spiked with a representative set of target analytes. The purpose of this QC analyte recoveries. Static Recovery Limits as per laboratory Data Quality Objectives (DQOs). Ideal recovery ranges stated may be waived in the event of sample matrix interference.

Sub-Matrix: SOIL				Ma	Matrix Spike (MS) Report		
				Spike	SpikeRecovery(%)	Recovery Limits (%)	imits (%)
Laboratory sample ID	Client sample ID	Method: Compound	CAS Number	Concentration	SM	том	High
EP231: Perfluorina	EP231: Perfluorinated Compounds (QCLot: 2846523)						
EM1304402-001	SS65	EP231: PFOS	1763-23-1	.0025 mg/kg	82.3	54	146
		EP231: PFOA	335-67-1	.0025 mg/kg	102	54	134
		EP231: 6:2 Fluorotelomer sulfonate (6:2 FtS)	27619-97-2	.0125 mg/kg	63.2	56	138
Sub-Matrix: WATER				Me	Matrix Spike (MS) Report		
				Spike	SpikeRecovery(%)	Recovery Limits (%)	imits (%)
Laboratory sample ID	Client sample ID	Method: Compound	CAS Number	Concentration	SW	том	High
EP231: Perfluorina	EP231: Perfluorinated Compounds(QCLot: 2847277)						
EM1304402-019	QCA	EP231: PFOS	1763-23-1	0.25 µg/L	106	70	136
		EP231: PFOA	335-67-1	0.25 µg/L	102	72	134
		EP231: 6:2 Fluorotelomer sulfonate (6:2 FtS)	27619-97-2	1.25 µg/L	98.8	61	145

# Matrix Spike (MS) and Matrix Spike Duplicate (MSD) Report

9 parameters are The quality control term Matrix Spike (MS) and Matrix Spike Duplicate (MSD) refers to intralaboratory split samples spiked with a representative set of target analytes. The purpose of these QC monitor potential matrix effects on analyte recoveries. Static Recovery Limits as per laboratory Data Quality Objectives (DQOS). Ideal recovery ranges stated may be waived in the event of sample matrix interference.

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Sub-Matrix: SOIL					Matrix Spike (I	Matrix Spike (MS) and Matrix Spike Duplicate (MSD) Report	ike Duplicate	(MSD) Repor	t	
				Spike	Spike Re	Spike Recovery (%)	Recovery	Recovery Limits (%)	RPDs (%)	(%)
Laboratory sample ID	Client sample ID	Method: Compound	CAS Number	Concentration	SM	MSD	том	High	Value	<b>Control Limit</b>
EP231: Perfluorina	EP231: Perfluorinated Compounds(QCLot: 2846523)									
EM1304402-001	SS65	EP231: PFOS	1763-23-1	.0025 mg/kg	82.3	1	54	146	-	
		EP231: PFOA	335-67-1	.0025 mg/kg	102		54	134		
		EP231: 6:2 Fluorotelomer sulfonate (6:2 FtS)	27619-97-2	27619-97-2 .0125 mg/kg	63.2		56	138		
Sub-Matrix: WATER					Matrix Spike (I	Matrix Spike (MS) and Matrix Spike Duplicate (MSD) Report	ike Duplicate	(MSD) Repor	t	
				Spike	Spike Re	Spike Recovery (%)	Recovery	Recovery Limits (%)	RPD	RPDs (%)
Laboratory sample ID	Client sample ID	Method: Compound	CAS Number	Concentration	SM	MSD	Том	High	Value	<b>Control Limit</b>
EP231: Perfluorina	EP231: Perfluorinated Compounds(QCLot: 2847277)									
EM1304402-019	QCA	EP231: PFOS	1763-23-1	0.25 µg/L	106		20	136		
		EP231: PFOA	335-67-1	0.25 µg/L	102		72	134		
		EP231: 6:2 Fluorotelomer sulfonate (6:2 FtS)	27619-97-2	1.25 µg/L	98.8		61	145	-	

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**Environmental Division** 

# INTERPRETIVE QUALITY CONTROL REPORT

Work Order	:EM1304402	Page	: 1 of 5
Client Contact Address	: CARDNO LANE PIPER PTY LTD : MS SRIJEETA DE : 154 HIGHBURY ROAD BURWOOD VIC, AUSTRALIA 3125	Laboratory Contact Address	: Environmental Division Melbourne : Carol Walsh : 4 Westall Rd Springvale VIC Australia 3171
E-mail	: srijeeta.de@cardno.com.au	E-mail	: carol.walsh@alsglobal.com
Telephone	: +61 03 98880100	Telephone	: +61-3-8549 9608
Facsimile	: +61 03 98083511	Facsimile	: +61-3-8549 9601
Project Site	: 212163 18 : CFA	QC Level	: NEPM 1999 Schedule B(3) and ALS QCS3 requirement
C-O-C number	: SDe	Date Samples Received	: 30-APR-2013
Sampler		Issue Date	: 07-MAY-2013
Order number	:	No. of samples received	: 23
Quote number	: MEBQ/115/12	No. of samples analysed	: 21

This report supersedes any previous report(s) with this reference. Results apply to the sample(s) as submitted. All pages of this report have been checked and approved for release.

This Interpretive Quality Control Report contains the following information:

- Analysis Holding Time Compliance
- Quality Control Parameter Frequency Compliance
  - Brief Method Summaries

    - Summary of Outliers

Address 4 Westall Rd Springvale VIC Australia 3171 | PHONE +61-3-8549 9600 | Facslimite +61-3-8549 9601 Environmental Division Melbourne ABN 84 009 936 029 Part of the ALS Group An ALS Limited Company

Environmental 🕅



## **Analysis Holding Time Compliance**

dilutions and reruns. Information is also provided re the sample container (preservative) from which the analysis aliquot was taken. Elapsed period to analysis represents number of days from sampling where no for laboratory produced leachates is assumed as the completion date of the leaching process. Outliers for holding time are based on USEPA SW 846, APHA, AS and NEPM (1999). A listing of breaches is provided in The following report summarises extraction / preparation and analysis times and compares with recommended holding times. Dates reported represent first date of extraction or analysis and precludes subsequent extraction / digestion is involved or period from extraction / digestion where this is present. For composite samples, sampling date is assumed to be that of the oldest sample contributing to the composite. Sample date the Summary of Outliers. Holding times for leachate methods (excluding elutriates) vary according to the analytes being determined on the resulting solution. For non-volatile analytes, the holding time compliance assessment compares the leach date with the shortest analyte holding time for the equivalent soil method. These soil holding times are: Organics (14 days); Mercury (28 days) & other metals (180 days). A recorded breach therefore does not guarantee a breach for all non-volatile parameters.

Matrix: SOIL					Evaluation:	<pre>x = Holding time t</pre>	Evaluation: $\mathbf{x}$ = Holding time breach ; $\mathbf{v}$ = Within holding time.	holding time.
Method		Sample Date	Ext	Extraction / Preparation			Analysis	
<b>Container</b> / Client Sample ID(s)			Date extracted	Due for extraction	Evaluation	Date analysed	Due for analysis	Evaluation
EA055: Moisture Content								
Soil Glass Jar - Unpreserved (EA055-103)								
SS65,	SS66,	29-APR-2013	-			06-MAY-2013	13-MAY-2013	>
SS67,	SS68,							
SS69,	SS70,							
SS71,	SS72,							
SS73,	SS74,							
SS75,	SS76,							
SS77,	SS78,							
SS79,	SS80,							
SS81,	SS82,							
QC1,	QC3							
EP231: Perfluorinated Compounds								
Soil Glass Jar - Unpreserved (EP231)								
SS65,	SS66,	29-APR-2013	02-MAY-2013	26-OCT-2013	>	02-MAY-2013	11-JUN-2013	>
SS67,	SS68,							
SS69,	SS70,							
SS71,	SS72,							
SS73,	SS74,							
SS75,	SS76,							
SS77,	SS78,							
SS79,	SS80,							
SS81,	SS82,							
ac1,	QC3							
Matrix: WATER					Evaluation:	<pre>x = Holding time t</pre>	Evaluation: $\mathbf{x}$ = Holding time breach ; $\checkmark$ = Within holding time.	holding time.
Method		Sample Date	Ext	Extraction / Preparation			Analysis	
<b>Container</b> / Client Sample ID(s)			Date extracted	Due for extraction	Evaluation	Date analysed	Due for analysis	Evaluation
EP231: Perfluorooctyl Acids and Sulfonates.								
Sterile Plastic Bottle - Sodium Thiosulfate (EP231) QCA		29-APR-2013	ł	26-OCT-2013		02-MAY-2013	26-OCT-2013	>

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The following report summarises the frequency of laboratory QC samples analysed within the analytical lot(s) in which the submitted sample(s) was(where) processed. Actual rate should be greater than or equal to the expected rate. A listing of breaches is provided in the Summary of Outliers.

Matrix: SOIL

Evaluation: **x** = Quality Control frequency not within specification;  $\checkmark$  = Quality Control frequency within specification.

						sour foundation to much foundation	
Quality Control Sample Type		ö	Count		Rate (%)		Quality Control Specification
Analytical Methods	Method	QC	Reaular	Actual	Expected	Evaluation	
Laboratory Duplicates (DUP)							
Moisture Content	EA055-103	2	20	10.0	10.0	>	NEPM 1999 Schedule B(3) and ALS QCS3 requirement
Perfluorooctyl Acids and Sulfonates by LC/MS/MS	EP231	2	20	10.0	10.0	>	NEPM 1999 Schedule B(3) and ALS QCS3 requirement
Laboratory Control Samples (LCS)							
Perfluorooctyl Acids and Sulfonates by LC/MS/MS	EP231	1	20	5.0	5.0	>	NEPM 1999 Schedule B(3) and ALS QCS3 requirement
Method Blanks (MB)							
Perfluorooctyl Acids and Sulfonates by LC/MS/MS	EP231	-	20	5.0	5.0	>	NEPM 1999 Schedule B(3) and ALS QCS3 requirement
Matrix Spikes (MS)							
Perfluorooctyl Acids and Sulfonates by LC/MS/MS	EP231	۲	20	5.0	5.0	>	ALS QCS3 requirement
Matrix: WATER				Evaluation:	× = Quality Cor	itrol frequency no	Evaluation: x = Quality Control frequency not within specification ; < = Quality Control frequency within specification.
Quality Control Sample Type		ö	Count		Rate (%)		Quality Control Specification
Analytical Methods	Method	QC	Reaular	Actual	Expected	Evaluation	
Laboratory Duplicates (DUP)							
PFOS and PFOA	EP231	٢	-	100.0	10.0	>	NEPM 1999 Schedule B(3) and ALS QCS3 requirement
Laboratory Control Samples (LCS)							
PFOS and PFOA	EP231	٢	-	100.0	5.0	>	NEPM 1999 Schedule B(3) and ALS QCS3 requirement
Method Blanks (MB)							
PFOS and PFOA	EP231	-	-	100.0	5.0	>	NEPM 1999 Schedule B(3) and ALS QCS3 requirement
Matrix Spikes (MS)							
PFOS and PFOA	EP231	+	-	100.0	5.0	>	ALS QCS3 requirement



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## **Brief Method Summaries**

The analytical procedures used by the Environmental Division have been developed from established internationally recognized procedures such as those published by the US EPA, APHA, AS and NEPM. In house developed procedures are employed in the absence of documented standards or by client request. The following report provides brief descriptions of the analytical procedures employed for results reported in the Certificate of Analysis. Sources from which ALS methods have been developed are provided within the Method Descriptions.

Analytical Methods	Method	Matrix	Method Descriptions
Moisture Content	EA055-103	SOIL	A gravimetric procedure based on weight loss over a 12 hour drying period at 103-105 degrees C. This method is compliant with NEPM (2010 Draft) Schedule B(3) Section 7.1 and Table 1 (14 day holding time).
Perfluorooctyl Acids and Sulfonates by LC/MS/MS	EP231	SOIL	In-House. A portion of soil is soaked in sodium hydroxide followed by extraction with methanol. The extract is neutralised with HCI and an aliquot taken to dryness, made up in mobile phase. Analysis is by LC/MSMS, ESI Negative Mode using MRM.
PFOS and PFOA	EP231	WATER	In-house: Direct injection analysis of fresh and diluted saline waters. In order to meet standard reporting limits, saline waters may be adsorped onto a solid phase extraction medium, the salt washed out and the sample eluted for analysis. Analysis by LC-Electrospray-MS-MS, Negative Mode using MRM.
Preparation Methods	Method	Matrix	Method Descriptions
Sample Extraction for Perfluoroalkyl Compounds	EP231-PR	SOIL	In-House

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### Summary of Outliers

## **Outliers : Quality Control Samples**

The following report highlights outliers flagged in the Quality Control (QC) Report. Surrogate recovery limits are static and based on USEPA SW 846 or ALS-QWI/EN/38 (in the absence of specific USEPA limits). This report displays QC Outliers (breaches) only.

# Duplicates, Method Blanks, Laboratory Control Samples and Matrix Spikes

- For all matrices, no Method Blank value outliers occur.
- For all matrices, no Duplicate outliers occur.
- For all matrices, no Laboratory Control outliers occur.
- For all matrices, no Matrix Spike outliers occur.

### Regular Sample Surrogates

For all regular sample matrices, no surrogate recovery outliers occur.

## **Outliers : Analysis Holding Time Compliance**

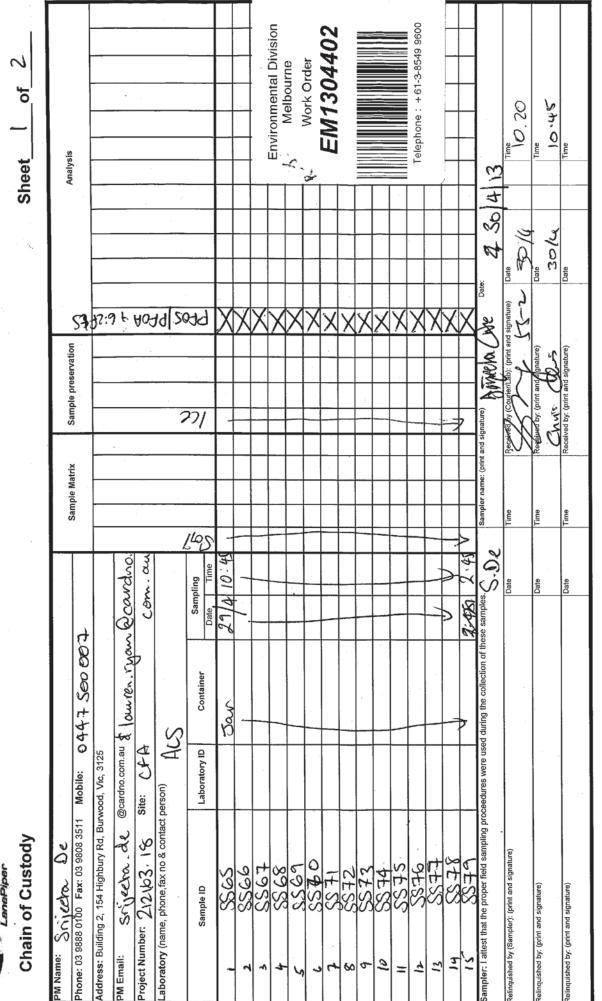
This report displays Holding Time breaches only. Only the respective Extraction / Preparation and/or Analysis component is/are displayed.

No Analysis Holding Time Outliers exist.

## **Outliers : Frequency of Quality Control Samples**

The following report highlights breaches in the Frequency of Quality Control Samples.

No Quality Control Sample Frequency Outliers exist.



Please circle Turn around time: (24 hour/48 hour/3 days(5 days))

Please supply results electronically in spreadsheet and ESDAT files.

Approved 3 Jan 2013 Revision 3

QF3.01 Chain of Custody.xltx

Printed 24/04/2013 Page 1 of 1

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Chain of Custody

Sheet 2 of 2

Phone: 03 9888 0100 Fax: 03 9808 3511	11 Mobile: 0447500007	67		Sample preservation	ş	Analysis
Address: Building 2, 154 Highbury Rd, Burwood, Vic, 3125	urwood, Vic, 3125					
PM Email: Srijeeja . Le	@cardno.com.au				2:9	
Project Number: 212163-18	Site: CFA				+4	
Laboratory (name, phone,fax no & contact person)	tct person) ALS				्रमिर	
Sample ID	Laboratory ID Container	Sampling Date Time	193	ונפ	HOI btoz	· · · · · · · · · · · · · · · · · · ·
					×	
16 SS80	Jaw	2014				
11 5581	1	<i>v</i>				
Is SS82	•				X	
Ì	Playisc bothe	6			X	
no gas	Vial		>>		X	
,	N					
21 OCI	Jar				X	
♦ ØC2					XJFORWARD	RO TO NGT
22 003					X	1
ou30/413 23 OCA	->	~	<i>≫</i>		X	
Sampler: I attest that the proper field sampling proceedures were used during the collection of these samples $S. W$	proceedures were used during the collection o	f these samples S. Q	Sampler name: (print and signature)	int and signature)	CAN Date: 30/4	
Relinquished by (Sampler): (print and signature)		Date	Time	Received by (Courier/Laby (print and signature)	signature) Date	Time
Relinquished by: (print and signature)		Date	Time	Received by: (print and signature)	Date 3 cb / C	Time Co.4.5
Relinquished by: (print and signature)		Date	Time	Received by: (print and signature)	Date	Time

Please supply results electronically in spreadsheet and ESDAT files Turn around time: (24 hour/48 hour/3 dayp/5 days)

Please circle

Revision 3 Approved 3 Jan 2013

QF3.01 Chain of Custody.xltx

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Melbourne 3-5 Kingston Town Close Oakleigh VIC 3166 Dankeigh XIC 3166 NATA # 1261 Site # 1254 & 14271 web : www.mgtlabmark.com.au ABN – 50 005 085 521 e.mail : enviro@mgtlabmark.com.au

**Brisbane** 1/21 Smallwood Place Murarie QLD 4172 Phone: +-61 7 3902 4600 NATA # 1261 Site # 20794 Sydney Unit F6, Building F 16 Mars Road Lane Cove West NSW 2096 Phone : +61 2 9900 8400 NATA # 1261 Sile # 18217

Company Name: Address:	Cardno Lane Piper Pty Ltd Building 2, 154 Highbury Road Burwood VIC 3125		Order No.: Report #: Phone: Fax:	377489 9888 0100 9808 3511	Received: Due: Priority: Contact Name:	Apr 30, 2013 2:13 PM May 7, 2013 5 Day Srijeeta De	
Client Job No.:	CFA 212163.18				Eurofins   mgt C	Eurofins   mgt Client Manager: Natalie Krasselt	
	Sample Detail	PFOS/PFOA					l
Laboratory where analysis is conducted	ialysis is conducted						
<b>Melbourne Laborator</b>	Melbourne Laboratory - NATA Site # 1254 & 14271						

 $\times$ 

Matrix

Sampling Time

Sample Date

**External Laboratory** Sample ID Apr 29, 2013

QC2

Sydney Laboratory - NATA Site # 18217 Brisbane Laboratory - NATA Site # 20794

×

M13-My01089 LAB ID

Soil

Date Reported:May 17, 2013



### mgt

### Eurofins | mgt Internal Quality Control Review and Glossary

### General

- 1. Laboratory QC results for Method Blanks, Duplicates, Matrix Spikes, and Laboratory Control Samples are included in this QC report where applicable. Additional QC data may be available on request.
- 2. All soil results are reported on a dry basis, unless otherwise stated.
- 3. Actual PQLs are matrix dependant. Quoted PQLs may be raised where sample extracts are diluted due to interferences.
- 4. Results are uncorrected for matrix spikes or surrogate recoveries.
- 5. SVOC analysis on waters are performed on homogenised, unfiltered samples, unless noted otherwise.
- 6. Samples were analysed on an 'as received' basis. 7. This report replaces any interim results previously issued.

### **Holding Times**

Please refer to 'Sample Preservation and Container Guide' for holding times (QS3001).

For samples received on the last day of holding time, notification of testing requirements should have been received at least 6 hours prior to sample receipt deadlines as stated on the Sample Receipt Acknowledgment.

If the Laboratory did not receive the information in the required timeframe, and regardless of any other integrity issues, suitably qualified results may still be reported. Holding times apply from the date of sampling, therefore compliance to these may be outside the laboratory's control.

\*\*NOTE: pH duplicates are reported as a range NOT as RPD

### UNITS

mg/kg: milligrams per Kilogram	mg/l: milligrams per litre
ug/l: micrograms per litre	ppm: Parts per million
ppb: Parts per billion	%: Percentage
org/100ml: Organisms per 100 millilitres	NTU: Units
MPN/100ml · Most Probable Number of organisms per 100 millilitres	

### TERMS

Dry	Where a moisture has been determined on a solid sample the result is expressed on a dry basis.
LOR	Limit of Reporting.
SPIKE	Addition of the analyte to the sample and reported as percentage recovery.
RPD	Relative Percent Difference between two Duplicate pieces of analysis.
LCS	Laboratory Control Sample - reported as percent recovery
CRM	Certified Reference Material - reported as percent recovery
Method Blank	In the case of solid samples these are performed on laboratory certified clean sands.
	In the case of water samples these are performed on de-ionised water.
Surr - Surrogate	The addition of a like compound to the analyte target and reported as percentage recovery.
Duplicate	A second piece of analysis from the same sample and reported in the same units as the result to show comparison.
Batch Duplicate	A second piece of analysis from a sample outside of the clients batch of samples but run within the laboratory batch of analysis.
Batch SPIKE	Spike recovery reported on a sample from outside of the clients batch of samples but run within the laboratory batch of analysis.
USEPA	United States Environment Protection Authority
APHA	American Public Health Association
ASLP	Australian Standard Leaching Procedure (AS4439.3)
TCLP	Toxicity Characteristic Leaching Procedure
COC	Chain of Custody
SRA	Sample Receipt Advice
CP	Client Parent - QC was performed on samples pertaining to this report
NCP	Non-Client Parent - QC performed on samples not pertaining to this report, QC is representative of the sequence or batch that client samples were analysed within

### **QC - ACCEPTANCE CRITERIA**

RPD Duplicates: Global RPD Duplicates Acceptance Criteria is 30% however the following acceptance guidelines are equally applicable:

Results <10 times the LOR : No Limit

Results between 10-20 times the LOR : RPD must lie between 0-50%

Results >20 times the LOR : RPD must lie between 0-30%

Surrogate Recoveries : Recoveries must lie between 50-150% - Phenols 20-130%.

### QC DATA GENERAL COMMENTS

- 1. Where a result is reported as a less than (<), higher than the nominated LOR, this is due to either matrix interference, extract dilution required due to interferences or contaminant levels within the sample, high moisture content or insufficient sample provided.
- 2. Duplicate data shown within this report that states the word "BATCH" is a Batch Duplicate from outside of your sample batch, but within the laboratory sample batch at a 1:10 ratio. The Parent and Duplicate data shown is not data from your samples.
- 3. Organochlorine Pesticide analysis where reporting LCS data, Toxophene & Chlordane are not added to the LCS.
- 4. Organochlorine Pesticide analysis where reporting Spike data, Toxophene is not added to the Spike.
- 5. Total Recoverable Hydrocarbons where reporting Spike & LCS data, a single spike of commercial Hydrocarbon products in the range of C12-C30 is added and it's Total Recovery is reported in the C10-C14 cell of the Report.
- 6. pH and Free Chlorine analysed in the laboratory Analysis on this test must begin within 30 minutes of sampling. Therefore laboratory analysis is unlikely to be completed within holding time. Analysis will begin as soon as possible after sample receipt.
- 7. Recovery Data (Spikes & Surrogates) where chromatographic interference does not allow the determination of Recovery the term "INT" appears against that analyte.
- 8. Polychlorinated Biphenyls are spiked only using Arochlor 1260 in Matrix Spikes and LCS's.
- 9. For Matrix Spikes and LCS results a dash " -" in the report means that the specific analyte was not added to the QC sample.
- 10. Duplicate RPD's are calculated from raw analytical data thus it is possible to have two sets of data.



### mgt

### Comments

NB: PFOS/PFOA analysis subcontracted to eurofins|GfA Lab Service, reference number AR-13-GF-011769-01, DAkkS accreditation number D-PL-14629-01-00.

N/A Yes Yes Yes Yes Yes

Sample Integrity
Custody Seals Intact (if used)
Attempt to Chill was evident
Sample correctly preserved
Organic samples had Teflon liners
Sample containers for volatile analysis received with minimal headspace
Samples received within HoldingTime
Some samples have been subcontracted

### Authorised By

Natalie Krasselt

Client Services

**Glenn Jackson** 

Laboratory Manager

Final report - this Report replaces any previously issued Report

- Indicates Not Requested

\* Indicates NATA accreditation does not cover the performance of this service

Uncertainty data is available on request

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# GfA Lab Service

Eurofins GfA Lab Service GmbH Otto-Hahn-Str. 22 D-48161 Münster GERMANY

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Eurofins GfA Lab Service GmbH · Otto-Hahn-Str.22 · D-48161 Münster

Mgt-LabMark Ltd attn. Results 2-5 Kingston Town Close Vic 3166 Oakleigh **AUSTRALIEN** 

Person	in	charg
ASM		

e Mr. J. Fuchs Mr. B. Homburg

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Report date 16.05.2013

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# Analytical report AR-13-GF-011769-01

Soil QC2

## Sample Code 710-2013-09129001

08.05.2013

01.05.2013

16.05.2013

M13-My01089

glass with screw closure

FedEx

377489

cooled

1

Tammy Lakeland

Reference

Sample sender **Reception date time** Transport by Client Purchase order nr. Purchase order date **Client sample code** Packaging Number of containers **Reception temperature End analysis** 

**Test results** 

CYP07 Method dry res	dry matter (°) (#) Internal method, produce dry matter of idue	original sample 91.7	%
<b>GF06J</b> Method	PFC (10 + H4PFOS) ~ environment (°) (# Internal method, LC-MS/MS	)	
	rooctane sulfonate (PFOS)	18.0	µg/kg dm
Perfluo	rooctanoic acid (PFOA)	< 2.2	µg/kg dm
total PF	FOS / PFOA excl. LOQ	18.0	µg/kg dm
total PF	FOS / PFOA incl. LOQ	20.2	µg/kg dm
Perfluo	rbutansulfonate (PFBS)	< 3.3	µg/kg dm
Perfluo	robutanoic acid (PFBA)	< 2.2	µg/kg dm
Perfluo	ropentane acid (PFPeA)	< 2.2	µg/kg dm
Perfluo	rohexane sulfonate (PFHxS)	< 3.3	µg/kg dm
Perfluo	rohexanoic acid (PFHxA)	< 2.2	µg/kg dm
Perfluo	rheptanoic acid (PFHpA)	< 2.2	µg/kg dm
	refer exclusively to the checked samples.		Durch die Deutsche Akkreditierungsstel

The results of examination refer exclusively to the checked samples. Duplicates - even in parts - must be authorized by the test laboratory in written form. Eurofins GfA Lab Service GmbH - Otto-Hahn-Str. 22 · D-48161 Münster Headquarters: Eurofins GfA Lab Service GmbH – Neuländer Kamp 1 D-21079 Hamburg HRB 115907 AG Hamburg General Manager: Dr. Christian Temme Our General Terms & Conditions of Sales are applicable. VAT No.: DE 275912372 Nord/LB • Bank code: 250 500 00 • Account No.: 199878695 • SWIFT-BIC: NOLADE2HXXX IBAN: DE37 2505 0000 0199 8786 95



DAkkS

stelle GmbH akkreditiertes Prüflaboratorium

DIN EN ISO/IEC 17025:2005 Aktreditierungsstelle D-PL-14629-01-00 Die Akkreditierung gilt für die in der Urkunde aufgeführten Prüfverfahren

Page 2/2



GfA Lab Service

Perfluorononanoic acid (PFNA)	< 2.2	µg/kg dm
Perfluordecanoic acid (PFDA)	< 2.2	µg/kg dm
6:2 Fluorotelomer sulfonate (FTS)	< 3.3	µg/kg dm
total PFC compounds excl. LOQ	18.0	µg/kg dm
total PFC compounds incl. LOQ	43.2	µg/kg dm

(°) = The test was performed at the site Hamburg.

(#) = Eurofins GfA Lab Service Gmbh (Hamburg) is accredited for this test.

< - Concentration below the indicated limit of quantification (LOQ)

This electronically generated test report has been checked and approved. It is also valid without signature.

Joachim Fuchs (Analytical Services Manager)

The results of examination refer exclusively to the checked samples. Duplicates - even in parts - must be authorized by the test laboratory in written form. Eurofins GfA Lab Service GmbH - Otto-Hahn-Str.22 · D-48161 Münster Headquarters: Eurofins GfA Lab Service GmbH – Neuländer Kamp 1 D-21079 Hamburg HRB 115907 AG Hamburg General Manager: Dr. Christian Temme Our General Terms & Conditions of Sales are applicable. VAT No. : DE 275912372 Nord/L B - Bank code: : 250 500 00 • Account No.: : 199878695 • SWIFT-BIC: NOLADE2HXXX IBAN: DE37 2505 0000 0199 8786 95



Durch die Deutsche Akkreditierungsstelle GmbH akkreditiertes Prüflaboratorium

LKS DIN EN ISO/IEC 17025:2005 Deutsche Abzeditierungsstelle DPU-14629-01-00 Die Akkreditierung gilt für die in der Urkunde aufgeführten Prüfverfahren



Cardno Lane Piper Pty Ltd Building 2, 154 Highbury Road Burwood VIC 3125



# Certificate of Analysis

NATA Accredited Accreditation Number 1261 Site Number 1254

Accredited for compliance with ISO/IEC 17025. The results of the tests, calibrations and/or measurements included in this document are traceable to Australian/national standards.

Atte	nti	on	:

Srijeeta De 377489-S

Report **Client Reference** Received Date

CFA 212163.18 Apr 30, 2013

Client Sample ID Sample Matrix Eurofins   mgt Sample No. Date Sampled Test/Reference	LOR	Unit	QC2 Soil M13-My01089 Apr 29, 2013
PFOS/PFOA			see attached

mgt



mgt

#### Sample History

Where samples are submitted/analysed over several days, the last date of extraction and analysis is reported. A recent review of our LIMS has resulted in the correction or clarification of some method identifications. Due to this, some of the method reference information on reports has changed. However, no substantive change has been made to our laboratory methods, and as such there is no change in the validity of current or previous results (regarding both quality and NATA accreditation).

Description

**Testing Site** 

Extracted

Holding Time



ABNO-50 005 085 521 e.mail : enviro@mgtlabmark.com.au web: www.mgtlabmark.com.au NATA #1261 e.mail : enviro@mgtlabmark.com.au web : www.mgtlabmark.com.au Site #1254 8,14271 NATA #1271 NAT

 Sydney
 Brisbane

 Unit F6, Building F
 1/21 Smallwood Place

 Unit F6, Building F
 1/21 Smallwood Place

 Inf Mars Road
 Murame QLD 4172

 Lane Cove Wers NSW 2066
 Phone : +617 3902 4600

 NATA # 1261 29900 8400
 NATA # 1261 Site # 20794

Eurofins | mgt Client Manager: Natalie Krasselt Apr 30, 2013 2:13 PM May 7, 2013 5 Day Srijeeta De Received: Due: Priority: Contact Name: 9888 0100 9808 3511 377489 Order No.: Report #: Phone: Fax: Cardno Lane Piper Pty Ltd Building 2, 154 Highbury Road Burwood VIC 3125 CFA 212163.18 Company Name: Address: Client Job No.:

PFOS/PFOA					×		×
% Moisture		×					×
						LAB ID	M13-My01089
		271				Matrix	Soil
Sample Detail	onducted	site # 1254 & 14	# 18217	Site # 18217 \ Site # 20794		Sampling Time	
	Laboratory where analysis is conducted	Melbourne Laboratory - NATA Site # 1254 & 14271	Sydney Laboratory - NATA Site # 18217	Brisbane Laboratory - NATA Site # 20794	itory	Sample Date	Apr 29, 2013
	Laboratory whe	Melbourne Labo	Sydney Laborat	Brisbane Labor	External Laboratory	Sample ID	QC2



mgt

ABN - 50 005 085 521 e.mail

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 Site # 1254 & 14271
 Site # 1254 & 14271

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Brisbane 1/21 Smallwood Place Murarrie QLD 4172 Phone : +61 7 3902 4600 NATA # 1261 Site # 20794

# Sample Receipt Advice

Company name:	Cardno Lane Piper Pty Ltd
Contact name:	Srijeeta De
Client job number:	CFA 212163.18
COC number:	Not provided
Turn around time:	5 Day
Date/Time received:	Apr 30, 2013 2:13 PM
Eurofins   mgt reference:	377489

#### Sample information

- A detailed list of analytes logged into our LIMS, is included in the attached summary table.
- a All samples have been received as described on the above COC.
- @ COC has been completed correctly.
- @ Attempt to chill was evident.
- @ Appropriately preserved sample containers have been used.
- @ All samples were received in good condition.
- Samples have been provided with adequate time to commence analysis in accordance with the relevant holding times.
- @ Organic samples had Teflon liners.
- : ¥:: Custody Seals intact (if used).

## **Contact notes**

If you have any questions with respect to these samples please contact:

Natalie Krasselt on Phone : (+61) (3) 8564 5000 or by e.mail: Natalie.Krasselt@mgtlabmark.com.au

Results will be delivered electronically via e.mail to Srijeeta De - srijeeta.de@lanepiper.com.au.

## Eurofins | mgt Sample Receipt



Environmental Laboratory N Air Analysis S Water Analysis T Soil Contamination Analysis C

NATA Accreditation Stack Emission Sampling & Analysis Trade Waste Sampling & Analysis Groundwater Sampling & Analysis



38 Years of Environmental Analysis & Experience

Chain of Custody				Ī	
РМ Name: 5. 0. Phone: 03 9888 0100 Fax: 03 9808 3511 Моbile: 0447.	0447500007	Sample Matrix	Matrix Sample preservation		Analysis
Address: Building 2, 154 Highbury Rd, Burwood, Vic, 3125 PM Email: Srijeera ・ んを @cardno.com.au Project Number: 21としる・18 Site: CFA Laboratory (name, phone,fax no & contact person) ALS				PPOA+6:2F	
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	Jar			X	
				X-JFORWARD	10 14/1
OC4	7	->		X	
Sampler: I attest that the proper field sampling proceedures were used during the collection of these samples $\lesssim \Omega$	the collection of these samples		Sampler name: (print and signature)	Arring a CAU Date: 30/4/13	
Relinquished by (Sampler): (print and signature)		Date	Received by (Courier/Laby (print and signature)	Date	Time
Relinquished by: (print and signature)		Date Time	Received by: (print and signature)	5 Date 30/4	Time [0.45
Relinquished by: (print and signature)		Date Time	Received by: (print and signature)	Date	Time 2:13 PM
Please supply results electronically in spreadsheet and ESDAT files.	(		+	0	botters t 0
Turn around time: (24 hour/48 hour/3 davs/5 davs)	davs) Please circle	<u>a</u>		Keper	1 Professor

QF3.01 Chain of Custody.xltx

Revision 3 Approved 3 Jan 2013

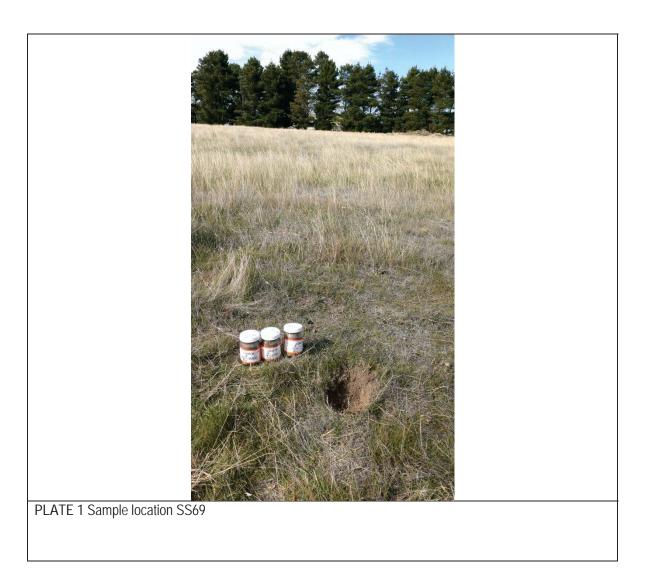
Printed 22/04/2013

# HUMAN HEALTH RISK ASSESSMENT – FISKVILLE COMMUNITY

# 4549 GEELONG-BALLAN RD, FISKVILLE VICTORIA

# **APPENDIX D – ATTACHMENT C**

# SOIL SAMPLING AWAY FROM TRAINING AREAS









# Appendix E

# Fish Sampling and QA/QC



# HUMAN HEALTH RISK ASSESSMENT – FISKVILLE COMMUNITY

# 4549 GEELONG-BALLAN RD, FISKVILLE VICTORIA

# **APPENDIX E**

# FISH QA/QC DATA REVIEW

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# APPENDIX E - FISH QA/QC DATA REVIEW

# **1** INTRODUCTION

This QA/QC summary is intended to provide a description of the laboratory analysis data for the biological tissue analysis conducted by Cardno Ecology Lab, Sydney NSW (Cardno Eco) at CFA Fiskville Training College, Fiskville Vic (the "Site"). The field work was conducted as per proposal reference 212163.10Proposal01.1 (dated 23 November 2012) under the instructions of Cardno Eco. This summary does not have nor provides any discussions with regards to results or corresponding criteria for the rabbit data as these are addressed in the main body of the report.

This summary collates the laboratory results and records for a review of the whole data quality as part of the assessment of fish tissue testing including:

- 1. Total number of samples;
- 2. Laboratory QA / QC review;
- 3. Surrogate recovery; and
- 4. Statistical summary of % surrogate recovery in muscle tissue.

## **1.1 Sample Locations**

The sampling field event, sample preservation, dissection and biometric data collection was conducted by Cardno Eco and not discussed in this review.

Sample locations within the surface water bodies are identified as:

- **Dam 3 –** (Results reported in certificate nos. DAU13\_37, DAU13\_038 and DAU13\_039);
  - CEL22, CEL23, CEL24, CEL25, CEL26, CEL27, CEL28, CEL29, CEL30.
- Lake Fiskville (*Results reported in certificate nos. DAU13\_016, DAU13\_017, DAU13\_037, DAU13\_038, DAU13\_039, DAU13\_116, DAU13\_152, 134672*);
  - CEL01, CEL02, CEL03, CEL03A, CEL04, CEL05, CEL06, CEL07, CEL08, CEL09, CEL10, CEL11, CEL12, CEL13, CEL14, CEL15, CEL16, CEL17, CEL18, CEL19, CEL20, CEL21; and
  - PF M5A/B, PF M6A/B, PFM7, PF M8A/B, PF M9A/B, PF M10A/B, PF M11A/B, PF M12A/B, PFM13, PF M14A/B, PFM15, PFM16, PFM17, PFM18, PFM19, PFM20, PFM21
- **Moorabool River (Site J), downstream from the Site –** (Results reported in certificate nos. DAU13\_116, DAU13\_061);
  - CEL31, CEL32, CEL33, CEL34, CEL022; and
- **Moorabool River, upstream of the Site –** (*Results reported in certificate nos. DAU13\_117, DAU13\_118, DAU13\_119*).
  - CEL035, CEL037, CEL039, CEL041, CEL043, CEL045, CEL047, CEL049, CEL064, CEL072, CEL076, CEL053, CEL055, CEL057, CEL094, CEL096.

The range of PFC concentrations reported for the different sample matrix or species are not discussed in the context of this summary..



## **1.2 Laboratory Analysis**

The samples were analysed by two laboratories as follows:

- National Measurement Institute (NMI), Sydney NSW, was the primary laboratory; and
- Asure Quality (AQ), Wellington NZ, was the secondary laboratory for Quality Control (QC).

The total analyses conducted are:

- NMI analysed a total of 60 samples for PFCs and metals, not including matrix spike, blank or surrogates; and
- AQ analysed a total of 8 inter-laboratory (i.e. fish muscle) split samples for PFCs only.

The corresponding number of samples analysed from each sample location, noted in Section 1.1, is provided in Table 1-1, and the data collated with corresponding sample ID, laboratory report and sample matrix are provided in Table A1, Attachment A..

		Number of samples analysed								
	Dam 3	Lake Fiskville	Moorabool Downstream	Moorabool Upstream						
Total	9	44	5	16						
Note:										

 Table 1-1: Summary of Sample Numbers and Analysis for Aquatic Species

1- QA/QC analysis were conducted and noted in Table 1A, Attachment A.

The analytical suite was for the Contaminant of Potential Concern (CoPC) taking into account the extended Perfluoro Compounds (PFCs) that are present in firefighting foams or breakdown products. The main PFCs analysed by both laboratories and included in this review were: PFPeA, PFHxS, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFDS, PFUdA, PFDoA, PFOS, 6:2 FtS and 8:2 FtS.

Copies of the corresponding laboratory reports and sample receipt records are included in Attachment B. The Quality Assurance and Quality Control (QA/QC) for the data analysis program is discussed in Section 3. Tabulated data for all laboratory results is provided in Attachment A.

# 2 AQUATIC BIOTA SAMPLING

The scope and method of the sampling event was prepared by Cardno Eco. The samples were collected were placed on ice and transported to Sydney, NSW. The dissection and biometric measurements was conducted at Cardno Eco laboratory in Sydney. Samples were weighed, labelled and frozen. A summary of sample type and matrix collated by Cardno Eco is provided in Table A1, Attachment A.

The blind inter-laboratory analysis was conducted from samples taken from either side of Redfin Perch. Then the two muscle tissue samples were labelled "A" and "B", shown in Table 3-1.



#### 3 QUALITY ASSURANCE AND QUALITY CONTROL REVIEW

## 3.1 Intra-Laboratory Analysis - NMI

NMI conducted a total of six internal duplicate assessments to assess the intra-laboratory reproducibility of the analysis. The duplicate samples are analysed concurrently with the parent sample. The Relative Percentage Difference (%RPD) calculated from the parent samples (i.e. CEL09 and CEL09D respectively) are provided in Table 3-1.

ID	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUdA	PFDoA	PFOS
CEL09	<10	4.73	16.8	<9	<40	99.6	293	16.1	122000
CEL09D <sup>1</sup>	<10	6.42	8.32	<5	<20	93.2	308	16.6	128000
%RPD	<loq< td=""><td>-30</td><td>67</td><td><loq< td=""><td><loq< td=""><td>7.0</td><td>-5.0</td><td>-3.0</td><td>-5.0</td></loq<></td></loq<></td></loq<>	-30	67	<loq< td=""><td><loq< td=""><td>7.0</td><td>-5.0</td><td>-3.0</td><td>-5.0</td></loq<></td></loq<>	<loq< td=""><td>7.0</td><td>-5.0</td><td>-3.0</td><td>-5.0</td></loq<>	7.0	-5.0	-3.0	-5.0
CEL04	-	-	<2	<2	<2	11.6	42.3	3.07	22300
CEL04D	-	-	<2	<2	<2	10.7	43.6	3.23	23000
%RPD	N/A	N/A	<loq< td=""><td><loq< td=""><td><loq< td=""><td>8.0</td><td>-3.0</td><td>-5.1</td><td>-3.0</td></loq<></td></loq<></td></loq<>	<loq< td=""><td><loq< td=""><td>8.0</td><td>-3.0</td><td>-5.1</td><td>-3.0</td></loq<></td></loq<>	<loq< td=""><td>8.0</td><td>-3.0</td><td>-5.1</td><td>-3.0</td></loq<>	8.0	-3.0	-5.1	-3.0
CEL25	3.39	2.8	<2	22.5	2.11	<2	2.56	3.97	3000
CEL25D	4.3	4.8	<2	38.8	4.1	2.66	5.06	5.99	3800
%RPD	27	-53	<loq< td=""><td>-53</td><td>64</td><td><loq< td=""><td>-66</td><td>-40</td><td>-23</td></loq<></td></loq<>	-53	64	<loq< td=""><td>-66</td><td>-40</td><td>-23</td></loq<>	-66	-40	-23
CEL23	5.3	10	8.53	8.96	4	20.4	46.2	25.9	260000
CEL23D	<5	9.84	7.77	8.25	3.89	17.1	39.6	23.4	280000
%RPD	<loq< td=""><td>2.0</td><td>9.0</td><td>8.2</td><td>2.7</td><td>17.6</td><td>15</td><td>10</td><td>-7.0</td></loq<>	2.0	9.0	8.2	2.7	17.6	15	10	-7.0
CEL28	14	26	6.25	11.5	<2	<2	<2	<2	6000
CEL28D	14	23	5.69	9.77	<2	<2	<2	<2	5000
%RPD	0	12	9.0	16	<loq< td=""><td><loq< td=""><td><loq< td=""><td><loq< td=""><td>18</td></loq<></td></loq<></td></loq<></td></loq<>	<loq< td=""><td><loq< td=""><td><loq< td=""><td>18</td></loq<></td></loq<></td></loq<>	<loq< td=""><td><loq< td=""><td>18</td></loq<></td></loq<>	<loq< td=""><td>18</td></loq<>	18
CEL32	<5	<5	<5	<5	<2	<2	<2	<2	25
CEL32D	<5	<5	<5	<5	<7	<2	<2	<2	24
%RPD	<loq< td=""><td><loq< td=""><td><loq< td=""><td><loq< td=""><td><loq< td=""><td><loq< td=""><td><loq< td=""><td><loq< td=""><td>4.0</td></loq<></td></loq<></td></loq<></td></loq<></td></loq<></td></loq<></td></loq<></td></loq<>	<loq< td=""><td><loq< td=""><td><loq< td=""><td><loq< td=""><td><loq< td=""><td><loq< td=""><td><loq< td=""><td>4.0</td></loq<></td></loq<></td></loq<></td></loq<></td></loq<></td></loq<></td></loq<>	<loq< td=""><td><loq< td=""><td><loq< td=""><td><loq< td=""><td><loq< td=""><td><loq< td=""><td>4.0</td></loq<></td></loq<></td></loq<></td></loq<></td></loq<></td></loq<>	<loq< td=""><td><loq< td=""><td><loq< td=""><td><loq< td=""><td><loq< td=""><td>4.0</td></loq<></td></loq<></td></loq<></td></loq<></td></loq<>	<loq< td=""><td><loq< td=""><td><loq< td=""><td><loq< td=""><td>4.0</td></loq<></td></loq<></td></loq<></td></loq<>	<loq< td=""><td><loq< td=""><td><loq< td=""><td>4.0</td></loq<></td></loq<></td></loq<>	<loq< td=""><td><loq< td=""><td>4.0</td></loq<></td></loq<>	<loq< td=""><td>4.0</td></loq<>	4.0

#### Table 3-1: %RPD Calculation for Intra-laboratory Assessment – NMI

3. N/A – not applicable.

The intra-laboratory assessment showed acceptable reproducibility with only four analytes (i.e. PFHxA, PFHpA PFNA and PFUdA) exceeding %RPD of 50%. Where compounds reported below the laboratory limit of reporting (<LOR), no %RPD was calculated.

## 3.2 Spiked Recovery - NMI

NMI conducted a total of eight spiked sample assessment as follows:

1. Sample CEL09 and CEL09D, spiked CEL09S (Certificate No. DAU13 016) was spiked with an internal standard with concentration of 104 ng/g for PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUdA, PFDoA, PFOS;



- 2. Sample CEL04 and CEL04D, spiked CEL04S (Certificate No. DAU13\_037) was spiked with an internal standard with concentration of 44 ng/g for PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFDA, PFDA, PFOS;
- Sample CEL25 and CEL25D, spiked CEL25S (Certificate No. DAU13\_016) was spiked with an internal standard with concentration of 45 ng/g for PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUA, PFDoA, PFOS;
- 4. Sample CEL23 and CEL23D, spiked CEL23S (Certificate No. DAU13\_038) was spiked with an internal standard with concentration of 44 ng/g for PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFDA, PFDA, PFDA, PFOS;
- 5. Sample CEL28and CEL28D, spiked CEL28S (Certificate No. DAU13\_039) was spiked with an internal standard with concentration of 45 ng/g for PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFDA, PFDoA, PFOS;
- 6. Sample CEL32 and CEL32D, spiked CEL32S (Certificate No. DAU13\_061) was spiked with an internal standard with concentration of 97 ng/g for PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUdA, PFDoA, PFOS, and 78 ng/g for 6:2 FtS;
- Sample PMF14A, spiked PMF14A S (Certificate No. DAU13\_117) was spiked with an internal standard with concentration of 21 ng/g for PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUA, PFDoA, PFOS, and 17 ng/g for 6:2 FtS; and
- 8. Sample CEL64, spiked CEL64S (Certificate No. DAU13\_119) was spiked with an internal standard with concentration of 21 ng/g for PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUdA, PFDoA, PFOS, and 17 ng/g for 6:2 FtS.

Table 3-2 provides a summary of the spiked sample calculations, % Recovery compared with primary and duplicate samples where applicable. Overall, the spiked analysis showed good reproducibility with either the primary or duplicate samples for the corresponding batches. However, some compounds highlighted with bold font in Table 3-2 exceeded 130% showing a potential bias for overestimating these compounds.

ID	PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUdA	PFDoA	PFOS	6:2 FtS
CEL09	-	172%	95%	282%	92%	69%	86%	98%	88%	101%	-
CEL09D	-	172%	93%	304%	94%	75%	89%	94%	88%	96%	-
CEL09S	-	187	103	341	100	85.7	175	389	106	123000	-
CEL04	-	-	-	113%	180%	136%	124%	121%	128%	106%	-
CEL04D	-	-	-	113%	180%	136%	126%	119%	127%	102%	-
CEL04S	-	-	-	51	81	61	69	104	60.2	23600	-
CEL25	-	91%	128%	117%	133%	137%	132%	139%	142%	72%	-
CEL25D	-	89%	122%	117%	107%	131%	128%	132%	137%	57%	-
CEL25S		44.0	61.0	54.0	89.9	64.4	60.9	65.9	69.7	2200	-
CEL23	-	81%	117%	255%	109%	111%	123%	119%	123%	92%	-
CEL23D	-	86%	117%	259%	110%	111%	129%	128%	128%	86%	-
CEL23S	-	40	63.1	134.0	57.7	53.3	79.1	107.0	86.2	240000	-
CEL28	-	114%	114%	119%	103%	118%	131%	125%	116%	76%	-
CEL28D	-	114%	119%	120%	106%	118%	131%	125%	116%	91%	-

## Table 3-2: Spiked Recovery Calculation – NMI



Privileged and Confidential Human Health Risk Assessment - Fiskville Community 4549 Geelong-Ballan Rd, Fiskville Victoria Ashurst

ID	PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUdA	PFDoA	PFOS	6:2 FtS
CEL28S)		67	81.0	60.8	58.0	54.5	60.3	57.4	53.2	4600	-
CEL32	84%	93%	99%	111%	92%	94%	90%	87%	99%	98%	99%
CEL32D	84%	93%	99%	110%	92%	92%	90%	87%	99%	98%	99%
CEL32S	86	93	99	110	92	92	88	85	97	119	78
PF M14A	-	-	-	-	-	-	136%	-	-	100%	99%
PFM14A S	29	31	32	41	27	29	39	-	-	7140	22
CEL064	127%	152%	162%	190%	119%	114%	143%	-	-	133%	103%
CEL064S	28	32	34	40	25	24	30	-	-	28	18

## 3.3 Inter-Laboratory Analysis – NMI and AQ

Eight samples, as shown in Table 3-3, were submitted to NMI and AQ as part of an interlaboratory assessment. The corresponding samples were taken from the same specimen (i.e. Redfin Perch) and labelled Sample A and Sample B as shown in Table 3-3.

Sample ID	Samp	ole Results	(ng/g)		% RPD	_
Sample ID	PFDA	PFOS	6:2 FtS	PFDA	PFOS	6:2 FtS
PF M5A	9	5,990	4.8	22	8.5	67
PF M5B	7.2	5,500	2.4	22	0.0	07
PF M6A	8.1	5,520	4.4	42	27	59
PF M6B	5.3	4,200	2.4	42	21	59
PF M8A	7.7	6,450	4.9	22	14	45
PF M8B	6.2	5,600	3.1	22	14	45
PF M9A	8.2	7,440	4.5	12	9.0	61
PF M9B	7.3	6,800	2.4	12	9.0	01
PF M10A	10	9,600	3.4	-9.0	9.0	6.0
PF M10B	11	8,800	3.2	-9.0	9.0	0.0
PF M11A	6.9	7,940	3.9	-16	18	100
PF M11B	8.1	6,600	1.3	-10	10	100
PF M12A	9.6	8,870	3.9	-30	-11.0	79
PF M12B	13	9,900	1.7	-30	-11.0	13
PF M14A	7.7	7,100	5.3	20	13	79
PF M14B	6.3	6,200	2.3	20	13	19

Cardno Lane Piper conducted a statistical summary of the %RPD for each corresponding sample within a single batch analysis for the 8 inter-laboratory samples. Note that the inter-



laboratory analysis was conducted for fish muscle samples from Lake Fiskville. The %RPD calculated are shown in Table A1, Attachment A.

The %RPD was calculated for the batch analysis for the Redfin Perch (muscle) for samples collected from Lake Fiskville only (Certificate numbers DAU13\_116 for NMI, and 134672 for AQ). The %RPD for PFOS for all 8 samples was less than 30%<sup>1</sup>. This is considered as a good and acceptable correlation between the primary sample and the inter-laboratory duplicate analysis of that sample. The %RPD for PFOA was less tha 50% for all samples, and it is considered as an acceptable result between the two laboratories. However, 6:2 FtS only reported one analysis less than 50% with the remainder of the analysis having a %RPD greater than 50%. This is considered not a reliable set of results for 6:2 FtS and it may be in part due to NMI reporting higher concentrations than AQ for most samples.

## 3.4 Surrogate Recovery – NMI

The results for the surrogate analysis for all samples provided by NMI are included in Table 1A, Attachment A. There are some recovery inconsistencies between batches, for the NMI reports. Table 3-4 provides a summary for all surrogate recovery analysis conducted by NMI and AQ.

	PFBA	PFHxA	PFOA	PFNA	PFDA	PFUdA	PFDoA	PFOS	6:2 FTS
Total No.	36	94	103	103	103	74	66	103	57
Average	27	95	68	45	63	111	93	47	72
Minimum	13	28	25	6	4	1	1	7	21
Lower Quartile	17	57	44	11	18	58	31	16	52
Median	19	75	67	38	47	86	76	33	70
Upper Quartile	23	91	88	75	94	121	99	80	90
Maximum	75	433	123	144	349	467	457	112	122

 Table 3-4: % Surrogate Recovery Summary for all Laboratory Data

The surrogate recoveries shown in the maximum values, Table 3-4, which have values up to 467% are from NMI's report batch DAU13\_016. These samples comprised of Redfin Perch liver tissues from Lake Fiskville and were among the first batch of samples to be analysed. However, the following comments are made with regards to sample ID CEL09 (Lab ID N12/034245) which had an internal laboratory duplicate analysis (ID for duplicate N12/034245DUP) and a matrix spike (ID for spike N12/034245SPK). The surrogate recovery for the primary, duplicate and spike for CEL09 is summarized in Table 3-5. The duplicate and spike samples have a lower surrogate recover than the primary sample for PFHxA, PFDA, PFUda and PFDoA. This inconsistency may be due in part to human error during the sample manipulation and set up.

 Table 3-5: % Surrogate Recover for Primary and Duplicate Sample – CEL09

	PFHxA	PFOA	PFNA	PFDA	PFUdA	PFDoA	PFOS
N12/034245	432.6	94	11	349	467	457	77

1 The Australian Standard AS4482.1 (2005) AS 4482.1-2005 *Guide to the investigation and sampling of sites with potentially contaminated soil - Non-volatile and semi-volatile* compounds recommends a %RPD range of 30 to 50% of mean concentration as an acceptance criteria for quality control samples.



	PFHxA	PFOA	PFNA	PFDA	PFUdA	PFDoA	PFOS
N12/034245DUP	96.2	37	10	65	93	98	18
N12/034245SPK <sup>1</sup>	62.4	26	11	55	74	75	15
Notes: 1- The spiked conc	entration w	as 104 ng/	/g.				

Table 3-6 shows varying levels of recoveries for different PFC compounds including an average and median values for the redfin muscle samples from Lake Fiskville only, and it includes the data provided by NMI and AQ.

Table	3-6: % S	Surrogate	Recove	r Summ	ary for	Redfin Pe	erch, Lake	e Fiskvil	le
	PFBA	PFHxA	PFOA	PFNA	PFDA	PFUdA	PFDoA	PFOS	6:2 FT
-1.81-	0	04	40	40	40	04	00	40	00

	PFBA	PFHxA	PFOA	PFNA	PFDA	PFUdA	PFDoA	PFOS	6:2 FTS
Total No.	9	31	40	40	40	31	23	40	30
Average	18	73	67	29	40	58	39	31	79
Minimum	13	40	26	6	6	4	2	8	25
Lower Quartile	16	62	54	8	20	46	13	12	62
Median	19	75	63	10	40	60	31	17	70
Upper Quartile	20	81	84	37	54	70	63	37	110
Maximum	23	107	100	106	112	95	90	101	122

Considering that this file note is summarizing the data for the analysis of muscle tissues for Redfin Perch only from Lake Fiskville, these samples were analysed in batches No.:

- DAU13 017 (Sample ID CEL1, 2, 3, 4, 5, 6 and 7);
- DAU13\_116 (Sample ID PFM5, 6, 8, 9, 10, 11, 12, 14 and CEL0222);
- DAU13\_152 (Sample ID PFM7, 13, 15, 16, 17, 18, 19, 20 and 21): and
- DAU13\_153 (Sample ID CEL04 and 06 repeat). •

The surrogate recovery data for AQ is within recommended range of 70 to 130%.

## 3.5 Laboratory QA/QC

Table 3-7 provides a summary of the QC program established by NMI and AQ. Internal laboratory blanks corresponding to a minimum of one blank per batch and summarized was conducted by both labs.

<sup>&</sup>lt;sup>2</sup> Sample ID CEL022 was collected from the Moorabool River, downstream and it is included here only due to batch completeness.



		QA/QC Ana	Ilysis	
Certificate No Lab	Blank	Spiked	Intra Laboratory Duplicate	Inter Laboratory Duplicate
DAU13_016 – NMI	1	1	1	0
DAU13_017 – NMI	1	1	1	0
DAU13_037 – NMI	1	1	1	0
DAU13_038 – NMI	1	1	1	0
DAU13_039 – NMI	1	1	1	0
DAU13_061 – NMI	1	1	1	0
DAU13_116 – NMI	1	0	0	8
DAU13_117 – NMI	0	1	0	0
DAU13_118 – NMI	1	0	0	0
DAU13_119 – NMI	0	1	0	0
134672 – AQ	1	0	0	8
Sum	9	8	6	16

## Table 3-7: Laboratory Quality Control

## 3.6 Summary of Aquatic Biota Results

A summary of the results is provided in Table 3-8, with analytes reporting greater than 50% detection rate highlighted with bold numbers.

	PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUdA	PFDoA	PFOS	6:2 FtS
Minimum	0.0	2.1	1.2	4.0	2.3	2.1	2.2	2.3	2.2	1.1	1.3
Median	< LOR	5.6	5.7	6.5	11.3	4.7	8.1	25.5	3.5	6650	3.4
Maximum	0.0	14	23	16.8	101	14	110	387	40	280000	5.3
Total	36	84	93	103	103	103	103	73	74	103	57
%detects	0	33	40	31	34	28	68	83	61	79	61

### Table 3-8: Summary of Aquatic Biota Analysis – Including all species

# 4 ATTACHMENTS

#### **Attachment A**

Table A1 - Summary %RPD and % for Primary Duplicate Recoveries

## Attachment B

Laboratory Reports

Cardno Lane Piper March 2014



3lanks Sum	AII < LOQ			0017 IIV	All < LOU					AII < LOQ						VI - 1 00	AILS LOQ					AII < LOQ					AII < 1.00					AII < LOQ										AII < LOQ	0017 IV	All < LOQ						ſ								AII < LOQ												
6:2 FtS																											<0.08	2	99	91	78	<02	2.4	4.4 2.4	4.9	3.1 4.5	2.4	3.2	3.9	3.9	5.3	2.3	<0.1	2.6	3.4 2.4	3.4	4.2	5.3 4.9	4.4	3.3	<0.1	<0.1 22	<0.1	<0.1	<0.1	<0.1	<0.1 <0.1	<0.1 <0.3	<0.1	18	<0.1		6:2 FtS 3.6	1.3	3.4	4.5 5.3	49	57 24	8	01/10
PFOS	<1 58200	122000	86000 128000	123000	12100	14900	13500	11200	23600	<100	2600	2000	3000	5200	5000 3800	2200	50000	30000	260000	260000 240000	280000	<80	1240	440 6000	6800	5000	4600	<10	8 28	33	119	<1	5500	5520 4200	6450	7440	6800	8800	7940 6600	8870 0000	7100	6200 <2.0	41	50.5 7600	7000 8300	6700	7300	5400 5400	5800	15000	- <del>-</del> -	<1 7140	r 1	<1 2.1	1.1	7	<1 2.3	4	2.6	28	22		PFOS 24082	1.1	6650	11875 280000	80	103	75	01.61
PEDoA	<0.4	16.1	23 18.8	106	2.17	3.48	221	2.9	323	40.1	<2	2.5	4.0	15.0	15.6 6.0	69.7	3.7	2.6 3.8	35.6	25.9 29.8	23.4	<1>	22	<2 <2	000	<2	532	~2	5 5	5	2× 97		<2.0	<2.0		0.2 >	<2.0	2.4	2.4	7.0	2.1	<2.0	4	<2	88	25	~2	<2 <2 <	<2 <	3.5													PFDoA 11.0	2.2	3.5	40.1	55	74	40 50	01.70
PFUdA	<0.3	293	363 263	389	24.7	40.1	27.8	27.5	104	<0.5 51.6	5.8	16.4	2.6	14.9	32.6	65.9	35.6	25.0	58.1	46.2 52.1	39.6	<1	2.6	80	98	78	57.4	44	99	91	85		18	14	00	N	23	35	26	42	42	24	4	28 5	18	24	20	14	15	9													16.2 56.3	2.3	25.5	42.2 387.0	55	73	54	00.70
1/g PFDA	<0.2	9 <del>.</del> 66	110	175	8.64	8.08	7.93	6.35	10.7	<0.6	2.4	2.2	84	4.9 8.9	7.7	609	12.3	6.3	19.6	20.4	17.1	<1	25	90	98	70	60.3	8	99	91	88	<0.5	7.2	8.1	7.7	8.2	7.3	2€	6.9 8.1	9.6	7.7	6.3	<0.5	7.6	5.1	4.8 6.2	6.5	5.4 4.8	4.3	12	<0.5	39 <0.5	<0.5	<0.5	0.5	0.5	<0.5 <0.5	<0.5	40.5 1	47 B	<0.5	s in ng/g	16.2	2.2	8.1	11.8	80	90 90	-92	00.78
esults in ng	<0.3	< 40	14	85.7	2 4 4	90	781	701	61	<0.5 10.8	6.7	83	2.1	8.6	4.5	64.4	6.4	2.3	4.7	42	3.9	<1>	98	80	80	78	54.5	8	99	91	92	<0.5 20.5	\$2.0	5°0	8	0.7 7	2:0	\$20	8.0	80	5.7	<2.0 <2.0	<0.5	<b>v</b> v	7	2	7	21	2	v v	0.5	29 29	<0.5	<0.5	9.9 9.9	0.5	<0.5 <0.5	<0.5 <0.5	0.5	24	<0.5	ry of Result	6.1 6.1	2.1	4.7	8.3	80	103	27	e/.07
PFOA	<0.5	883	14	7 8 1	2 0 1	40	701	701	81	<0.5 21.0	18.2	23.2	22.5 E2.5	101.0	19.4 28.8	89.9	4.5	23	11.3	9.0	8.3	<1	20	115	10.0	9.8	58.0	\$	8 V	ψĻ	62 6	<0.5	2.0	<0.5	0.5	<0.0>	20	2.0	40.5	<0.5 <>0.5	<0.5	<2.0 <2.0	<0.5	<ul><li>0.5</li><li></li></ul>	€.6 2.5	<0.5 20.5	\$0.5	<0.5	<0.5 20.5	0.5	0.5	<0.5 27	<0.5	<0.5	0.5	0.5	<0.5 <0.5	<ul><li>0.5</li><li>0.5</li></ul>	0.5	22 52	<0.5	Summa	20.1 20.1	2.3	11.3	21.0	80	103	32.8	r,
PEHDA	<0.6	16.8	15 8 33	341	2 4 4	40	701	701	51	<0.5 4.0	8	88	83	4.5	44	54.0	2.5	25	11.0	8.5 6.5	7.8	12	20	63	6.5	5.7	60.8	\$	<del>۵</del> ۵	ŝ	110	<0.5	<1.0	<0.5	<0.5	<0.5	<1.0	<1.0	<0.5	<0.5	<0.5	<1.0	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5 10.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	40.5	<0.5		PFHpA 7.8	4.0	6.5	8.9 16.8	80	103	29	97.10
PEHXA	< 0.6	4.73	5.5 6.42	103						<0.5	12	2.1	2.8	4.0	6.9	61.0	<0.4 <2	4	12.0	10.0 8.3	9.8	<0.9	5.6	32	23.0	23.0	81.0	\$	9 V	\$\$ Y	9 66	<0.5	<1.0	<0.5	<0.5	<0.5	<1.0	<1.0	<0.5	<0.5	<0.5	<1.0 <2.0	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5 32	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	20 20 20 20 20 20 20 20 20 20 20 20 20 2	<0.5		PEHxA 7.9	12	5.7	9.4 23.0	72	93 51	. B	40.78
PFP.eA	<0.9	<10	10,200	187						<0.5 5.9	3.8	3.4	3.4	0.0 4.6	11.0	44.0	<0.4	42	99	5.3 <5	<5	<0.7	2.8	Q 2	: <del>1</del> 2	14	67	\$	Q Q	ŵί	8	<0.5	202	<0.5	<0.5	<0.5	4.9	0.02	<0.5	<0.5	<0.5		<0.5	<ul><li>0.5</li></ul>	0.5	<0.5 20.5	<0.5	<0.5	<0.5	40°2	0.5	31	<0.5	<0.5	0.5	<0.5 0.5	0.5 0.5	<0.5 <0.5	<0.5 5 T	32 0.5	<0.5		۰.	2.1		11.0	64	51	25	20.20
PFBA																											ų	<10	<10 <10	<10	86	25	7	<2	<2	<2	ı q	7	2	<2	<2		<2							Ş	~2	29 <2	<2 <2	<2 <2	101	22	5 V	5 S	59	28	\$		<pre>&gt; LOR</pre>	0.0	< LOR	< LOR	300	88	;03	2.9
Certificate	DAU13 016	DAU13_016	DAU13 016	DAU13 016	DAU13 017	DAU13_017	DAU13 017	DAU13 017	DAU13_017	DAU13_037	DAU 13_037	DAU 13_037 DAU 13_037	DAU 13 037	DAU 13_037	DAU 13_037	DAU13 037	DAU 13 038	DAU13_038	DAU 13_038	DAU 13 038 DAU 13 038	DAU13_038	DAU 13 039	DAU 13 039	DAU13 039 DAU13 039	DAU 13 039	DAU 13 039	DAU 13_039 DAU 13_061	DAU 13_061	DAU13_061 DAU13_061	DAU13_061	DAU13_061	DAU13_116	134672	DAU13_116 134672	DAU13_116	DAU13_116	134672	134672	DAU13_116 134672	DAU13_116	DAU13_116	134672 134672	DAU13_116	DAU13_15	DAU13 15: DAU13 15:	DAU13 15	DAU13 15	DAU13_15.	DAU13_15	DAU13 15	DAU13 117	DAU13 117 DAU13 117	DAU13_117 DAU13_118	DAU13_118 DAU13_118	DAU13 118	DAU13_116	DAU13_118 DAU13_118	DAU13_118 DAU13_118	DAU13 115	DAU13 119	DAU13_119							e & Dup) anks)	()	
Samole Matrix	l iver	Liver	Liver			Muscle		Muscle		Simle Whole and	Single Whole ani	i lo	Single Whole and	Single Whole and			Composite whole	Composite whole	Composite whole	Composite whole Composite whole			Composite Whole Composite Whole	Composite Whold	Composite Whole			Muscle	Muscle	Muscle		Minorla	Muscle	Muscle Muscle	Muscle	Muscle	Muscle	Muscle	Muscle Muscle	Muscle	Muscle	Muscle	Composite whole		Muscle Muscle		Muscle	Muscle Muscle	Muscle	Muscle	Muscle	Muscle	Muscle		Muscle				Muscle	Muscle	Muscle						ank, Spike & Dup)	is (Inc. Blank, Spik n I.OO /Not inc. htt	ts (Not inc. spikes	de detecto ( i conalan eße
Sample Type	Bartin Parch	Redfin Perch	Redfin Perch				Redfin Perch	Redfin Perch		Yabby	Yabby	ter shrin		Yabby			Mosquito Fish	Mosquito Fish	Mosquito Fish	Mosquito Fish Mosquito Fish					Macrophyle			Trout	Trench Eel	Redfin		Dodfo Doroh	Redfin Perch	Redfin Perch Redfin Perch	Redfin Perch	Redfin Perch	Redfin Perch	Redfin Perch	Redfin Perch Redfin Perch	Redfin Perch Badfin Perch		Redfin Perch	Brown trout	Redfin Perch	Redfin Perch Redfin Perch	Redfin Perch	Redfin Perch	Redfin Perch Redfin Perch	Redfin Perch Devite Perch	Redfin Perch	Roach	Roach	Roach Roach	Roach Carp			Brown trout Muscle Tench Muscle	Short-finned eel	Short-finned eel	Short-Inned eel	Short-finned eel		Average	Minimum	Median	Upper Quartile Maximum	Total (Not Inc. Blar	Total No. Analys Analysis less tha	Number of Deter	Skillpre r el vurv
Sample Location	l aka Fielvilla	Lake Fiskvile	Lake Fiskvile			1.1	Lake Fiskvile	11		ake Fiskvile	Lake Fiskvile	Lake Fiskvile Lake Fiskvile	Dam 3 Dam 2	Dam 3	Lake Fiskvile		Lake Fiskvile	Lake Fiskvile	Dam 3	Dam 3 Dam 3					Dam 3			Moorabool Site (J	Moorabool Site (J Trench Moorabool Site (J Eel	Moorabool Site (J		I also Eiclevillo	Lake Fiskvile	Lake Fiskvile Lake Fiskvile	Lake Fiskvile	Lake Fiskvile	Lake Fiskvile		Lake Fiskvile Lake Fiskvile		Lake Fiskville		Moorabool	Lake Fiskvile Lake Fiskvile	Lake Fiskvile Lake Fiskvile			Lake Fiskvile Lake Fiskvile		Lake Fiskvile	Moorabool u/s	Moorabool u/s	Moorabool u/s Moorabool u/s					Moorabool u/s	Moorabool u/s	Moorabool U/S	Moorabool u/s									
CARDNO Ref.	CELOR	CEL09	CEL11		CEL01	CEL03	CEL05	CEL07		CF115	CEL16	CEL17 CEL18	CEL25	CEL27	CEL03A		CEL12	CEL13	CE122	CEL23 CEL24			CEL19 CEL20	CEL21 ICF1 28	CEL29	CELLAN			CEL32 CEL33			DC MCA	PF M5B	PF M6A PF M6B	PF M8A	PF M9A	PF M9B	PF M10B	PF M11A PF M11B	PF M12A DF M12B	PF M14A	PF M14B	CEL022	PFM7	PFM13 PFM15					CELO6	CEL037		CEL041 CEL043				CEL076 CEL053	CEL055	CEL057	CELUSH	CEL096				79	10 6	0 00	8 10	6	> 1:10
NMILRN	BLK L841 N12/0742344	N12033245	N12/034247 N12/034247 N12/034246N11D	N12/0345SPK (104 ng/g)	BLK L840 N12/034237	N12/034239	N12/2/0372440 N12/2/032241	N12/034242 N12/0342442 N12/034243	N12/034240DUP N12/034240SPK (44 ng/g)	BLK L846 N12/034251	N12/034252	N12/034253 N12/034254	N12/034261	N12/034262 N12/034263	N12/034267 N12/0342841011D	N12/034261SPK (45 ng/g)	BLK L845 N12/034248	N12/034249 N12/0342560	N12/034258	N12/034259 N12/034260	N12/034259DUP N12/0342565EbK (44 mole)	BLK L849	N12/034255 N12/034256	N12/034257 N12/034264	N12/034265 N12/034265	N12/034264DUP	N12/034264SPK (45 ng/g) RI K I 854	N13/006929	N13/006930 N13/006931	N13/006932	N13/006930SPK (97 ng/g, and 6:2 78 ng/g)	BLK L873	134672-1 DUP (Asure Quality)	N13/014203 134672-2 DUP (Asure Quality)	N13/014204	134672-3 U.D.P. (Asure Quality) N13/014205	134672.4 DUP (Asure Quality)	134672-5 DUP (Asure Quality)	N13/014207 134672-6 DUP (Asure Quality)	N13/014208 134672-7 DLIP (&sine Onelle))	13/01/2209 N13/01/209	134672-8 DUP 134672-8L	N13/014210	BLK L884 N13/017964	N13/017965 N13/017966	N13/017967	N13/017969	N13/017970 N13/017971	N13/017972	N12/034242Repeat	N13/014212	N13/014213 N13/014209SPK (21 ng/g, and 6:2 17 ng/g )	N13/014214 N13/014215	N13/014216 N13/014217	N1301422	N13/014220	N13/014221 N13/014222	N13/014223 BLK L874	N13/014224	N13/014225 N13/014219SPK (21 ng/g, and 6:2 17 ng/g )		-			Number of Samples (total)	Number of Blanks - NMI & AQ (Analysis) Number of Dunitertes - NMI (Analysis)	Number of Interlaboratory (Analysis)	Number of Spikes (Analysis) Total Number of Analysis NMI	Total Number of Analysis AQ 9	NMI עאיעט המנוט

Attachment A

Certificate		DAU13_016				DAU13_017		Ţ			DAU13_037					DAU13_038					DAU13_039				DAU13_061							DAU13_116 &	134672							DAU13_152			DA1142 4E2	DAU13_153	DAU13 117	1			DAL13 118				DAU13_119		_					_
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CARDNO Ref.	CEL08 CEL09	CEL10 CEL11		CEL01	CEL03	CEL05	CEL07		CEL15 CEL16	CEL17	CEL25	CEL27	CEL03A		CEL12 CEL13	CEL14 CEL22	CEL23 CEL24		CEL19	CEL20 SEL21	CEL28 CEL28	CEL30		CEL31	CEL33	CEL34		PF M5A PF M5B	PF M6A	PF M8A	PF M9A	PF M9B PF M10A	PF M10B	PF M11B	PF M12B	PF M14A PF M14B	CEL022	oFM7	PFM13 PFM15	PFM16 2FM17	PFM18	PFM19 PFM20	DEL04	CEL06 CEL06	CEL037	i u	шц	ūΞ	CEL049 CEL064	Ξ	CEL053 CEL055	2F1.057	CEL094	CEL096				79	9	
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ttachment A



**National Measurement Institute** 

	CERTIFICATE OF A	ANALYSIS # DAU	J13_016
Client	Cardno Ecology Lab	Job No.	CARD20/121218
	L9, 203 Pacific Highway, St I	Leonards	
	NSW, 2065	Sampled by	Client
		Date Sampled	not specified
Contact	Marcus Lincoln-Smith	Date Received	18-Dec-12
	The resu	ults relate only to the same	ole(s) tested.

Method	AUTL_07	Date Reported	22-Feb-2013

# **Details** The method is for determination of Perfluoroalkyl substances (PFASs) in biota samples by High Performance Liquid Chromatography tandem Mass Spectrometry (UPLC-MSMS). All results are corrected for labelled surrogates and are reported on a fresh weight basis.

Prior to extraction the sample is spiked with a range of isotopically labelled surrogate standards. Extraction is by organic solvent, with purification using activated silica gel. An aliquot of extract is injected onto the UPLC and detected using mass spectrometry.

Authorisation

Haw

Gavin Stevenson Manager Dioxin Analysis Unit

Dr Alan Yates Senior Analyst Dioxin Analysis Unit

	Sample Details : Job No. CARD20/121218								
Laboratory Reg. No.	Client Sample Ref.	Matrix	Description						
N12/034244	CEL08	Fish Livers	Freshwater. Dec 2012						
N12/034245	CEL09	Fish Livers	Freshwater. Dec 2012						
N12/034246	CEL10	Fish Livers	Freshwater. Dec 2012						
N12/034247	CEL11	Fish Livers	Freshwater. Dec 2012						
N12/034245DUP	Duplicate	Fish Livers	Duplicate Sample						
N12/034245SPK	Spike	Fish Livers	Spiked sample (104 ng/g)						
BLK L841	Lab Blank	Lab Blank	Lab Blank						

Project Details
Project Name

Fiskville Study

NA49913-034

Project Number

Кеу		
Analytes		Surrogate
PFPeA	Perfluoro-n-pentanoic acid	
PFHxA	Perfluoro-n-hexanoic acid	Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]hexanoic acid
PFHpA	Perfluoro-n-heptanoic acid	
PFOA	Perfluoro-n-octanoic acid	Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]octanoic acid
PFNA	Perfluoro-n-nonanoic acid	Perfluoro-n-[1,2,3,4,5- <sup>13</sup> C <sub>5</sub> ]nonanoic acid
PFDA	Perfluoro-n-decanoic acid	Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]decanoic acid
PFUdA	Perfluoro-n-undecanoic acid	Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]undecanoic acid
PFDoA	Perfluoro-n-dodecanoic acid	Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]dodecanoic acid
PFOS	Perfluoro-n-octanesulfonate	Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]octanesulfonate
Units & Abbreviations	3	
ng/g	nanograms per gram	
<	level less than limit of detection (LOD)	
Surrogate Recovery	percentage recovery for <sup>13</sup> C <sub>12</sub> labelled surrogate star	ndard
þ	Laboratory surrogate recovery outside normal accept	tance criteria (25 - 125%)

#### Laboratory Reg. No. N12/034244

Client Sample Ref. CEL08 Matrix Fish Livers Description Freshwater. Dec 2012 Extraction Date 2-Jan-13 Analysis Date 21-Jan-13

	Level ng/g	Labelled Sur recove	-
PFPeA PFHxA	<10 5.0	288	F
PFHpA PFOA	9.4 <6	88	
PFNA	<10	54	
PFDA	55	265	þ
PFUdA	170	363	þ
PFDoA	12	336	þ
PFOS	56300	88	

#### Laboratory Reg. No. N12/034245

Client Sample Ref. CEL09 Matrix Fish Livers Description Freshwater. Dec 2012 Extraction Date 2-Jan-13 Analysis Date 21-Jan-13

	Level ng/g	Labelled Sur recove	
PFPeA PFHxA	<10 4.7	433	ħ
PFHpA PFOA	17 <9	94	
PFNA	<40	11	þ
PFDA	100	349	þ
PFUdA	290	467	þ
PFDoA	16	457	þ
PFOS	122000	77	

#### Laboratory Reg. No. N12/034246

Client Sample Ref. CEL10 Matrix Fish Livers Description Freshwater. Dec 2012 Extraction Date 2-Jan-13 Analysis Date 21-Jan-13

	Level ng/g	Labelled Sur recove	-
PFPeA PFHxA	<10 8.2	150	þ
PFHpA PFOA	6.0 6.1	65	
PFNA	<10	17	þ
PFDA	81	112	
PFUdA	390	121	
PFDoA	29	130	þ
PFOS	91400	98	

#### Laboratory Reg. No. N12/034247

Client Sample Ref. CEL11 Matrix Fish Livers Description Freshwater. Dec 2012 Extraction Date 2-Jan-13 Analysis Date 21-Jan-13

	Level ng/g	Labelled Sur recove	<u> </u>
PFPeA PFHxA	<10 5.5	348	Þ
PFHpA PFOA	15 14	60	
PFNA	14	44	
PFDA	110	232	þ
PFUdA	360	320	þ
PFDoA	23	306	þ
PFOS	86000	124	

#### Laboratory Reg. No. N12/034245DUP

Client Sample Ref. Duplicate Matrix Fish Livers Description Duplicate Sample Extraction Date 2-Jan-13 Analysis Date 21-Jan-13

	Level ng/g	Labelled Sur recover	-
PFPeA PFHxA	<10 6.4	96	
PFHpA PFOA	8.3 <5	37	
PFNA	<20	10	þ
PFDA	93	65	
PFUdA	310	93	
PFDoA	17	98	
PFOS	128000	18	þ

#### Laboratory Reg. No. N12/034245SPK

Client Sample Ref. Spike Matrix Fish Livers Description Spiked sample (104 ng/g) Extraction Date 2-Jan-13 Analysis Date 21-Jan-13

	Level ng/g	Labelled Sur recove	
PFPeA PFHxA	190 100	62	
PFHpA PFOA	340 100	26	
PFNA	86	11	þ
PFDA	180	55	
PFUdA	390	74	
PFDoA	110	75	
PFOS	123000	15	þ

#### Laboratory Reg. No. BLK L841

Client Sample Ref. Lab Blank Matrix Lab Blank Description Lab Blank Extraction Date 2-Jan-13 Analysis Date 21-Jan-13

	Level ng/g	Labelled Surrogate recovery	
PFPeA PFHxA	<0.9 <0.6	78	
PFHpA PFOA	<0.6 <0.5	72	
PFNA	<0.3	82	
PFDA	<0.2	86	
PFUdA	<0.3	88	
PFDoA	<0.4	80	
PFOS	<1	66	



**National Measurement Institute** 

CERTIFICATE OF ANALYSIS # DAU13_017				
Client	Cardno Ecology Lab	Job No.	CARD20/121218	
	L9, 203 Pacific Highway, St Leonards			
	NSW, 2065	Sampled by	Client	
		Date Sampled	not specified	
Contact	Marcus Lincoln-Smith	Date Received	18-Dec-12	
The results relate only to the sample(s) tested.				

Method	AUTL_07	Date Reported	1-Feb-2013

# **Details** The method is for determination of Perfluoroalkyl substances (PFASs) in biota samples by High Performance Liquid Chromatography tandem Mass Spectrometry (UPLC-MSMS). All results are corrected for labelled surrogates and are reported on a fresh weight basis.

Prior to extraction the sample is spiked with a range of isotopically labelled surrogate standards. Extraction is by organic solvent, with purification using activated silica gel. An aliquot of extract is injected onto the UPLC and detected using mass spectrometry.

Authorisation

Haw

Gavin Stevenson Manager Dioxin Analysis Unit

Dr Alan Yates Senior Analyst Dioxin Analysis Unit

Sample Details : Job No. CARD20/121218			
Laboratory Reg. No.	Client Sample Ref.	Matrix	Description
N12/034237	CEL01	Fish muscle	Freshwater. Dec 2012
N12/034238	CEL02	Fish muscle	Freshwater. Dec 2012
N12/034239	CEL03	Fish muscle	Freshwater. Dec 2012
N12/034240	CEL04	Fish muscle	Freshwater. Dec 2012
N12/034241	CEL05	Fish muscle	Freshwater. Dec 2012
N12/034242	CEL06	Fish muscle	Freshwater. Dec 2012
N12/034243	CEL07	Fish muscle	Freshwater. Dec 2012
N12/034240DUP	Duplicate	Fish muscle	Duplicate Sample
N12/034240SPK	Spike	Fish muscle	Spiked sample (44 ng/g)
BLK L840	Lab Blank	Lab Blank	Lab Blank

# Project Details Project Name

Project Number

Fiskville Study NA49913-034

Key		
Analytes		Surrogate
PFHpA	Perfluoro-n-heptanoic acid	
PFOA	Perfluoro-n-octanoic acid	Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]octanoic acid
PFNA	Perfluoro-n-nonanoic acid	Perfluoro-n-[1,2,3,4,5- <sup>13</sup> C <sub>5</sub> ]nonanoic acid Surrogate
PFDA	Perfluoro-n-decanoic acid	Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]decanoic acid Surrogate
PFUdA	Perfluoro-n-undecanoic acid	Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]undecanoic acid Surrogate
PFDoA	Perfluoro-n-dodecanoic acid	Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]dodecanoic acid Surrogate
PFOS	Perfluoro-n-octanesulfonate	Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]octanesulfonate
Units & Abbrev	iations	
ng/g	nanograms per gram	
<	level less than limit of reporting (LOR)	

#### Laboratory Reg. No. N12/034237

Client Sample Ref. CEL01 Matrix Fish muscle Description Freshwater. Dec 2012 Extraction Date 2-Jan-13 Analysis Date 21-Jan-13

	Level ng/g	Labelled Sur recove	-
PFHpA PFOA	<2 <2	55	
PFNA	<2	9	þ
PFDA	8.6	51	
PFUdA	25	63	
PFDoA	2.2	60	
PFOS	12100	11	þ

#### Laboratory Reg. No. N12/034238

Client Sample Ref. CEL02 Matrix Fish muscle Description Freshwater. Dec 2012 Extraction Date 2-Jan-13 Analysis Date 21-Jan-13

	Level ng/g	Labelled Surrogate recovery	
PFHpA PFOA	<2 <2	56	
PFNA	<2	8	þ
PFDA	13	46	
PFUdA	46	61	
PFDoA	3.1	68	
PFOS	22100	9	þ

#### Laboratory Reg. No. N12/034239

Client Sample Ref. CEL03 Matrix Fish muscle Description Freshwater. Dec 2012 Extraction Date 2-Jan-13 Analysis Date 21-Jan-13

	Level ng/g	Labelled Surrogate recovery	
PFHpA PFOA	<2 <2	63	
PFNA	<2	9	þ
PFDA	8.1	49	
PFUdA	40	60	
PFDoA	3.5	61	
PFOS	14900	10	þ

#### Laboratory Reg. No. N12/034240

Client Sample Ref. CEL04 Matrix Fish muscle Description Freshwater. Dec 2012 Extraction Date 2-Jan-13 Analysis Date 21-Jan-13

	Level ng/g	Labelled Surrogate recovery	
PFHpA PFOA	<2 <2	58	
PFNA	<2	7	þ
PFDA	12	46	
PFUdA	42	61	
PFDoA	3.1	64	
PFOS	22300	9	þ

### Laboratory Reg. No. N12/034241

Client Sample Ref. CEL05 Matrix Fish muscle Description Freshwater. Dec 2012 Extraction Date 2-Jan-13 Analysis Date 21-Jan-13

	Level ng/g	Labelled Sur recove	-
PFHpA PFOA	<2 <2	57	
PFNA	<2	8	þ
PFDA	7.9	47	
PFUdA	28	58	
PFDoA	2.2	64	
PFOS	13500	10	þ

### Laboratory Reg. No. N12/034242

Client Sample Ref. CEL06 Matrix Fish muscle Description Freshwater. Dec 2012 Extraction Date 2-Jan-13 Analysis Date 21-Jan-13

	Level ng/g	Labelled Sur recove	-
PFHpA PFOA	<2 <2	63	
PFNA	<2	7	þ
PFDA	13	44	
PFUdA	41	61	
PFDoA	2.7	68	
PFOS	23500	8	þ

### Laboratory Reg. No. N12/034243

Client Sample Ref. CEL07 Matrix Fish muscle Description Freshwater. Dec 2012 Extraction Date 2-Jan-13 Analysis Date 21-Jan-13

	Level ng/g	Labelled Sur recove	
PFHpA PFOA	<2 <2	64	
PFNA	<2	10	þ
PFDA	6.4	59	
PFUdA	28	69	
PFDoA	2.9	75	
PFOS	11200	12	þ

### Laboratory Reg. No. N12/034240DUP

Client Sample Ref. Duplicate Matrix Fish muscle Description Duplicate Sample Extraction Date 2-Jan-13 Analysis Date 21-Jan-13

	Level ng/g	Labelled Sur recove	-
PFHpA PFOA	<2 <2	55	
PFNA	<2	7	þ
PFDA	11	45	
PFUdA	44	57	
PFDoA	3.2	62	
PFOS	23000	8	þ

### Laboratory Reg. No. N12/034240SPK

Client Sample Ref. Spike Matrix Fish muscle Description Spiked sample (44 ng/g) Extraction Date 2-Jan-13 Analysis Date 21-Jan-13

	Level ng/g	Labelled Sur recover	
PFHpA PFOA	81 62	51	
PFNA	61	7	þ
PFDA	69	43	
PFUdA	100	51	
PFDoA	60	61	
PFOS	23600	9	þ

### Laboratory Reg. No. BLK L840

Client Sample Ref. Lab Blank Matrix Lab Blank Description Lab Blank Extraction Date 2-Jan-13 Analysis Date 21-Jan-13

	Level	Labelled Surrogate
	ng/g	recovery
PFHpA	<2	
PFOA	<2	59
PFNA	<2	63
PFDA	<0.5	57
PFUdA	<0.5	66
PFDoA	<0.5	61
PFOS	<0.5	62



**National Measurement Institute** 

CERTIFICATE OF ANALYSIS # DAU13_037			
Client	Cardno Ecology Lab	Job No.	CARD20/121218
	L9, 203 Pacific Highway, St L		
	NSW, 2065	Sampled by	Client
	•	Date Sampled	not specified
Contact	Marcus Lincoln-Smith	Date Received	18-Dec-12
The results relate only to the sample(s) tested.			

Method	AUTL_07	Date Reported	21-Feb-2013

# **Details** The method is for determination of Perfluoroalkyl substances (PFASs) in biota samples by High Performance Liquid Chromatography tandem Mass Spectrometry (UPLC-MSMS). All results are corrected for labelled surrogates and are reported on a fresh weight basis.

Prior to extraction the sample is spiked with a range of isotopically labelled surrogate standards. Extraction is by organic solvent, with purification using activated silica gel. An aliquot of extract is injected onto the UPLC and detected using mass spectrometry.

Authorisation

au

Gavin Stevenson Manager Dioxin Analysis Unit

Dr Alan Yates Senior Analyst Dioxin Analysis Unit

Sample Details : Job No. CARD20/121218				
Laboratory Reg. No.	Client Sample Ref.	Matrix	Description	
N12/034251	CEL15	Yabbie	Freshwater. Dec 2012	
N12/034252	CEL16	Yabbie	Freshwater. Dec 2012	
N12/034253	CEL17	Yabbie	Freshwater. Dec 2012	
N12/034254	CEL18	Shrimp	Freshwater. Dec 2012	
N12/034261	CEL25	Yabbie	Freshwater. Dec 2012	
N12/034262	CEL26	Yabbie	Freshwater. Dec 2012	
N12/034263	CEL27	Yabbie	Freshwater. Dec 2012	
N12/034267	CEL03A	Yabbie	Freshwater. Dec 2012	
N12/034261DUP	Duplicate	Yabbie	Duplicate Sample	
N12/034261SPK	Spike	Yabbie	Spiked sample (45 ng/g)	

Project Details	
Project Name	F

Project Number

Fiskville Study NA49913-034

Analytes		Surrogate
PFPeA	Perfluoro-n-pentanoic acid	- 13 -
PFHxA	Perfluoro-n-hexanoic acid	Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]hexanoic acid
PFHpA	Perfluoro-n-heptanoic acid	
PFOA	Perfluoro-n-octanoic acid	Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]octanoic acid
PFNA	Perfluoro-n-nonanoic acid	Perfluoro-n-[1,2,3,4,5- <sup>13</sup> C <sub>5</sub> ]nonanoic acid
PFDA	Perfluoro-n-decanoic acid	Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]decanoic acid
PFUdA	Perfluoro-n-undecanoic acid	Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]undecanoic acid
PFDoA	Perfluoro-n-dodecanoic acid	Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]dodecanoic acid
PFOS	Perfluoro-n-octanesulfonate	Perfluoro-n-[1,2,3,4-13C4]octanesulfonate
Units & Abbrev	iations	
ng/g	nanograms per gram	
<	level less than limit of reporting (LOR)	

### Laboratory Reg. No. N12/034251

Client Sample Ref. CEL15 Matrix Yabbie Description Freshwater. Dec 2012 Extraction Date 24-Jan-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Surrogate recovery	
PFPeA PFHxA	5.9 <5	130	Þ
PFHpA PFOA	4.0 21	52	
PFNA	11	17	þ
PFDA	15	4	þ
PFUdA	52	1	þ
PFDoA	40	1	þ
PFOS	560	77	

### Laboratory Reg. No. N12/034252

Client Sample Ref. CEL16 Matrix Yabbie Description Freshwater. Dec 2012 Extraction Date 24-Jan-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Surrogate recovery
PFPeA PFHxA	<5 <5	82
PFHpA PFOA	<2 18	83
PFNA	6.7	87
PFDA	2.4	103
PFUdA	5.8	121
PFDoA	<2	77
PFOS	2600	90

### Laboratory Reg. No. N12/034253

Client Sample Ref. CEL17 Matrix Yabbie Description Freshwater. Dec 2012 Extraction Date 24-Jan-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Surrogate recovery
PFPeA PFHxA	<5 <5	75
PFHpA PFOA	<2 23	87
PFNA	8.3	88
PFDA	4.3	95
PFUdA	16	102
PFDoA	2.5	42
PFOS	2000	99

### Laboratory Reg. No. N12/034254

Client Sample Ref. CEL18 Matrix Shrimp Description Freshwater. Dec 2012 Extraction Date 24-Jan-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Surrogate recovery
PFPeA PFHxA	<5 <5	82
PFHpA PFOA	<2 <2	93
PFNA	2.3	101
PFDA	2.2	86
PFUdA	2.5	100
PFDoA	<2	84
PFOS	260	99

### Laboratory Reg. No. N12/034261

Client Sample Ref. CEL25 Matrix Yabbie Description Freshwater. Dec 2012 Extraction Date 24-Jan-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Sur recove	
PFPeA PFHxA	<5 <5	75	
PFHpA PFOA	<2 23	90	
PFNA	2.1	88	
PFDA	<2	118	
PFUdA	2.6	144	þ
PFDoA	4.0	91	
PFOS	3000	93	

### Laboratory Reg. No. N12/034262

Client Sample Ref. CEL26 Matrix Yabbie Description Freshwater. Dec 2012 Extraction Date 24-Jan-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Surrogate recovery	
PFPeA PFHxA	5.6 12	82	
PFHpA PFOA	4.1 53	79	
PFNA	7.5	59	
PFDA	4.9	112	
PFUdA	4.7	<b>140</b> 🖟	
PFDoA	4.8	94	
PFOS	8000	112	

### Laboratory Reg. No. N12/034263

Client Sample Ref. CEL27 Matrix Yabbie Description Freshwater. Dec 2012 Extraction Date 24-Jan-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Suri recover	-
PFPeA PFHxA	<5 <5	77	
PFHpA PFOA	4.5 100	77	
PFNA	8.6	54	
PFDA	8.9	45	
PFUdA	15	24	þ
PFDoA	15	11	þ
PFOS	5200	93	

### Laboratory Reg. No. N12/034267

Client Sample Ref. CEL03A Matrix Yabbie Description Freshwater. Dec 2012 Extraction Date 24-Jan-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Sur recove	
PFPeA PFHxA	11 6.9	64	
PFHpA PFOA	4.4 19	80	
PFNA	4.5	38	
PFDA	7.7	21	þ
PFUdA	33	9	þ
PFDoA	16	4	þ
PFOS	5000	75	

### Laboratory Reg. No. N12/034261DUP

Client Sample Ref. Duplicate Matrix Yabbie Description Duplicate Sample Extraction Date 24-Jan-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Surrogate recovery
PFPeA PFHxA	<5 <5	75
PFHpA PFOA	<2 39	79
PFNA	4.1	66
PFDA	2.7	95
PFUdA	5.1	120
PFDoA	6.0	71
PFOS	3800	88

### Laboratory Reg. No. N12/034261SPK

Client Sample Ref. Spike Matrix Yabbie Description Spiked sample (45 ng/g) Extraction Date 24-Jan-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Surrogate recovery
PFPeA PFHxA	44 61	103
PFHpA PFOA	54 90	105
PFNA	64	75
PFDA	61	61
PFUdA	66	60
PFDoA	70	32
PFOS	2200	99



**National Measurement Institute** 

	CERTIFICATE OF A	ANALYSIS # DAU	J13_038
Client	Cardno Ecology Lab L9, 203 Pacific Highway, St I	Job No.	CARD20/121218
	NSW, 2065	Sampled by	Client
		Date Sampled	not specified
Contact	Marcus Lincoln-Smith	Date Received	18-Dec-12
The results relate only to the sample(s) tested.			

Method	AUTL_07	Date Reported	21-Feb-2013

# **Details** The method is for determination of Perfluoroalkyl substances (PFASs) in biota samples by High Performance Liquid Chromatography tandem Mass Spectrometry (UPLC-MSMS). All results are corrected for labelled surrogates and are reported on a fresh weight basis.

Prior to extraction the sample is spiked with a range of isotopically labelled surrogate standards. Extraction is by organic solvent, with purification using activated silica gel. An aliquot of extract is injected onto the UPLC and detected using mass spectrometry.

Authorisation

aun

Gavin Stevenson Manager Dioxin Analysis Unit

Dr Alan Yates Senior Analyst Dioxin Analysis Unit

Sample Details : Job No. CARD20/121218			
Laboratory Reg. No.	Client Sample Ref.	Matrix	Description
N12/034248	CEL12	Mosquito Fish	Freshwater. Dec 2012
N12/034249	CEL13	Mosquito Fish	Freshwater. Dec 2012
N12/034250	CEL14	Mosquito Fish	Freshwater. Dec 2012
N12/034258	CEL22	Mosquito Fish	Freshwater. Dec 2012
N12/034259	CEL23	Mosquito Fish	Freshwater. Dec 2012
N12/034260	CEL24	Mosquito Fish	Freshwater. Dec 2012
N12/034259DUP	Duplicate	Mosquito Fish	Duplicate Sample
N12/034259SPK	Spike	Mosquito Fish	Spiked sample (44 ng/g)
BLK L845	Blank	Blank	Laboratory Blank
Project Details			
Project Name	Fiskville Study		
Project Number	NA49913-034		
Key			
Analytes			Surrogate
PFPeA	Perfluoro-n-pentanoic acid	l	
PFHxA	Perfluoro-n-hexanoic acid		Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]hexanoic acid
PFHpA	Perfluoro-n-heptanoic acid	I	
PFOA	Perfluoro-n-octanoic acid		Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]octanoic acid
PFNA	Perfluoro-n-nonanoic acid		Perfluoro-n-[1,2,3,4,5- $^{13}C_5$ ]nonanoic acid
PFDA	Perfluoro-n-decanoic acid		Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]decanoic acid
PFUdA	Perfluoro-n-undecanoic acid		Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]undecanoic acid
PFDoA	Perfluoro-n-dodecanoic acid		Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]dodecanoic acid
PFOS	Perfluoro-n-octanesulfonate		Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]octanesulfonate
Units & Abbreviations			

ng/g < ns nanograms per gram level less than limit of reporting (LOR)

Surrogate Recovery percentage recovery for <sup>13</sup>C<sub>12</sub> labelled surrogate standard

D Laboratory surrogate recovery outside normal acceptance criteria (25 - 125%)

### Laboratory Reg. No. N12/034248

Client Sample Ref. CEL12 Matrix Mosquito Fish Description Freshwater. Dec 2012 Extraction Date 22-Jan-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Sur recove	
PFPeA PFHxA	<5 <5	192	F
PFHpA PFOA	2.5 4.5	102	
PFNA	6.4	81	
PFDA	12	270	þ
PFUdA	36	392	þ
PFDoA	3.7	318	þ
PFOS	50000	65	

### Laboratory Reg. No. N12/034249

Client Sample Ref. CEL13 Matrix Mosquito Fish Description Freshwater. Dec 2012 Extraction Date 22-Jan-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Sur recove	-
PFPeA PFHxA	<5 <5	182	þ
PFHpA PFOA	<2 2.3	102	
PFNA	2.3	88	
PFDA	6.3	131	þ
PFUdA	25	274	þ
PFDoA	2.6	196	þ
PFOS	30000	82	

### Laboratory Reg. No. N12/034250

Client Sample Ref. CEL14 Matrix Mosquito Fish Description Freshwater. Dec 2012 Extraction Date 22-Jan-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Su recove	
PFPeA PFHxA	<5 <5	286	ħ
PFHpA PFOA	2.5 3.0	112	
PFNA	5.3	144	þ
PFDA	9.0	199	þ
PFUdA	40	412	þ
PFDoA	3.8	290	þ
PFOS	36000	85	

### Laboratory Reg. No. N12/034258

Client Sample Ref. CEL22 Matrix Mosquito Fish Description Freshwater. Dec 2012 Extraction Date 22-Jan-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Sur recove	
PFPeA PFHxA	<5 12	236	F
PFHpA PFOA	11 11	88	
PFNA	<5	36	
PFDA	20	127	þ
PFUdA	58	227	þ
PFDoA	36	196	þ
PFOS	260000	40	

### Laboratory Reg. No. N12/034259

Client Sample Ref. CEL23 Matrix Mosquito Fish Description Freshwater. Dec 2012 Extraction Date 22-Jan-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Sur recove	-
PFPeA PFHxA	5.3 10	240	þ
PFHpA PFOA	8.5 9.0	94	
PFNA	<4	41	
PFDA	20	109	
PFUdA	46	236	þ
PFDoA	26	179	þ
PFOS	260000	39	

### Laboratory Reg. No. N12/034260

Client Sample Ref. CEL24 Matrix Mosquito Fish Description Freshwater. Dec 2012 Extraction Date 22-Jan-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Sur recove	
PFPeA PFHxA	<5 8.3	277	F
PFHpA PFOA	6.5 7.3	114	
PFNA	4.2	38	
PFDA	17	136	þ
PFUdA	52	229	þ
PFDoA	30	192	þ
PFOS	240000	36	

### Laboratory Reg. No. N12/034259DUP

Client Sample Ref. Duplicate Matrix Mosquito Fish Description Duplicate Sample Extraction Date 22-Jan-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Sur recove	
PFPeA PFHxA	<5 9.8	276	F
PFHpA PFOA	7.8 8.3	113	
PFNA	3.9	38	
PFDA	17	148	þ
PFUdA	40	271	þ
PFDoA	23	208	þ
PFOS	240000	38	

### Laboratory Reg. No. N12/034259SPK

Client Sample Ref. Spike Matrix Mosquito Fish Description Spiked sample (44 ng/g) Extraction Date 22-Jan-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Sur recove	
PFPeA PFHxA	40 63	262	ħ
PFHpA PFOA	130 58	115	
PFNA	53	38	
PFDA	79	118	
PFUdA	110	219	þ
PFDoA	86	186	þ
PFOS	280000	36	

### Laboratory Reg. No. BLK L845

Client Sample Ref. Blank Matrix Blank Description Laboratory Blank Extraction Date 22-Jan-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Surrogate recovery
PFPeA PFHxA	<0.4 <0.4	68
PFHpA PFOA	<0.4 <0.4	79
PFNA	<0.3	86
PFDA	<0.3	84
PFUdA	<0.5	121
PFDoA	<0.3	104
PFOS	<100	101



**National Measurement Institute** 

	CERTIFICATE OF A	NALYSIS # DAU	J13_039
Client	Cardno Ecology Lab	Job No.	CARD20/121218
	L9, 203 Pacific Highway, St L		Oliont
	NSW, 2065	Sampled by	Client
	_	Date Sampled	not specified
Contact	Marcus Lincoln-Smith	Date Received	18-Dec-12
The results relate only to the sample(s) tested.			

Method	AUTL_07	Date Reported	22-Feb-2013

# **Details** The method is for determination of Perfluoroalkyl substances (PFASs) in biota samples by High Performance Liquid Chromatography tandem Mass Spectrometry (UPLC-MSMS). All results are corrected for labelled surrogates and are reported on a fresh weight basis.

Prior to extraction the sample is spiked with a range of isotopically labelled surrogate standards. Extraction is by organic solvent, with purification using activated silica gel. An aliquot of extract is injected onto the UPLC and detected using mass spectrometry.

Authorisation

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Gavin Stevenson Manager Dioxin Analysis Unit

Dr Alan Yates Senior Analyst Dioxin Analysis Unit

Sample Details : Job No. CARD20/121218				
Laboratory Reg. No.	Client Sample Ref.	Matrix	Description	
N12/034255	CEL19	Macrophyte	Freshwater. Dec 2012	
N12/034256	CEL20	Macrophyte	Freshwater. Dec 2012	
N12/034257	CEL21	Macrophyte	Freshwater. Dec 2012	
N12/034264	CEL28	Macrophyte	Freshwater. Dec 2012	
N12/034265	CEL29	Macrophyte	Freshwater. Dec 2012	
N12/034266	CEL30	Macrophyte	Freshwater. Dec 2012	
N12/034264DUP N12/034264SPK	Duplicate	Macrophyte	Duplicate Sample	
BLK L849	Spike Blank	Macrophyte Blank	Spiked sample (45 ng/g)	
BLK L849	ыапк	Blank	Laboratory Blank	
Project Details				
Project Name	Fiskville Study			
Project Number	NA49913-034			
Key				
Analytes			Surrogate	
PFPeA	Perfluoro-n-pentanoic acid			
PFHxA	Perfluoro-n-hexanoic acid		Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]hexanoic acid	
PFHpA	Perfluoro-n-heptanoic acid	l		
PFOA	Perfluoro-n-octanoic acid		Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]octanoic acid	
PFNA	Perfluoro-n-nonanoic acid		Perfluoro-n-[1,2,3,4,5- <sup>13</sup> C <sub>5</sub> ]nonanoic acid	
PFDA	PFDA Perfluoro-n-decanoic acid		Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]decanoic acid	
PFUdA	JdA Perfluoro-n-undecanoic acid		Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]undecanoic acid	
PFDoA	Perfluoro-n-dodecanoic acid		Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]dodecanoic acid	
PFOS	Perfluoro-n-octanesulfonate		Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]octanesulfonate	
Units & Abbreviations				
ng/g	nanograms per gram			

Surrogate Recovery percentage recovery for <sup>13</sup>C<sub>12</sub> labelled surrogate standard

D Laboratory surrogate recovery outside normal acceptance criteria (25 - 125%)

### Laboratory Reg. No. N12/034255

Client Sample Ref. CEL19 Matrix Macrophyte Description Freshwater. Dec 2012 Extraction Date 1-Feb-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Surrogate recovery	
PFPeA PFHxA	6.2 5.7	79	
PFHpA PFOA	<2 3.2	81	
PFNA	<2	56	
PFDA	<2	33	
PFUdA	<2	43	
PFDoA	<2	77	
PFOS	1440	83	

### Laboratory Reg. No. N12/034256

Client Sample Ref. CEL20 Matrix Macrophyte Description Freshwater. Dec 2012 Extraction Date 1-Feb-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Sur recover	
PFPeA PFHxA	<5 5.6	98	
PFHpA PFOA	<2 <2	95	
PFNA	<2	98	
PFDA	<2	105	
PFUdA	<3	135	þ
PFDoA	<2	98	
PFOS	1240	82	

### Laboratory Reg. No. N12/034257

Client Sample Ref. CEL21 Matrix Macrophyte Description Freshwater. Dec 2012 Extraction Date 1-Feb-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Surrogate recovery
PFPeA PFHxA	<2 3.2	81
PFHpA PFOA	<2 <2	88
PFNA	<2	106
PFDA	<2	101
PFUdA	<2	133 🕞
PFDoA	<2	99
PFOS	440	80

### Laboratory Reg. No. N12/034264

Client Sample Ref. CEL28 Matrix Macrophyte Description Freshwater. Dec 2012 Extraction Date 1-Feb-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Surrogate recovery	
PFPeA PFHxA	14 26	73	
PFHpA PFOA	6.3 12	72	
PFNA	<2	41	
PFDA	<2	80	
PFUdA	<2	106	
PFDoA	<2	78	
PFOS	6000	78	

### Laboratory Reg. No. N12/034265

Client Sample Ref. CEL29 Matrix Macrophyte Description Freshwater. Dec 2012 Extraction Date 1-Feb-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Surrogate recovery	
PFPeA PFHxA	11 23	105	
PFHpA PFOA	6.5 10	90	
PFNA	<2	50	
PFDA	<2	99	
PFUdA	<2	<b>133</b> 🕀	
PFDoA	<2	114	
PFOS	6800	80	

#### Laboratory Reg. No. N12/034266

Client Sample Ref. CEL30 Matrix Macrophyte Description Freshwater. Dec 2012 Extraction Date 1-Feb-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Surrogate recovery
PFPeA PFHxA	13 22	97
PFHpA PFOA	6.7 11	87
PFNA	<2	69
PFDA	<2	101
PFUdA	<2	105
PFDoA	<2	102
PFOS	3600	80

#### Laboratory Reg. No. N12/034264DUP

Client Sample Ref. Duplicate Matrix Macrophyte Description Duplicate Sample Extraction Date 1-Feb-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Surrogate recovery
PFPeA PFHxA	14 23	91
PFHpA PFOA	5.7 9.8	90
PFNA	<2	55
PFDA	<2	87
PFUdA	<2	124
PFDoA	<2	97
PFOS	5000	82

#### Laboratory Reg. No. N12/034264SPK

Client Sample Ref. Spike Matrix Macrophyte Description Spiked sample (45 ng/g) Extraction Date 1-Feb-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Surrogate recovery
PFPeA PFHxA	67 81	67
PFHpA PFOA	61 58	63
PFNA	55	44
PFDA	60	59
PFUdA	57	77
PFDoA	53	62
PFOS	4600	82

#### Laboratory Reg. No. BLK L849

Client Sample Ref. Blank Matrix Blank Description Laboratory Blank Extraction Date 1-Feb-13 Analysis Date 19-Feb-13

	Level ng/g	Labelled Surrogate recovery
PFPeA PFHxA	<0.7 <0.9	28
PFHpA PFOA	<1 <1	31
PFNA	<1	33
PFDA	<1	30
PFUdA	<1	35
PFDoA	<1	28
PFOS	<80	90



**National Measurement Institute** 

CERTIFICATE OF ANALYSIS # DAU13_061			
Client	Cardno Ecology Lab	Job No.	CARD20/130315
	L9, 203 Pacific Highway, St L		Client
	NSW 2065	Sampled by Date Sampled	Client
Contact	Marcus Lincoln-Smith	Date Received	15-Mar-13
The results relate only to the sample(s) tested.			

Method	AUTL_07	Date Reported	8-Apr-2013

# **Details** The method is for determination of Perfluoroalkyl substances (PFASs) in biota samples by High Performance Liquid Chromatography tandem Mass Spectrometry (UPLC-MSMS). All results are corrected for labelled surrogates and are reported on a fresh weight basis.

Prior to extraction the sample is spiked with a range of isotopically labelled surrogate standards. Extraction is by organic solvent, with purification using activated silica gel. An aliquot of extract is injected onto the UPLC and detected using mass spectrometry.

Authorisation

aun

Gavin Stevenson Manager Dioxin Analysis Unit

Dr Alan Yates Senior Analyst Dioxin Analysis Unit

	Sample Details : Job No. CARD20/130315			
Laboratory Reg. No.	Client Sample Ref.	Matrix	Description	
N13/006929	CEL31	Fish muscle	Freshwater. Dec 2012	
N13/006930	CEL32	Fish muscle	Freshwater. Dec 2012	
N13/006931	CEL33	Fish muscle	Freshwater. Dec 2012	
N13/006932	CEL34	Fish muscle	Freshwater. Dec 2012	
N13/006930DUP	Duplicate	Fish muscle	Duplicate Sample	
N13/006930SPK BLK L854	Spike Lab Blank	Fish muscle Lab Blank	Spiked sample (97 ng/g, 78 ng/g for FTS) Lab Blank	
BER L034				
Project Details				
Project Name	Fiskville Study			
Project Number	NA49913-034			
Key				
Analytes Surrogate				
PFBA	Perfluoro-n-butanoic acid		Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]butanoic acid	
PFPeA	Perfluoro-n-pentanoic acid			
PFHxA	Perfluoro-n-hexanoic acid		Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]hexanoic acid Surrogate	
PFHpA	Perfluoro-n-heptanoic acid			
PFOA	Perfluoro-n-octanoic acid		Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]octanoic acid	
PFNA	Perfluoro-n-nonanoic acid		Perfluoro-n-[1,2,3,4,5- <sup>13</sup> C <sub>5</sub> ]nonanoic acid	
PFDA	Perfluoro-n-decanoic acid		Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]decanoic acid	
PFUdA	Perfluoro-n-undecanoic ac	id	Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]undecanoic acid	
PFDoA	Perfluoro-n-dodecanoic acid		Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]dodecanoic acid	
PFOS	Perfluoro-n-octanesulfonate		Perfluoro-n-[1,2,3,4-13C4]octanesulfonate	
6:2 FTS	1H,1H,2H,2H-perfluoro-n-octane sulfonate		1H,1H,2H,2H-perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]octane sulfonate	
Units & Abbreviations				
ng/g	nanograms per gram			
<	level less than limit of reporting	(LOR)		

#### Laboratory Reg. No. N13/006929

Client Sample Ref. CEL31 Matrix Fish muscle Description Freshwater. Dec 2012 Extraction Date 22-Mar-13 Analysis Date 3-Apr-13

	Level ng/g	Labelled Surrogate recovery
PFBA	<10	45
PFPeA PFHxA	<5 <5	58
PFHpA PFOA	<5 <5	65
PFNA	<6	95
PFDA	<2	74
PFUdA	<2	99
PFDoA	<2	83
PFOS	<10	66
6:2 FTS	<2	81

#### Laboratory Reg. No. N13/006930

Client Sample Ref. CEL32 Matrix Fish muscle Description Freshwater. Dec 2012 Extraction Date 22-Mar-13 Analysis Date 3-Apr-13

	Level ng/g	Labelled Surrogate recovery
PFBA	<10	58
PFPeA PFHxA	<5 <5	70
PFHpA PFOA	<5 <5	78
PFNA	<2	110
PFDA	<2	92
PFUdA	<2	108
PFDoA	<2	98
PFOS	25	82
6:2 FTS	<2	84

#### Laboratory Reg. No. N13/006931

Client Sample Ref. CEL33 Matrix Fish muscle Description Freshwater. Dec 2012 Extraction Date 22-Mar-13 Analysis Date 3-Apr-13

	Level ng/g	Labelled Surrogate recovery
PFBA	<10	53
PFPeA PFHxA	<5 <5	66
PFHpA PFOA	<5 <5	77
PFNA	<2	120
PFDA	<2	84
PFUdA	<2	90
PFDoA	<2	95
PFOS	60	83
6:2 FTS	<2	115

#### Laboratory Reg. No. N13/006932

Client Sample Ref. CEL34 Matrix Fish muscle Description Freshwater. Dec 2012 Extraction Date 22-Mar-13 Analysis Date 3-Apr-13

	Level ng/g	Labelled Surrogate recovery
PFBA	<10	67
PFPeA PFHxA	<5 <5	75
PFHpA PFOA	<5 <5	72
PFNA	<2	105
PFDA	<2	98
PFUdA	<2	110
PFDoA	<2	100
PFOS	33	80
6:2 FTS	<2	100

#### Laboratory Reg. No. N13/006930DUP

Client Sample Ref. Duplicate Matrix Fish muscle Description Duplicate Sample Extraction Date 22-Mar-13 Analysis Date 3-Apr-13

	Level ng/g	Labelled Surrogate recovery
PFBA	<10	75
PFPeA PFHxA	<5 <5	70
PFHpA PFOA	<5 <5	75
PFNA	<7	103
PFDA	<2	95
PFUdA	<2	110
PFDoA	<2	96
PFOS	24	65
6:2 FTS	<2	88

#### Laboratory Reg. No. N13/006930SPK

Client Sample Ref. Spike Matrix Fish muscle Description Spiked sample (97 ng/g, 78 ng/g for FTS) Extraction Date 22-Mar-13 Analysis Date 3-Apr-13

	Level ng/g	Labelled Surrogate recovery
PFBA	86	62
PFPeA PFHxA	93 99	75
PFHpA PFOA	110 92	79
PFNA	92	97
PFDA	88	94
PFUdA	85	98
PFDoA	97	90
PFOS	120	73
6:2 FTS	78	103

#### Laboratory Reg. No. BLK L854

Client Sample Ref. Lab Blank Matrix Lab Blank Description Lab Blank Extraction Date 22-Mar-13 Analysis Date 3-Apr-13

	Level	Labelled Surrogate
	ng/g	recovery
PFBA	<6	62
PFPeA PFHxA	<1 <1	66
PFHpA PFOA	<1 <1	83
PFNA	<1	85
PFDA	<1	86
PFUdA	<2	84
PFDoA	<0.9	67
PFOS	<4	65
6:2 FTS	<0.08	93



**National Measurement Institute** 

CERTIFICATE OF ANALYSIS # DAU13_116			
Client	Cardno Ecology Lab	Job No.	CARD20/130527
	L9, 203 Pacific Highway, St I		
	NSW 2065	Sampled by	Client
		Date Sampled	5/13-Dec-2012
Contact	Marcus Lincoln-Smith	Date Received	27-May-2013
The results relate only to the sample(s) tested.			
	The rest	uits relate only to the samp	Die(S) lesieu.

Method	AUTL_07	Date Reported	21-Jun-2013

# **Details** The method is for determination of Perfluoroalkyl substances (PFASs) in biota samples by High Performance Liquid Chromatography tandem Mass Spectrometry (UPLC-MSMS). All results are corrected for labelled surrogates and are reported on a fresh weight basis.

Prior to extraction the sample is spiked with a range of isotopically labelled surrogate standards. Extraction is by organic solvent, with purification using activated silica gel. An aliquot of extract is injected onto the UPLC and detected using mass spectrometry.

Authorisation

aun

Gavin Stevenson Manager Dioxin Analysis Unit

Dr Alan Yates Senior Analyst Dioxin Analysis Unit

Sample Details : Job No. CARD20/130527			
Laboratory Reg. No.	Client Sample Ref.	Matrix	Description
N13/014202X	PFM5A	Fish muscle	Fish Muscle 13/12/2012
N13/014203X	PFM6A	Fish muscle	Fish Muscle 13/12/2012
N13/014204X	PFM8A	Fish muscle	Fish Muscle 13/12/2012
N13/014205X	PFM9A	Fish muscle	Fish Muscle 13/12/2012
N13/014206X	PFM10A	Fish muscle	Fish Muscle 13/12/2012
N13/014207X	PFM11A	Fish muscle	Fish Muscle 13/12/2012
N13/014208X	PFM12A	Fish muscle	Fish Muscle 13/12/2012
N13/014209X	PFM14A	Fish muscle	Fish Muscle 13/12/2012
N13/014210X	CEL022	Fish muscle	Fish Muscle 5/12/2012
BLK L873	Lab Blank	Lab Blank	Lab Blank
Project Details			
Project Name	Fiskville Study		
Project Number	NA49913-034		

Key		
Analytes		Surrogate
PFBA	Perfluoro-n-butanoic acid	Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]butanoic acid
PFPeA	Perfluoro-n-pentanoic acid	
PFHxA	Perfluoro-n-hexanoic acid	Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]hexanoic acid Surrogate
PFHpA	Perfluoro-n-heptanoic acid	
PFOA	Perfluoro-n-octanoic acid	Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]octanoic acid
PFNA	Perfluoro-n-nonanoic acid	Perfluoro-n-[1,2,3,4,5- <sup>13</sup> C <sub>5</sub> ]nonanoic acid
PFDA	Perfluoro-n-decanoic acid	Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]decanoic acid
PFOS	Perfluoro-n-octanesulfonate	Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]octanesulfonate
6:2 FTS	1H,1H,2H,2H-perfluoro-n-octane sulfonate	1H,1H,2H,2H-perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]octane sulfonate
Units & Abbreviation	ıs	
ng/g	nanograms per gram	
<	level less than limit of reporting (LOR)	
<u>ل</u>	surrogate recovery outside normal method range (25-	125%)

#### Laboratory Reg. No. N13/014202X

Client Sample Ref. PFM5A Matrix Fish muscle Description Fish Muscle 13/12/2012 Extraction Date 3-Jun-13 Analysis Date 14-Jun-13

	Level ng/g	Labelled Surrogate recovery	
PFBA	<2	16	þ
PFPeA PFHxA	<0.5 <0.5	49	
PFHpA PFOA	<0.5 <0.5	42	
PFNA	<2	6	þ
PFDA	9.0	22	þ
PFOS	5990	13	þ
6:2 FTS	4.8	52	

#### Laboratory Reg. No. N13/014203X

Client Sample Ref. PFM6A Matrix Fish muscle Description Fish Muscle 13/12/2012 Extraction Date 3-Jun-13 Analysis Date 14-Jun-13

	Level ng/g	Labelled Surrogate recovery	
PFBA	<2	23	þ
PFPeA PFHxA	<0.5 <0.5	55	
PFHpA PFOA	<0.5 <0.5	50	
PFNA	<2	9	þ
PFDA	8.1	31	
PFOS	5520	17	þ
6:2 FTS	4.4	82	

#### Laboratory Reg. No. N13/014204X

Client Sample Ref. PFM8A Matrix Fish muscle Description Fish Muscle 13/12/2012 Extraction Date 3-Jun-13 Analysis Date 14-Jun-13

	Level ng/g	Labelled Surrogate recovery	
PFBA	<2	20	þ
PFPeA PFHxA	<0.5 <0.5	59	
PFHpA PFOA	<0.5 <0.5	45	
PFNA	<2	8	þ
PFDA	7.7	26	
PFOS	6450	14	þ
6:2 FTS	4.9	80	

#### Laboratory Reg. No. N13/014205X

Client Sample Ref. PFM9A Matrix Fish muscle Description Fish Muscle 13/12/2012 Extraction Date 3-Jun-13 Analysis Date 14-Jun-13

	Level ng/g	Labelled Surrogate recovery	
PFBA	<2	15	þ
PFPeA PFHxA	<0.5 <0.5	50	
PFHpA PFOA	<0.5 <0.6	38	
PFNA	<2	7	þ
PFDA	8.2	23	þ
PFOS	7440	12	þ
6:2 FTS	4.5	52	

#### Laboratory Reg. No. N13/014206X

Client Sample Ref. PFM10A Matrix Fish muscle Description Fish Muscle 13/12/2012 Extraction Date 3-Jun-13 Analysis Date 14-Jun-13

	Level ng/g	Labelled Surrogate recovery	
PFBA	<2	19	þ
PFPeA PFHxA	<0.5 <0.5	54	
PFHpA PFOA	<0.5 <0.5	42	
PFNA	<2	7	þ
PFDA	10	29	
PFOS	9600	13	þ
6:2 FTS	3.4	60	

#### Laboratory Reg. No. N13/014207X

Client Sample Ref. PFM11A Matrix Fish muscle Description Fish Muscle 13/12/2012 Extraction Date 3-Jun-13 Analysis Date 14-Jun-13

	Level ng/g	Labelled Surrogate recovery	
PFBA	<2	17	þ
PFPeA PFHxA	<0.5 <0.5	52	
PFHpA PFOA	<0.5 <0.5	42	
PFNA	<2	6	þ
PFDA	6.9	22	þ
PFOS	7940	11	þ
6:2 FTS	3.9	51	

#### Laboratory Reg. No. N13/014208X

Client Sample Ref. PFM12A Matrix Fish muscle Description Fish Muscle 13/12/2012 Extraction Date 3-Jun-13 Analysis Date 14-Jun-13

	Level ng/g	Labelled Sur recove	-
PFBA	<2	20	þ
PFPeA PFHxA	<0.5 <0.5	55	
PFHpA PFOA	<0.5 <0.5	47	
PFNA	<2	7	þ
PFDA	9.6	27	
PFOS	8870	14	þ
6:2 FTS	3.9	70	

#### Laboratory Reg. No. N13/014209X

Client Sample Ref. PFM14A Matrix Fish muscle Description Fish Muscle 13/12/2012 Extraction Date 3-Jun-13 Analysis Date 14-Jun-13

	Level ng/g	Labelled Sur recove	-
PFBA	<2	22	þ
PFPeA PFHxA	<0.5 <0.5	67	
PFHpA PFOA	<0.5 <0.5	52	
PFNA	<2	9	þ
PFDA	7.7	37	
PFOS	7100	16	þ
6:2 FTS	5.3	79	

#### Laboratory Reg. No. N13/014210X

Client Sample Ref. CEL022 Matrix Fish muscle Description Fish Muscle 5/12/2012 Extraction Date 3-Jun-13 Analysis Date 14-Jun-13

	Level ng/g	Labelled Sur recove	-
PFBA	<2	23	þ
PFPeA PFHxA	<0.5 <0.5	75	
PFHpA PFOA	<0.5 <0.5	42	
PFNA	<0.5	48	
PFDA	<0.5	19	þ
PFOS	41	33	
6:2 FTS	<0.1	78	

#### Laboratory Reg. No. BLK L873

Client Sample Ref. Lab Blank Matrix Lab Blank Description Lab Blank Extraction Date 3-Jun-13 Analysis Date 14-Jun-13

	Level ng/g	Labelled Su recove	-
PFBA	<2	13	þ
PFPeA PFHxA	<0.5 <0.5	40	
PFHpA PFOA	<0.5 <0.5	26	
PFNA	<0.5	18	þ
PFDA	<0.5	6	þ
PFOS	<1	26	
6:2 FTS	<0.1	62	



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# **Certificate of Analysis**

Date Issued:	28 June 2013
Client:	Cardno LanePiper Building 2 154 Highbury Road Burwood Victoria 3125
Attention:	Marcus Lincoln Smith
AsureQuality Lab. Reference:	134672
Sample Type(s):	Fish Muscle
Analysis:	Perfluorinated Compounds (PFCs)
Method:	In-House LC-MS/MS Method

Results are reported as nanograms per gram (ng/g), on an as received basis to two significant figures. The LOR value is reported to two significant figures. Results have been corrected for recovery.

Unless requested, samples will be disposed of eight weeks from the date of this report.

#### **Comments:**

The requirement for dilution has resulted in a higher than normal LOR for PFOS.

Phil Bridgen Senior Scientist AsureQuality Limited

Laboratory Reference: 134672-1

Sample Identification: PFM5B - Fish Muscle

Date Received: 06 Jun 2013 Date Extracted: 14 Jun 2013		Date Analysed: 17 Jun 2013	
Analyte <sup>1</sup>	$\operatorname{Conc.}^{2}(ng/g)$	LOR (ng/g)	Data Qualifiers
Perfluoroalkylsulfonic acids			
Perfluorobutanesulfonic acid (PFBS)	ND	1.0	
Perfluorohexanesulfonic acid (PFHxS)	11	1.0	
Perfluorooctanesulfonic acid (PFOS) <sup>3</sup>	5500	400	
Perfluorodecanesulfonic acid (PFDS)	12	1.0	
Perfluoroalkylcarboxylic acids			
Perfluorohexanoic acid (PFHxA)	ND	1.0	
Perfluoroheptanoic acid (PFHpA)	ND	1.0	
Perfluorooctanoic acid (PFOA)	ND	2.0	
Perfluorononanoic acid (PFNA)	ND	2.0	
Perfluorodecanoic acid (PFDA)	7.2	2.0	
Perfluoroundecanoic acid (PFUnA)	18	1.0	Е
Perfluorododecanoic acid (PFDoA)	ND	2.0	
Perfluorotridecanoic acid (PFTrDA)	2.1	1.0	
Perfluorotetradecanoic acid (PFTeDA)	ND	1.0	
Other PFCs			
Perfluorooctanesulfonamide (PFOSA)	2.0	1.0	
N-ethyl-perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	ND	1.0	
N-methyl-perfluorooctanesulfonamidoacetic acid (NMeFOSAA)	ND	1.0	
1H,1H,2H,2H-perfluorooctanesulfonic acid (6:2 FTS)	2.4	1.0	
1H,1H,2H,2H-perfluorodecanesulfonic acid (8:2 FTS)	23	2.0	
Footnotes:	Abbreviations:		
<sup>1</sup> The analytes listed represent the linear isomer.	LOR: Limit of Reporting		
<sup>2</sup> Results are reported on an as received basis.	ND: Not Detected		
<sup>3</sup> The result for PFOS also includes its salts and	E: Estimated value		
perfluorooctanesulfonyl fluoride (PFOSF).			

Lab Analyst: CFH/CA

Data Analyst: CFH

Laboratory Reference: 134672-2

Sample Identification: PFM6B - Fish Muscle

Date Received: 06 Jun 2013 Date Extracted: 14 Jun 2013		Date Analysed: 17 Jun 2013	
Analyte <sup>1</sup>	Conc. <sup>2</sup> (ng/g)	LOR (ng/g)	Data Qualifiers
Perfluoroalkylsulfonic acids			
Perfluorobutanesulfonic acid (PFBS)	ND	1.0	
Perfluorohexanesulfonic acid (PFHxS)	6.5	1.0	
Perfluorooctanesulfonic acid (PFOS) <sup>3</sup>	4200	400	
Perfluorodecanesulfonic acid (PFDS)	11	1.0	
Perfluoroalkylcarboxylic acids			
Perfluorohexanoic acid (PFHxA)	ND	1.0	
Perfluoroheptanoic acid (PFHpA)	ND	1.0	
Perfluorooctanoic acid (PFOA)	ND	2.0	
Perfluorononanoic acid (PFNA)	ND	2.0	
Perfluorodecanoic acid (PFDA)	5.3	2.0	
Perfluoroundecanoic acid (PFUnA)	14	1.0	
Perfluorododecanoic acid (PFDoA)	ND	2.0	
Perfluorotridecanoic acid (PFTrDA)	1.6	1.0	
Perfluorotetradecanoic acid (PFTeDA)	ND	1.0	
Other PFCs			
Perfluorooctanesulfonamide (PFOSA)	2.0	1.0	
N-ethyl-perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	ND	1.0	
N-methyl-perfluorooctanesulfonamidoacetic acid (NMeFOSAA)	ND	1.0	
1H,1H,2H,2H-perfluorooctanesulfonic acid (6:2 FTS)	2.4	1.0	
1H,1H,2H,2H-perfluorodecanesulfonic acid (8:2 FTS)	20	2.0	
Footnotes:	Abbreviations:		
<sup>1</sup> The analytes listed represent the linear isomer.	LOR: Limit of Reporting	5	
<sup>2</sup> Results are reported on an as received basis.	ND: Not Detected		
<sup>3</sup> The result for PFOS also includes its salts and			
perfluorooctanesulfonyl fluoride (PFOSF).			

Lab Analyst: CFH/CA

Data Analyst: CFH

Laboratory Reference: 134672-3

Sample Identification: PFM8B - Fish Muscle

Date Received: 06 Jun 2013 Date Extracted: 14 Jun 2013		Date Analysed: 17 Jun 2		
Analyte <sup>1</sup>	$\operatorname{Conc.}^2(\operatorname{ng/g})$	LOR (ng/g)	Data Qualifiers	
Perfluoroalkylsulfonic acids				
Perfluorobutanesulfonic acid (PFBS)	ND	1.0		
Perfluorohexanesulfonic acid (PFHxS)	15	1.0		
Perfluorooctanesulfonic acid (PFOS) <sup>3</sup>	5600	400		
Perfluorodecanesulfonic acid (PFDS)	15	1.0		
Perfluoroalkylcarboxylic acids				
Perfluorohexanoic acid (PFHxA)	ND	1.0		
Perfluoroheptanoic acid (PFHpA)	ND	1.0		
Perfluorooctanoic acid (PFOA)	ND	2.0		
Perfluorononanoic acid (PFNA)	ND	2.0		
Perfluorodecanoic acid (PFDA)	6.2	2.0		
Perfluoroundecanoic acid (PFUnA)	20	1.0		
Perfluorododecanoic acid (PFDoA)	ND	2.0		
Perfluorotridecanoic acid (PFTrDA)	2.4	1.0		
Perfluorotetradecanoic acid (PFTeDA)	ND	1.0		
Other PFCs				
Perfluorooctanesulfonamide (PFOSA)	2.5	1.0		
N-ethyl-perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	ND	1.0		
N-methyl-perfluorooctanesulfonamidoacetic acid (NMeFOSAA)	ND	1.0		
1H,1H,2H,2H-perfluorooctanesulfonic acid (6:2 FTS)	3.1	1.0		
1H,1H,2H,2H-perfluorodecanesulfonic acid (8:2 FTS)	26	2.0		
Footnotes:	Abbreviations:			
<sup>1</sup> The analytes listed represent the linear isomer.	LOR: Limit of Reporting	3		
<sup>2</sup> Results are reported on an as received basis.	ND: Not Detected			
<sup>3</sup> The result for PFOS also includes its salts and				
perfluorooctanesulfonyl fluoride (PFOSF).				

Lab Analyst: CFH/CA

Data Analyst: CFH

Laboratory Reference: 134672-4

Sample Identification: PFM9B - Fish Muscle

Date Received: 06 Jun 2013 Date Extracted: 14 Jun 2013		Date Analysed: 17 Jun 2013	
Analyte <sup>1</sup>	Conc. <sup>2</sup> (ng/g)	LOR (ng/g)	Data Qualifiers
Perfluoroalkylsulfonic acids			
Perfluorobutanesulfonic acid (PFBS)	ND	1.0	
Perfluorohexanesulfonic acid (PFHxS)	16	1.0	
Perfluorooctanesulfonic acid (PFOS) <sup>3</sup>	6800	400	
Perfluorodecanesulfonic acid (PFDS)	15	1.0	
Perfluoroalkylcarboxylic acids			
Perfluorohexanoic acid (PFHxA)	ND	1.0	
Perfluoroheptanoic acid (PFHpA)	ND	1.0	
Perfluorooctanoic acid (PFOA)	ND	2.0	
Perfluorononanoic acid (PFNA)	ND	2.0	
Perfluorodecanoic acid (PFDA)	7.3	2.0	
Perfluoroundecanoic acid (PFUnA)	23	1.0	
Perfluorododecanoic acid (PFDoA)	ND	2.0	
Perfluorotridecanoic acid (PFTrDA)	2.4	1.0	
Perfluorotetradecanoic acid (PFTeDA)	ND	1.0	
Other PFCs			
Perfluorooctanesulfonamide (PFOSA)	2.8	1.0	
N-ethyl-perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	ND	1.0	
N-methyl-perfluorooctanesulfonamidoacetic acid (NMeFOSAA)	ND	1.0	
1H,1H,2H,2H-perfluorooctanesulfonic acid (6:2 FTS)	2.4	1.0	
1H,1H,2H,2H-perfluorodecanesulfonic acid (8:2 FTS)	30	2.0	
Footnotes:	Abbreviations:		
<sup>1</sup> The analytes listed represent the linear isomer.	LOR: Limit of Reporting	5	
<sup>2</sup> Results are reported on an as received basis.	ND: Not Detected	-	
<sup>3</sup> The result for PFOS also includes its salts and			
perfluorooctanesulfonyl fluoride (PFOSF).			

Lab Analyst: CFH/CA

Data Analyst: CFH

Laboratory Reference: 134672-5

Sample Identification: PFM10B - Fish Muscle

Date Received: 06 Jun 2013 Date Extracted: 14 Jun 2013		Date Analysed: 17 Jun 2013	
Analyte <sup>1</sup>	Conc. <sup>2</sup> (ng/g)	LOR (ng/g)	Data Qualifiers
Perfluoroalkylsulfonic acids			
Perfluorobutanesulfonic acid (PFBS)	ND	1.0	
Perfluorohexanesulfonic acid (PFHxS)	9.4	1.0	
Perfluorooctanesulfonic acid (PFOS) <sup>3</sup>	8800	400	
Perfluorodecanesulfonic acid (PFDS)	22	1.0	
Perfluoroalkylcarboxylic acids			
Perfluorohexanoic acid (PFHxA)	ND	1.0	
Perfluoroheptanoic acid (PFHpA)	ND	1.0	
Perfluorooctanoic acid (PFOA)	ND	2.0	
Perfluorononanoic acid (PFNA)	ND	2.0	
Perfluorodecanoic acid (PFDA)	11	2.0	
Perfluoroundecanoic acid (PFUnA)	35	1.0	Е
Perfluorododecanoic acid (PFDoA)	2.4	2.0	
Perfluorotridecanoic acid (PFTrDA)	4.0	1.0	
Perfluorotetradecanoic acid (PFTeDA)	ND	1.0	
Other PFCs			
Perfluorooctanesulfonamide (PFOSA)	1.9	1.0	
N-ethyl-perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	ND	1.0	
N-methyl-perfluorooctanesulfonamidoacetic acid (NMeFOSAA)	ND	1.0	
1H,1H,2H,2H-perfluorooctanesulfonic acid (6:2 FTS)	3.2	1.0	
1H,1H,2H,2H-perfluorodecanesulfonic acid (8:2 FTS)	23	2.0	
Footnotes:	Abbreviations:		
<sup>1</sup> The analytes listed represent the linear isomer.	LOR: Limit of Reporting		
<sup>2</sup> Results are reported on an as received basis.	ND: Not Detected		
<sup>3</sup> The result for PFOS also includes its salts and perfluorooctanesulfonyl fluoride (PFOSF).	E: Estimated value		

Lab Analyst: CFH/CA

Data Analyst: CFH

Laboratory Reference: 134672-6

Sample Identification: PFM11B - Fish Muscle

Date Received: 06 Jun 2013 Date Extracted: 14 Jun 2013		Date Analysed: 17 Jun 2013	
Analyte <sup>1</sup>	$\operatorname{Conc.}^{2}(\operatorname{ng/g})$	LOR (ng/g)	Data Qualifiers
Perfluoroalkylsulfonic acids			
Perfluorobutanesulfonic acid (PFBS)	ND	1.0	
Perfluorohexanesulfonic acid (PFHxS)	13	1.0	
Perfluorooctanesulfonic acid (PFOS) <sup>3</sup>	6600	400	
Perfluorodecanesulfonic acid (PFDS)	20	1.0	
Perfluoroalkylcarboxylic acids			
Perfluorohexanoic acid (PFHxA)	ND	1.0	
Perfluoroheptanoic acid (PFHpA)	ND	1.0	
Perfluorooctanoic acid (PFOA)	ND	2.0	
Perfluorononanoic acid (PFNA)	ND	2.0	
Perfluorodecanoic acid (PFDA)	8.1	2.0	
Perfluoroundecanoic acid (PFUnA)	26	1.0	
Perfluorododecanoic acid (PFDoA)	2.4	2.0	
Perfluorotridecanoic acid (PFTrDA)	4.5	1.0	
Perfluorotetradecanoic acid (PFTeDA)	ND	1.0	
Other PFCs			
Perfluorooctanesulfonamide (PFOSA)	2.7	1.0	
N-ethyl-perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	ND	1.0	
N-methyl-perfluorooctanesulfonamidoacetic acid (NMeFOSAA)	ND	1.0	
1H,1H,2H,2H-perfluorooctanesulfonic acid (6:2 FTS)	1.3	1.0	
1H,1H,2H,2H-perfluorodecanesulfonic acid (8:2 FTS)	32	2.0	
Footnotes:	Abbreviations:		
<sup>1</sup> The analytes listed represent the linear isomer.	LOR: Limit of Reportin	g	
<sup>2</sup> Results are reported on an as received basis.	ND: Not Detected		
<sup>3</sup> The result for PFOS also includes its salts and			
perfluorooctanesulfonyl fluoride (PFOSF).			

Lab Analyst: CFH/CA

Data Analyst: CFH

Laboratory Reference: 134672-7

Sample Identification: PFM12B - Fish Muscle

Analyte <sup>1</sup>			
Maryte	$\operatorname{Conc.}^2(\operatorname{ng/g})$	LOR (ng/g)	Data Qualifiers
Perfluoroalkylsulfonic acids			
Perfluorobutanesulfonic acid (PFBS)	ND	1.0	
Perfluorohexanesulfonic acid (PFHxS)	15	1.0	
Perfluorooctanesulfonic acid (PFOS) <sup>3</sup>	9900	400	
Perfluorodecanesulfonic acid (PFDS)	22	1.0	
Perfluoroalkylcarboxylic acids			
Perfluorohexanoic acid (PFHxA)	ND	1.0	
Perfluoroheptanoic acid (PFHpA)	ND	1.0	
Perfluorooctanoic acid (PFOA)	ND	2.0	
Perfluorononanoic acid (PFNA)	ND	2.0	
Perfluorodecanoic acid (PFDA)	13	2.0	
Perfluoroundecanoic acid (PFUnA)	42	1.0	
Perfluorododecanoic acid (PFDoA)	2.7	2.0	
Perfluorotridecanoic acid (PFTrDA)	4.8	1.0	
Perfluorotetradecanoic acid (PFTeDA)	ND	1.0	
Other PFCs			
Perfluorooctanesulfonamide (PFOSA)	2.2	1.0	
N-ethyl-perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	ND	1.0	
N-methyl-perfluorooctanesulfonamidoacetic acid (NMeFOSAA)	ND	1.0	
H,1H,2H,2H-perfluorooctanesulfonic acid (6:2 FTS)	1.7	1.0	
H,1H,2H,2H-perfluorodecanesulfonic acid (8:2 FTS)	25	2.0	
Footnotes:	Abbreviations:		
<sup>1</sup> The analytes listed represent the linear isomer.	LOR: Limit of Reportin	g	
<ul> <li><sup>2</sup> Results are reported on an as received basis.</li> <li><sup>3</sup> The result for PFOS also includes its salts and</li> </ul>	ND: Not Detected		

Lab Analyst: CFH/CA

Data Analyst: CFH

Laboratory Reference: 134672-8

Sample Identification: PFM14B - Fish Muscle

Date Received: 06 Jun 2013 Date Extracted: 14 Jun 2013		Date Analysed: 17 Jun 2013	
Analyte <sup>1</sup>	$\operatorname{Conc.}^2(\operatorname{ng/g})$	LOR (ng/g)	Data Qualifiers
Perfluoroalkylsulfonic acids			
Perfluorobutanesulfonic acid (PFBS)	ND	1.0	
Perfluorohexanesulfonic acid (PFHxS)	8.8	1.0	
Perfluorooctanesulfonic acid (PFOS) <sup>3</sup>	6200	400	
Perfluorodecanesulfonic acid (PFDS)	15	1.0	
Perfluoroalkylcarboxylic acids			
Perfluorohexanoic acid (PFHxA)	ND	1.0	
Perfluoroheptanoic acid (PFHpA)	ND	1.0	
Perfluorooctanoic acid (PFOA)	ND	2.0	
Perfluorononanoic acid (PFNA)	ND	2.0	
Perfluorodecanoic acid (PFDA)	6.3	2.0	
Perfluoroundecanoic acid (PFUnA)	24	1.0	
Perfluorododecanoic acid (PFDoA)	ND	2.0	
Perfluorotridecanoic acid (PFTrDA)	2.8	1.0	
Perfluorotetradecanoic acid (PFTeDA)	ND	1.0	
Other PFCs			
Perfluorooctanesulfonamide (PFOSA)	1.7	1.0	
N-ethyl-perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	ND	1.0	
N-methyl-perfluorooctanesulfonamidoacetic acid (NMeFOSAA)	ND	1.0	
1H,1H,2H,2H-perfluorooctanesulfonic acid (6:2 FTS)	2.3	1.0	
1H,1H,2H,2H-perfluorodecanesulfonic acid (8:2 FTS)	24	2.0	
Footnotes:	Abbreviations:		
<sup>1</sup> The analytes listed represent the linear isomer.	LOR: Limit of Reportin	g	
<ul> <li><sup>2</sup> Results are reported on an as received basis.</li> <li><sup>3</sup> The result for PFOS also includes its salts and perfluorooctanesulfonyl fluoride (PFOSF).</li> </ul>	ND: Not Detected		

Lab Analyst: CFH/CA

Data Analyst: CFH

Laboratory Reference: 134672-BL

Sample Identification: Laboratory Blank

Date Received: Not Applicable Date Extracted: 14 Jun 2013		Date Analyse	Date Analysed: 17 Jun 2013	
Analyte <sup>1</sup>	Conc. <sup>2</sup> (ng/g)	LOR (ng/g)	Data Qualifiers	
Perfluoroalkylsulfonic acids				
Perfluorobutanesulfonic acid (PFBS)	ND	1.0		
Perfluorohexanesulfonic acid (PFHxS)	ND	1.0		
Perfluorooctanesulfonic acid (PFOS) <sup>3</sup>	ND	2.0		
Perfluorodecanesulfonic acid (PFDS)	ND	1.0		
Perfluoroalkylcarboxylic acids				
Perfluorohexanoic acid (PFHxA)	ND	1.0		
Perfluoroheptanoic acid (PFHpA)	ND	1.0		
Perfluorooctanoic acid (PFOA)	ND	2.0		
Perfluorononanoic acid (PFNA)	ND	2.0		
Perfluorodecanoic acid (PFDA)	ND	2.0		
Perfluoroundecanoic acid (PFUnA)	ND	1.0		
Perfluorododecanoic acid (PFDoA)	ND	2.0		
Perfluorotridecanoic acid (PFTrDA)	ND	1.0		
Perfluorotetradecanoic acid (PFTeDA)	ND	1.0		
Other PFCs				
Perfluorooctanesulfonamide (PFOSA)	ND	1.0		
N-ethyl-perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	ND	1.0		
N-methyl-perfluorooctanesulfonamidoacetic acid (NMeFOSAA)	ND	1.0		
1H,1H,2H,2H-perfluorooctanesulfonic acid (6:2 FTS)	ND	1.0		
1H,1H,2H,2H-perfluorodecanesulfonic acid (8:2 FTS)	ND	2.0		
Footnotes:	Abbreviations:			
<sup>1</sup> The analytes listed represent the linear isomer	LOR: Limit of Reporting	g		
<sup>2</sup> The results are calculated using the average weight	ND: Not Detected			
of samples in this batch				
<sup>3</sup> The result for PFOS also includes its salts and				
perfluorooctanesulfonyl fluoride (PFOSF).				

Lab Analyst: CFH/CA

Data Analyst: CFH



**National Measurement Institute** 

CERTIFICATE OF ANALYSIS # DAU13_152				
Client	Cardno Ecology Lab L9, 203 Pacific Highway, St L	Job No.	CARD20/130711	
	NSW 2065	Sampled by Date Sampled	Client 4-Dec-2012	
Contact	Marcus Lincoln-Smith	Date Received		
The results relate only to the sample(s) tested.				

Method	AUTL_07	Date Reported	23-Jul-2013

# **Details** The method is for determination of Perfluoroalkyl substances (PFASs) in biota samples by High Performance Liquid Chromatography tandem Mass Spectrometry (UPLC-MSMS). All results are corrected for labelled surrogates and are reported on a fresh weight basis.

Prior to extraction the sample is spiked with a range of isotopically labelled surrogate standards. Extraction is by organic solvent, with purification using activated silica gel. An aliquot of extract is injected onto the UPLC and detected using mass spectrometry.

Authorisation

aur

Gavin Stevenson Manager Dioxin Analysis Unit

Dr Alan Yates Senior Analyst Dioxin Analysis Unit

Sample Details : Job No. CARD20/130711			
Laboratory Reg. No.	Client Sample Ref.	Matrix	Description
N13/017964X	PFM7	Fish muscle	Fish Muscle 4/12/2012
N13/017965X	PFM13	Fish muscle	Fish Muscle 4/12/2012
N13/017966X	PFM15	Fish muscle	Fish Muscle 4/12/2012
N13/017967X	PFM16	Fish muscle	Fish Muscle 4/12/2012
N13/017968X	PFM17	Fish muscle	Fish Muscle 4/12/2012
N13/017969X	PFM18	Fish muscle	Fish Muscle 4/12/2012
N13/017970X	PFM19	Fish muscle	Fish Muscle 4/12/2012
N13/017971X	PFM20	Fish muscle	Fish Muscle 4/12/2012
N13/017972X	PFM21	Fish muscle	Fish Muscle 4/12/2012
BLK L884	Lab Blank	Lab Blank	Lab Blank

# Project Details

Project Name Project Number Fiskville Study NA49913-034

Key		
Analytes		Surrogate
PFPeA	Perfluoro-n-pentanoic acid	
PFHxA	Perfluoro-n-hexanoic acid	Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]hexanoic acid Surrogate
PFHpA	Perfluoro-n-heptanoic acid	
PFOA	Perfluoro-n-octanoic acid	Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]octanoic acid
PFNA	Perfluoro-n-nonanoic acid	Perfluoro-n-[1,2,3,4,5-13C5]nonanoic acid
PFDA	Perfluoro-n-decanoic acid	Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]decanoic acid
PFUdA	Perfluoro-n-undecanoic acid	Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]undecanoic acid
PFDoA	Perfluoro-n-dodecanoic acid	Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]dodecanoic acid
PFOS	Perfluoro-n-octanesulfonate	Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]octanesulfonate
6:2 FTS	1H,1H,2H,2H-perfluoro-n-octane sulfonate	1H,1H,2H,2H-perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]octane sulfonate
Units & Abbrevia	ations	
ng/g	nanograms per gram	
<	level less than limit of reporting (LOR)	
þ	surrogate recovery outside normal method range (25-	125%)

#### Laboratory Reg. No. N13/017964X

Client Sample Ref. PFM7 Matrix Fish muscle Description Fish Muscle 4/12/2012 Extraction Date 12-Jul-13 Analysis Date 19-Jul-13

	Level ng/g	Labelled Surrogate recovery	
PFPeA PFHxA	<0.5 <0.5	76	
PFHpA PFOA	<0.5 <0.5	79	
PFNA	<1	9	þ
PFDA	7.6	58	
PFUdA	26	49	
PFDoA	<2	20	þ
PFOS	7600	18	þ
6:2 FTS	2.6	77	

#### Laboratory Reg. No. N13/017965X

Client Sample Ref. PFM13 Matrix Fish muscle Description Fish Muscle 4/12/2012 Extraction Date 12-Jul-13 Analysis Date 19-Jul-13

	Level ng/g	Labelled Surrogate recovery	
PFPeA PFHxA	<0.5 <0.5	96	
PFHpA PFOA	<0.5 <0.5	96	
PFNA	<1	12	þ
PFDA	5.1	71	
PFUdA	18	50	
PFDoA	<2	18	þ
PFOS	7000	19	þ
6:2 FTS	3.4	69	

#### Laboratory Reg. No. N13/017966X

Client Sample Ref. PFM15 Matrix Fish muscle Description Fish Muscle 4/12/2012 Extraction Date 12-Jul-13 Analysis Date 19-Jul-13

	Level ng/g	Labelled Surrogate recovery	
PFPeA PFHxA	<0.5 <0.5	91	
PFHpA PFOA	<0.5 <0.5	85	
PFNA	<1	10	þ
PFDA	4.9	63	
PFUdA	21	45	
PFDoA	<2	16	þ
PFOS	8300	17	þ
6:2 FTS	2.4	70	

#### Laboratory Reg. No. N13/017967X

Client Sample Ref. PFM16 Matrix Fish muscle Description Fish Muscle 4/12/2012 Extraction Date 12-Jul-13 Analysis Date 19-Jul-13

	Level ng/g	Labelled Surrogate recovery	
PFPeA PFHxA	<0.5 <0.5	106	
PFHpA PFOA	<0.5 <0.5	95	
PFNA	<1	12	þ
PFDA	4.8	72	
PFUdA	24	47	
PFDoA	<2	13	þ
PFOS	6700	21	þ
6:2 FTS	3.4	63	

# Page 7 of 12

# Results : Job No. CARD20/130711

#### Laboratory Reg. No. N13/017968X

Client Sample Ref. PFM17 Matrix Fish muscle Description Fish Muscle 4/12/2012 Extraction Date 12-Jul-13 Analysis Date 19-Jul-13

	Level ng/g	Labelled Surrogate recovery	
PFPeA PFHxA	<0.5 <0.5	80	
PFHpA PFOA	<0.5 <0.5	79	
PFNA	<1	10	þ
PFDA	6.2	61	
PFUdA	24	43	
PFDoA	<2	14	þ
PFOS	8000	17	þ
6:2 FTS	4.3	69	

#### Laboratory Reg. No. N13/017969X

Client Sample Ref. PFM18 Matrix Fish muscle Description Fish Muscle 4/12/2012 Extraction Date 12-Jul-13 Analysis Date 19-Jul-13

	Level ng/g	Labelled Surrogate recovery	
PFPeA PFHxA	<0.5 <0.5	80	
PFHpA PFOA	<0.5 <0.5	73	
PFNA	<1	9	þ
PFDA	6.5	52	
PFUdA	20	37	
PFDoA	<2	12	þ
PFOS	7300	16	þ
6:2 FTS	4.2	63	

#### Laboratory Reg. No. N13/017970X

Client Sample Ref. PFM19 Matrix Fish muscle Description Fish Muscle 4/12/2012 Extraction Date 12-Jul-13 Analysis Date 19-Jul-13

	Level ng/g	Labelled Surrogate recovery	
PFPeA PFHxA	<0.5 <0.5	79	
PFHpA PFOA	<0.5 <0.5	67	
PFNA	<2	9	þ
PFDA	5.4	44	
PFUdA	14	26	
PFDoA	<2	8	þ
PFOS	5500	17	þ
6:2 FTS	5.3	66	

#### Laboratory Reg. No. N13/017971X

### Client Sample Ref. PFM20 Matrix Fish muscle Description Fish Muscle 4/12/2012 Extraction Date 12-Jul-13 Analysis Date 19-Jul-13

	Level ng/g	Labelled Surrogate recovery	
PFPeA PFHxA	<0.5 <0.5	81	
PFHpA PFOA	<0.5 <0.5	82	
PFNA	<1	11	þ
PFDA	4.8	69	
PFUdA	17	44	
PFDoA	<2	20	þ
PFOS	5400	22	þ
6:2 FTS	4.9	70	

#### Laboratory Reg. No. N13/017972X

Client Sample Ref. PFM21 Matrix Fish muscle Description Fish Muscle 4/12/2012 Extraction Date 12-Jul-13 Analysis Date 19-Jul-13

	Level ng/g	Labelled Surrogate recovery	
PFPeA PFHxA	<0.5 <0.5	107	
PFHpA PFOA	<0.5 <0.5	100	
PFNA	<1	15	þ
PFDA	4.3	94	
PFUdA	15	69	
PFDoA	<2	31	
PFOS	5800	29	
6:2 FTS	4.4	111	

#### Laboratory Reg. No. BLK L884

Client Sample Ref. Lab Blank Matrix Lab Blank Description Lab Blank Extraction Date 12-Jul-13 Analysis Date 19-Jul-13

	Level ng/g	Labelled Surrogate recovery	
PFPeA PFHxA	<0.5 <0.5	93	
PFHpA PFOA	<0.5 <0.5	55	
PFNA	<1	28	
PFDA	<0.5	9	þ
PFUdA	<2	4	þ
PFDoA	<2	2	þ
PFOS	<0.5	20	þ
6:2 FTS	<0.5	25	



**National Measurement Institute** 

CERTIFICATE OF ANALYSIS # DAU13_153					
Client	Cardno Ecology Lab	Job No.	CARD20/130711		
	L9, 203 Pacific Highway, St I	_eonards			
	NSW 2065	Sampled by	Client		
		Date Sampled	not specified		
Contact	Marcus Lincoln-Smith	Date Received	18-Dec-2012		
The results relate only to the sample(s) tested.					

Method	AUTL_07	Date Reported	23-Jul-2013	

# **Details** The method is for determination of Perfluoroalkyl substances (PFASs) in biota samples by High Performance Liquid Chromatography tandem Mass Spectrometry (UPLC-MSMS). All results are corrected for labelled surrogates and are reported on a fresh weight basis.

Prior to extraction the sample is spiked with a range of isotopically labelled surrogate standards. Extraction is by organic solvent, with purification using activated silica gel. An aliquot of extract is injected onto the UPLC and detected using mass spectrometry.

Authorisation

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Gavin Stevenson Manager Dioxin Analysis Unit

Dr Alan Yates Senior Analyst Dioxin Analysis Unit

Sample Details : Job No. CARD20/130711				
Laboratory Reg. No.	Client Sample Ref.	Matrix	Description	
N12/034240X	CEL04	Fish muscle	Repeat of Freshwater. Dec 2012	
N12/034242X	CEL06	Fish muscle	Repeat of Freshwater. Dec 2012	
	· · ·		·	
Project Details				
Project Name	Fiskville Study			
Project Number	NA49913-034			
Key				
Analytes			Surrogate	
PFPeA	Perfluoro-n-pentanoic acid			
PFHxA	Perfluoro-n-hexanoic acid		Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]hexanoic acid Surrogate	
PFHpA	Perfluoro-n-heptanoic acid			
PFOA	Perfluoro-n-octanoic acid		Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]octanoic acid	
PFNA	Perfluoro-n-nonanoic acid		Perfluoro-n-[1,2,3,4,5- <sup>13</sup> C <sub>5</sub> ]nonanoic acid	
PFDA	Perfluoro-n-decanoic acid		Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]decanoic acid	
PFUdA	Perfluoro-n-undecanoic acid		Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]undecanoic acid	
PFDoA	Perfluoro-n-dodecanoic acid		Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]dodecanoic acid	
PFOS	Perfluoro-n-octanesulfonate		Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]octanesulfonate	
6:2 FTS	1H,1H,2H,2H-perfluoro-n-oc	tane sulfonate	1H,1H,2H,2H-perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]octane sulfonate	
Units & Abbreviations	; ;			
ng/g	nanograms per gram			
< n.	level less than limit of reporting (LC	,	05%()	
þ.	surrogate recovery outside normal	method range (25-1	25%)	

#### Laboratory Reg. No. N12/034240X

Client Sample Ref. CEL04 Matrix Fish muscle Description Repeat of Freshwater. Dec 2012 Extraction Date 12-Jul-13 Analysis Date 19-Jul-13

	Level ng/g	Labelled Surrogate recovery	
PFPeA PFHxA	<0.5 <0.5	88	
PFHpA PFOA	<0.5 <0.5	58	
PFNA	<1	10	þ
PFDA	10	20	þ
PFUdA	43	12	þ
PFDoA	3.2	6	þ
PFOS	15000	13	þ
6:2 FTS	4.7	35	

#### Laboratory Reg. No. N12/034242X

Client Sample Ref. CEL06 Matrix Fish muscle Description Repeat of Freshwater. Dec 2012 Extraction Date 12-Jul-13 Analysis Date 19-Jul-13

	Level ng/g	Labelled Surrogate recovery	
PFPeA PFHxA	<0.5 <0.5	86	
PFHpA PFOA	<0.5 <0.5	63	
PFNA	<1	11	þ
PFDA	12	34	
PFUdA	40	20	þ
PFDoA	3.5	8	þ
PFOS	15000	18	þ
6:2 FTS	3.3	41	



**National Measurement Institute** 

CERTIFICATE OF ANALYSIS # DAU13_117					
Client	Cardno Ecology Lab	Job No.	CARD20/130527		
	L9, 203 Pacific Highway, St Leonards NSW 2065 Sampled by Client				
		Date Sampled			
Contact	Marcus Lincoln-Smith	Date Received	27-May-2013		
	The results relate only to the sample(s) tested.				

Method	AUTL_07	Date Reported	21-Jun-2013

# **Details** The method is for determination of Perfluoroalkyl substances (PFASs) in biota samples by High Performance Liquid Chromatography tandem Mass Spectrometry (UPLC-MSMS). All results are corrected for labelled surrogates and are reported on a fresh weight basis.

Prior to extraction the sample is spiked with a range of isotopically labelled surrogate standards. Extraction is by organic solvent, with purification using activated silica gel. An aliquot of extract is injected onto the UPLC and detected using mass spectrometry.

Authorisation

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Gavin Stevenson Manager Dioxin Analysis Unit

Dr Alan Yates Senior Analyst Dioxin Analysis Unit

Sample Details : Job No. CARD20/130527				
Laboratory Reg. No.	Client Sample Ref.	Matrix	Description	
N13/014211X N13/014212X	CEL035 CEL037	Fish muscle Fish muscle	Fish Muscle 27/03/2013 Fish Muscle 27/03/2013	
N13/014212X	CEL037 CEL039	Fish muscle	Fish Muscle 27/03/2013	
N13/014213X	CEL039 CEL041	Fish muscle	Fish Muscle 27/03/2013	
N13/014209X SPK	Spike	Fish muscle	Spiked sample (21 ng/g, 17 ng/g for 6:2FTS)	
Project Details				
Project Name	Fiskville Study			
Project Number	NA49913-034			
Key				
Analytes			Surrogate	
	Perfluoro-n-butanoic acid		Surrogate Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]butanoic acid	
Analytes	Perfluoro-n-butanoic acid Perfluoro-n-pentanoic acid		Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]butanoic acid	
Analytes PFBA PFPeA PFHxA	Perfluoro-n-pentanoic acid Perfluoro-n-hexanoic acid			
Analytes PFBA PFPeA	Perfluoro-n-pentanoic acid		Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]butanoic acid Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]hexanoic acid Surrogate	
Analytes PFBA PFPeA PFHxA	Perfluoro-n-pentanoic acid Perfluoro-n-hexanoic acid		Perfluoro-n- $[1,2,3,4^{-13}C_4]$ butanoic acid Perfluoro-n- $[1,2^{-13}C_2]$ hexanoic acid Surrogate Perfluoro-n- $[1,2,3,4^{-13}C_4]$ octanoic acid	
Analytes PFBA PFPeA PFHxA PFHpA	Perfluoro-n-pentanoic acid Perfluoro-n-hexanoic acid Perfluoro-n-heptanoic acid		Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]butanoic acid Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]hexanoic acid Surrogate	
Analytes PFBA PFPeA PFHxA PFHpA PFOA	Perfluoro-n-pentanoic acid Perfluoro-n-hexanoic acid Perfluoro-n-heptanoic acid Perfluoro-n-octanoic acid		Perfluoro-n- $[1,2,3,4^{-13}C_4]$ butanoic acid Perfluoro-n- $[1,2^{-13}C_2]$ hexanoic acid Surrogate Perfluoro-n- $[1,2,3,4^{-13}C_4]$ octanoic acid	
Analytes PFBA PFPeA PFHxA PFHpA PFOA PFNA	Perfluoro-n-pentanoic acid Perfluoro-n-hexanoic acid Perfluoro-n-heptanoic acid Perfluoro-n-octanoic acid Perfluoro-n-nonanoic acid		Perfluoro-n- $[1,2,3,4-^{13}C_4]$ butanoic acid Perfluoro-n- $[1,2-^{13}C_2]$ hexanoic acid Surrogate Perfluoro-n- $[1,2,3,4-^{13}C_4]$ octanoic acid Perfluoro-n- $[1,2,3,4,5-^{13}C_5]$ nonanoic acid	
Analytes PFBA PFPeA PFHxA PFHpA PFOA PFNA PFDA	Perfluoro-n-pentanoic acid Perfluoro-n-hexanoic acid Perfluoro-n-heptanoic acid Perfluoro-n-octanoic acid Perfluoro-n-nonanoic acid Perfluoro-n-decanoic acid	ie	Perfluoro-n- $[1,2,3,4^{-13}C_4]$ butanoic acid Perfluoro-n- $[1,2^{-13}C_2]$ hexanoic acid Surrogate Perfluoro-n- $[1,2,3,4^{-13}C_4]$ octanoic acid Perfluoro-n- $[1,2,3,4,5^{-13}C_5]$ nonanoic acid Perfluoro-n- $[1,2^{-13}C_2]$ decanoic acid	
Analytes PFBA PFPeA PFHxA PFHpA PFOA PFNA PFDA PFOS	Perfluoro-n-pentanoic acid Perfluoro-n-hexanoic acid Perfluoro-n-heptanoic acid Perfluoro-n-octanoic acid Perfluoro-n-nonanoic acid Perfluoro-n-decanoic acid Perfluoro-n-octanesulfonat 1H,1H,2H,2H-perfluoro-n-o	ie	Perfluoro-n-[1,2,3,4- $^{13}C_4$ ]butanoic acidPerfluoro-n-[1,2- $^{13}C_2$ ]hexanoic acid SurrogatePerfluoro-n-[1,2,3,4- $^{13}C_4$ ]octanoic acidPerfluoro-n-[1,2,3,4,5- $^{13}C_5$ ]nonanoic acidPerfluoro-n-[1,2- $^{13}C_2$ ]decanoic acidPerfluoro-n-[1,2,3,4- $^{13}C_4$ ]octanesulfonate	
Analytes PFBA PFPeA PFHxA PFHpA PFOA PFNA PFDA PFOS 6:2 FTS	Perfluoro-n-pentanoic acid Perfluoro-n-hexanoic acid Perfluoro-n-heptanoic acid Perfluoro-n-octanoic acid Perfluoro-n-nonanoic acid Perfluoro-n-decanoic acid Perfluoro-n-octanesulfona 1H,1H,2H,2H-perfluoro-n-o	te octane sulfonate	Perfluoro-n-[1,2,3,4- $^{13}C_4$ ]butanoic acidPerfluoro-n-[1,2- $^{13}C_2$ ]hexanoic acid SurrogatePerfluoro-n-[1,2,3,4- $^{13}C_4$ ]octanoic acidPerfluoro-n-[1,2,3,4,5- $^{13}C_5$ ]nonanoic acidPerfluoro-n-[1,2- $^{13}C_2$ ]decanoic acidPerfluoro-n-[1,2,3,4- $^{13}C_4$ ]octanesulfonate	
Analytes PFBA PFPeA PFHxA PFHpA PFOA PFNA PFDA PFDS 6:2 FTS Units & Abbreviations	Perfluoro-n-pentanoic acid Perfluoro-n-hexanoic acid Perfluoro-n-heptanoic acid Perfluoro-n-octanoic acid Perfluoro-n-nonanoic acid Perfluoro-n-decanoic acid Perfluoro-n-octanesulfonat 1H,1H,2H,2H-perfluoro-n-o	te octane sulfonate (LOR)	Perfluoro-n-[1,2,3,4- $^{13}C_4$ ]butanoic acid Perfluoro-n-[1,2- $^{13}C_2$ ]hexanoic acid Surrogate Perfluoro-n-[1,2,3,4- $^{13}C_4$ ]octanoic acid Perfluoro-n-[1,2,3,4,5- $^{13}C_5$ ]nonanoic acid Perfluoro-n-[1,2- $^{13}C_2$ ]decanoic acid Perfluoro-n-[1,2,3,4- $^{13}C_4$ ]octanesulfonate 1H,1H,2H,2H-perfluoro-n-[1,2- $^{13}C_2$ ]octane sulfonate	

#### Laboratory Reg. No. N13/014211X

Client Sample Ref. CEL035 Matrix Fish muscle Description Fish Muscle 27/03/2013 Extraction Date 3-Jun-13 Analysis Date 14-Jun-13

	Level ng/g	Labelled Surrogate recovery	
PFBA	<2	23	þ
PFPeA PFHxA	<0.5 <0.5	57	
PFHpA PFOA	<0.5 <0.5	39	
PFNA	<0.5	29	
PFDA	<0.5	10	þ
PFOS	<1	16	þ
6:2 FTS	<0.1	61	

#### Laboratory Reg. No. N13/014212X

Client Sample Ref. CEL037 Matrix Fish muscle Description Fish Muscle 27/03/2013 Extraction Date 3-Jun-13 Analysis Date 14-Jun-13

	Level ng/g	Labelled Surrogate recovery	
PFBA	<2	24	þ
PFPeA PFHxA	<0.5 <0.5	66	
PFHpA PFOA	<0.5 <0.5	51	
PFNA	<0.5	47	
PFDA	<0.5	26	
PFOS	<1	30	
6:2 FTS	<0.1	75	

#### Laboratory Reg. No. N13/014213X

Client Sample Ref. CEL039 Matrix Fish muscle Description Fish Muscle 27/03/2013 Extraction Date 3-Jun-13 Analysis Date 14-Jun-13

	Level ng/g	Labelled Surrogate recovery	
PFBA	<2	22	þ
PFPeA PFHxA	<0.5 <0.5	63	
PFHpA PFOA	<0.5 <0.5	41	
PFNA	<0.5	42	
PFDA	<0.5	17	þ
PFOS	<1	23	þ
6:2 FTS	<0.1	72	

#### Laboratory Reg. No. N13/014214X

Client Sample Ref. CEL041 Matrix Fish muscle Description Fish Muscle 27/03/2013 Extraction Date 3-Jun-13 Analysis Date 14-Jun-13

	Level ng/g	Labelled Surrogate recovery	
PFBA	<2	24	þ
PFPeA PFHxA	<0.5 <0.5	67	
PFHpA PFOA	<0.5 <0.5	49	
PFNA	<0.5	44	
PFDA	<0.5	21	þ
PFOS	<1	30	
6:2 FTS	<0.1	56	

#### Laboratory Reg. No. N13/014209X SPK

Client Sample Ref. Spike Matrix Fish muscle Description Spiked sample (21 ng/g, 17 ng/g for 6:2FTS) Extraction Date 3-Jun-13 Analysis Date 14-Jun-13

	Level ng/g	Labelled Surrogate recovery	
PFBA	29	17	þ
PFPeA PFHxA	31 32	45	
PFHpA PFOA	41 27	35	
PFNA	29	6	þ
PFDA	39	17	þ
PFOS	7140	11	þ
6:2 FTS	22	50	



**National Measurement Institute** 

CERTIFICATE OF ANALYSIS # DAU13_118			
Client	Cardno Ecology Lab L9, 203 Pacific Highway, St L	Job No.	CARD20/130527
	NSW 2065	Sampled by	Client
Contact	Marcus Lincoln-Smith	Date Sampled Date Received	27-Mar-2013 27-May-2013
The results relate only to the sample(s) tested.			

Method	AUTL_07	Date Reported	19-Jun-2013

# **Details** The method is for determination of Perfluoroalkyl substances (PFASs) in biota samples by High Performance Liquid Chromatography tandem Mass Spectrometry (UPLC-MSMS). All results are corrected for labelled surrogates and are reported on a fresh weight basis.

Prior to extraction the sample is spiked with a range of isotopically labelled surrogate standards. Extraction is by organic solvent, with purification using activated silica gel. An aliquot of extract is injected onto the UPLC and detected using mass spectrometry.

Authorisation

aun

Gavin Stevenson Manager Dioxin Analysis Unit

Dr Alan Yates Senior Analyst Dioxin Analysis Unit

Sample Details : Job No. CARD20/130527			
Laboratory Reg. No.	Client Sample Ref.	Matrix	Description
N13/014215X	CEL043	Fish muscle	Fish Muscle 27/03/2013
N13/014216X	CEL045	Fish muscle	Fish Muscle 27/03/2013
N13/014217X	CEL047	Fish muscle	Fish Muscle 27/03/2013
N13/014218X	CEL049	Fish muscle	Fish Muscle 27/03/2013
N13/014219X	CEL064	Fish muscle	Fish Muscle 27/03/2013
N13/014220X	CEL072	Fish muscle	Fish Muscle 27/03/2013
N13/014221X	CEL076	Fish muscle	Fish Muscle 27/03/2013
N13/014222X	CEL053	Fish muscle	Fish Muscle 27/03/2013
N13/014223X	CEL055	Fish muscle	Fish Muscle 27/03/2013
BLK	Lab Blank	Lab Blank	Lab Blank

### **Project Details**

Project Name Project Number Fiskville Study NA49913-034

Key		
Analytes		Surrogate
PFBA	Perfluoro-n-butanoic acid	Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]butanoic acid
PFPeA	Perfluoro-n-pentanoic acid	
PFHxA	Perfluoro-n-hexanoic acid	Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]hexanoic acid Surrogate
PFHpA	Perfluoro-n-heptanoic acid	
PFOA	Perfluoro-n-octanoic acid	Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]octanoic acid
PFNA	Perfluoro-n-nonanoic acid	Perfluoro-n-[1,2,3,4,5- <sup>13</sup> C <sub>5</sub> ]nonanoic acid
PFDA	Perfluoro-n-decanoic acid	Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]decanoic acid
PFOS	Perfluoro-n-octanesulfonate	Perfluoro-n-[1,2,3,4-13C4]octanesulfonate
6:2 FTS	1H,1H,2H,2H-perfluoro-n-octane sulfonate	1H,1H,2H,2H-perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]octane sulfonate
Units & Abbrevia	ations	
ng/g	nanograms per gram	
<	level less than limit of reporting (LOR)	
þ	surrogate recovery outside normal method range (25-	125%)

#### Laboratory Reg. No. N13/014215X

Client Sample Ref. CEL043 Matrix Fish muscle Description Fish Muscle 27/03/2013 Extraction Date 14-May-13 Analysis Date 22-May-13

	Level ng/g	Labelled Surrogate recovery	
PFBA	<2	17	þ
PFPeA PFHxA	<0.5 <0.5	49	
PFHpA PFOA	<0.5 <0.5	34	
PFNA	<0.2	27	
PFDA	<0.5	9	þ
PFOS	<1	18	þ
6:2 FTS	<0.1	39	

#### Laboratory Reg. No. N13/014216X

Client Sample Ref. CEL045 Matrix Fish muscle Description Fish Muscle 27/03/2013 Extraction Date 14-May-13 Analysis Date 22-May-13

	Level ng/g	Labelled Surrogate recovery	
PFBA	<2	17	þ
PFPeA PFHxA	<0.5 <0.5	48	
PFHpA PFOA	<0.5 <0.5	37	
PFNA	<0.2	32	
PFDA	<0.5	12	þ
PFOS	<1	22	þ
6:2 FTS	<0.1	38	

#### Laboratory Reg. No. N13/014217X

Client Sample Ref. CEL047 Matrix Fish muscle Description Fish Muscle 27/03/2013 Extraction Date 14-May-13 Analysis Date 22-May-13

	Level ng/g	Labelled Surrogate recovery	
PFBA	<2	17	þ
PFPeA PFHxA	<0.5 <0.5	42	
PFHpA PFOA	<0.5 <0.5	27	
PFNA	<0.2	22	þ
PFDA	<0.5	8	þ
PFOS	2.1	14	þ
6:2 FTS	<0.1	39	

#### Laboratory Reg. No. N13/014218X

Client Sample Ref. CEL049 Matrix Fish muscle Description Fish Muscle 27/03/2013 Extraction Date 14-May-13 Analysis Date 22-May-13

	Level ng/g	Labelled Surrogate recovery	
PFBA	<2	18	þ
PFPeA PFHxA	<0.5 <0.5	44	
PFHpA PFOA	<0.5 <0.5	39	
PFNA	<0.2	40	
PFDA	<0.5	19	þ
PFOS	1.1	24	þ
6:2 FTS	<0.1	90	

#### Laboratory Reg. No. N13/014219X

Client Sample Ref. CEL064 Matrix Fish muscle Description Fish Muscle 27/03/2013 Extraction Date 14-May-13 Analysis Date 22-May-13

	Level ng/g	Labelled Surrogate recovery	
PFBA	<2	19	þ
PFPeA PFHxA	<0.5 <0.5	46	
PFHpA PFOA	<0.5 <0.5	31	
PFNA	<0.2	25	
PFDA	<0.5	7	þ
PFOS	<1	13	þ
6:2 FTS	<0.1	28	

#### Laboratory Reg. No. N13/014220X

Client Sample Ref. CEL072 Matrix Fish muscle Description Fish Muscle 27/03/2013 Extraction Date 14-May-13 Analysis Date 22-May-13

	Level ng/g	Labelled Surrogate recovery	
PFBA	<2	18	þ
PFPeA PFHxA	<0.5 <0.5	45	
PFHpA PFOA	<0.5 <0.5	38	
PFNA	<0.2	39	
PFDA	<0.5	17	þ
PFOS	<1	25	
6:2 FTS	<0.1	56	

#### Laboratory Reg. No. N13/014221X

Client Sample Ref. CEL076 Matrix Fish muscle Description Fish Muscle 27/03/2013 Extraction Date 14-May-13 Analysis Date 22-May-13

	Level ng/g	Labelled Surrogate recovery	
PFBA	<2	19	þ
PFPeA PFHxA	<0.5 <0.5	50	
PFHpA PFOA	<0.5 <0.5	36	
PFNA	<0.2	37	
PFDA	<0.5	18	þ
PFOS	<1	25	
6:2 FTS	<0.1	65	

#### Laboratory Reg. No. N13/014222X

Client Sample Ref. CEL053 Matrix Fish muscle Description Fish Muscle 27/03/2013 Extraction Date 14-May-13 Analysis Date 22-May-13

	Level ng/g	Labelled Sur recove	
PFBA	<2	16	þ
PFPeA PFHxA	<0.5 <0.5	46	
PFHpA PFOA	<0.5 <0.5	36	
PFNA	<0.2	30	
PFDA	<0.5	14	þ
PFOS	2.3	20	þ
6:2 FTS	<0.1	37	

#### Laboratory Reg. No. N13/014223X

Client Sample Ref. CEL055 Matrix Fish muscle Description Fish Muscle 27/03/2013 Extraction Date 14-May-13 Analysis Date 22-May-13

	Level ng/g	Labelled Sur recove	-
PFBA	<2	20	þ
PFPeA PFHxA	<0.5 <0.5	45	
PFHpA PFOA	<0.5 <0.5	25	
PFNA	<0.2	19	þ
PFDA	<0.5	6	þ
PFOS	2.0	9	þ
6:2 FTS	<0.1	48	

#### Laboratory Reg. No. BLK

Client Sample Ref. Lab Blank Matrix Lab Blank Description Lab Blank Extraction Date 14-May-13 Analysis Date 22-May-13

	Level ng/g	Labelled Sur recove	
PFBA	<2	17	þ
PFPeA PFHxA	<0.5 <0.5	45	
PFHpA PFOA	<0.5 <0.5	26	
PFNA	<0.2	15	þ
PFDA	<0.5	4	þ
PFOS	<1	7	þ
6:2 FTS	<0.1	21	þ



**National Measurement Institute** 

CERTIFICATE OF ANALYSIS # DAU13_119			
Client	Cardno Ecology Lab L9, 203 Pacific Highway, St I	Job No.	CARD20/130527
	NSW 2065	Sampled by	Client
			27/28-Mar-2013
Contact	Marcus Lincoln-Smith	Date Received	27-May-2013
The results relate only to the sample(s) tested.			

Method	AUTL_07	Date Reported	19-Jun-2013

# **Details** The method is for determination of Perfluoroalkyl substances (PFASs) in biota samples by High Performance Liquid Chromatography tandem Mass Spectrometry (UPLC-MSMS). All results are corrected for labelled surrogates and are reported on a fresh weight basis.

Prior to extraction the sample is spiked with a range of isotopically labelled surrogate standards. Extraction is by organic solvent, with purification using activated silica gel. An aliquot of extract is injected onto the UPLC and detected using mass spectrometry.

Authorisation

aun

Gavin Stevenson Manager Dioxin Analysis Unit

Dr Alan Yates Senior Analyst Dioxin Analysis Unit

Sample Details : Job No. CARD20/130527				
Laboratory Reg. No.	Client Sample Ref.	Matrix	Description	
N13/014224X N13/014225X N13/014226X N13/014219X SPK	CEL057 CEL094 CEL096 Spike	Fish muscle Fish muscle Fish muscle Fish muscle	Fish Muscle 27/03/2013 Fish Muscle 28/03/2013 Fish Muscle 28/03/2013 Spiked sample (21 ng/g, 17 ng/g for 6:2FTS)	
Project Details Project Name Project Number Key	Fiskville Study NA49913-034			
Analytes			Surrogate	
PFBA	Perfluoro-n-butanoic acid		Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]butanoic acid	
PFPeA PFHxA	Perfluoro-n-pentanoic acio Perfluoro-n-hexanoic acid	I	Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]hexanoic acid Surrogate	
PFHpA PFOA	Perfluoro-n-heptanoic acio Perfluoro-n-octanoic acid	I	Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]octanoic acid	
PFNA	Perfluoro-n-nonanoic acid		Perfluoro-n-[1,2,3,4,5- <sup>13</sup> C <sub>5</sub> ]nonanoic acid	
PFDA	Perfluoro-n-decanoic acid		Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]decanoic acid	
PFOS	Perfluoro-n-octanesulfonate		Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]octanesulfonate	
6:2 FTS	1H,1H,2H,2H-perfluoro-n-octane sulfonate		1H,1H,2H,2H-perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]octane sulfonate	
Units & Abbreviations				
ng/g				
<b>८</b>				

#### Laboratory Reg. No. N13/014224X

Client Sample Ref. CEL057 Matrix Fish muscle Description Fish Muscle 27/03/2013 Extraction Date 14-May-13 Analysis Date 22-May-13

	Level ng/g	Labelled Su recove	
PFBA	<2	21	þ
PFPeA PFHxA	<0.5 <0.5	45	
PFHpA PFOA	<0.5 <0.5	38	
PFNA	<0.2	38	
PFDA	<0.5	15	þ
PFOS	2.6	21	þ
6:2 FTS	<0.1	89	

#### Laboratory Reg. No. N13/014225X

Client Sample Ref. CEL094 Matrix Fish muscle Description Fish Muscle 28/03/2013 Extraction Date 14-May-13 Analysis Date 22-May-13

	Level ng/g	Labelled Sur recove	
PFBA	<2	19	þ
PFPeA PFHxA	<0.5 <0.5	45	
PFHpA PFOA	<0.5 <0.5	34	
PFNA	<0.2	29	
PFDA	<0.5	11	þ
PFOS	5.9	14	þ
6:2 FTS	<0.1	80	

#### Laboratory Reg. No. N13/014226X

Client Sample Ref. CEL096 Matrix Fish muscle Description Fish Muscle 28/03/2013 Extraction Date 14-May-13 Analysis Date 22-May-13

	Level ng/g	Labelled Sur recove	
PFBA	<2	16	þ
PFPeA PFHxA	<0.5 <0.5	43	
PFHpA PFOA	<0.5 <0.5	30	
PFNA	<0.2	20	þ
PFDA	<0.5	6	þ
PFOS	2.2	8	þ
6:2 FTS	<0.1	45	

#### Laboratory Reg. No. N13/014219X SPK

Client Sample Ref. Spike Matrix Fish muscle Description Spiked sample (21 ng/g, 17 ng/g for 6:2FTS) Extraction Date 14-May-13 Analysis Date 22-May-13

	Level ng/g	Labelled Sur recove	
PFBA	28	15	þ
PFPeA PFHxA	32 34	36	
PFHpA PFOA	40 25	29	
PFNA	24	25	
PFDA	30	10	þ
PFOS	28	15	þ
6:2 FTS	18	32	

# Appendix F 38 Pages

# Rabbit Sampling and QA/QC



# HUMAN HEALTH RISK ASSESSMENT – FISKVILLE COMMUNITY

# 4549 GEELONG-BALLAN RD, FISKVILLE VICTORIA

# **APPENDIX F**

# **RABBIT DATA COLLECTION**

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# **APPENDIX F - RABBIT DATA COLLECTION**

# **1** INTRODUCTION

This summary is intended to provide a description of the rabbit sampling conducted by Cardno Ecology Lab, Sydney NSW (Cardno Eco) at CFA Fiskville Training College, Fiskville Vic (the "Site"). The field work was conducted as per proposal reference 212163.18Proposal01.2 (dated 18 April 2013) under the instructions of Cardno Eco. This summary does not have nor provides any discussions with regards to results or corresponding criteria for the rabbit data as these are addressed in the main body of the report.

# 1.1 Objective

The collection of rabbits and the analysis for the presence of Perfluoro Compounds (PFCs) in muscle tissue was in order to assess the livestock pathway as part of the Human Health Risk Assessment of the Fiskville Community.

# **1.2 Sampling Event and Sample Locations**

The field event was conducted on 4 May 2013. A total of 10 rabbits were culled by a professional shooter as per the Department of Primary Industries (DPI) guidelines<sup>1</sup>. The approximate locations for the rabbits collected are shown in Figure 1-1. The corresponding sample identification number and approximate georeferenced locations are provided in Table 1-1.



Figure 1-1: Rabbit Sample Locations

<sup>&</sup>lt;sup>1</sup> Department of Primary Industries RAB009 Ground Shooting of Rabbits, date if issue 1 October 2004.



•						
Sample ID	Easting <sup>1</sup> (m)	Northing (m)				
065-RA1	254708	5825458				
066-RA2	254686	5825572				
067-RA3	254593	5825592				
068-RA4	254708	5825513				
069-RA5&6	254897	5825530				
070-RA7	254789	5825554				
071-RA8	254701	5825697				
074-RA9	254705	5825533				
073-RA10	254694	5825515				
Notes: 1. UTM Zone 55 (MGA94) and all decimal units rounded to metre. The GPS system reports an error or ± 5 m.						

#### Table 1-1: Sample Location ID and Georeferenced Positions

The rabbits were all collected in the training area. A search of other areas around the site was conducted including around Lake Fiskville; however, rabbits were not encountered there. It is not clear why the rabbits were located in the training area except that Cardno Eco noted that:

- There appeared to be a lack of feed for rabbits in the vicinity of Lake Fiskville;
- The terrain in the training area is suitable for rabbits as it contains embankments, greenery (e.g. plants) and moist soils; and
- The terrain around the lake contains dry hard soils and minimal embankments. This is not ideal for rabbits to burrow.

# 2 RABBIT SAMPLING

# 2.1 Sample Strategy

The scope and method of the sampling event was prepared by Cardno Eco. The samples were collected on-site, placed on ice and transported to Sydney, NSW. The dissection and biometric measurements were conducted on the 7 May 2013. Samples were weighed, labelled and frozen. A summary of muscle samples collated by Cardno Eco is provided in Table 2-1.

			-
Sample number	Total Weight (g)	Sample 1 Weight (g)	Sample 2 Weight (g)
RA1	1,335.2	60.4	60.1
RA2	1,404.3	45.6	33.8
RA3	1,557.5	49.4	48.2
RA4	1,500.7	59.1	49.5
RA5	1,666.2	59.2	50.1
RA6	1,727.0	55.8	52.0
RA7	1,425.4	45.8	45.4

## Table 2-1: Rabbit Sample Weight Summary



Sample number	Total Weight (g)	Sample 1 Weight (g)	Sample 2 Weight (g)
RA8	1,743.5	53.1	57.0
RA9	1,969.1	63.0	64.5
RA10	1,418.1	39.0	38.5

Two muscle tissue samples was taken from each specimen as shown in Table 2-1 (e.g. Sample 1 and Sample 2), in order to conduct a laboratory analysis at different laboratories (inter-laboratory) discussed in Section 2.2.

# 2.2 Laboratory Analysis

The rabbit samples were analysed by two laboratories as follows:

- 1. National Measurement Institute (NMI), Sydney NSW, was the primary laboratory; and
- 2. Asure Quality (AQ), Wellington NZ, was the secondary laboratory for Quality Control (QC).

The analytical suite was for the Contaminant of Potential Concern (CoPC) taking into account the extended Perfluoro Compounds (PFCs) that are present in firefighting foams or breakdown products. The main PFCs analysed by both laboratories and included in this review were: PFPeA, PFHxS, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFDS, PFUdA, PFDoA, PFOS, 6:2 FtS and 8:2 FtS.

An initial muscle analysis was conducted by NMI on 4 samples RA1-1, RA2-1, RA5-1 and RA8-1, 13 May 2013. The second batch analysis, which included the inter-laboratory analysis was conducted

Copies of the corresponding laboratory reports and sample receipt records are included in Attachment A. The Quality Assurance and Quality Control (QA/QC) for the rabbit analysis program is discussed in Section 3. Tabulated data for all laboratory results is provided in Attachment B.

# 3 QUALITY ASSURANCE AND QUALITY CONTROL REVIEW

## 3.1 Intra-Laboratory Analysis - NMI

Two samples, RA6-1 and RA8-1 were selected by NMI to assess the intra-laboratory reproducibility of the analysis. The duplicate samples are analysed concurrently with the parent sample. The Relative Percentage Difference (RPD) calculated from the parent samples (i.e. SS69 and SS78 respectively) are provided in Table 3-1.

#### Table 3-1: %RPD Calculation for Intra-laboratory Assessment – NMI (Units ng/g)

ID	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUdA	PFDoA	PFOS	6:2 FTS
RA6-1	0.83	<0.5	<0.5	<0.5	<0.5	<0.5	<1	<0.5	220	<0.5
RA6-1D <sup>1</sup>	0.46	<0.5	<0.5	<0.5	<0.5	<0.5	<1	<0.5	220	<0.5
%RPD	<u>57.4</u>	<lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td>0.0</td><td><lor< td=""></lor<></td></lor<></td></lor<></td></lor<></td></lor<></td></lor<></td></lor<></td></lor<>	<lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td>0.0</td><td><lor< td=""></lor<></td></lor<></td></lor<></td></lor<></td></lor<></td></lor<></td></lor<>	<lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td>0.0</td><td><lor< td=""></lor<></td></lor<></td></lor<></td></lor<></td></lor<></td></lor<>	<lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td>0.0</td><td><lor< td=""></lor<></td></lor<></td></lor<></td></lor<></td></lor<>	<lor< td=""><td><lor< td=""><td><lor< td=""><td>0.0</td><td><lor< td=""></lor<></td></lor<></td></lor<></td></lor<>	<lor< td=""><td><lor< td=""><td>0.0</td><td><lor< td=""></lor<></td></lor<></td></lor<>	<lor< td=""><td>0.0</td><td><lor< td=""></lor<></td></lor<>	0.0	<lor< td=""></lor<>
RA8-1	2.2	0.58	<0.5	<0.5	<0.5	0.93	<1	<0.5	380	<0.5



ID	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUdA	PFDoA	PFOS	6:2 FTS
RA8-1D <sup>1</sup>	3.2	0.88	<0.5	<0.5	<0.5	1.2	<1	<0.5	510	<0.5
%RPD	-37.0	-41.1	<lor< td=""><td><lor< td=""><td><lor< td=""><td>-25.4</td><td><lor< td=""><td><lor< td=""><td>-29.2</td><td>-37.0</td></lor<></td></lor<></td></lor<></td></lor<></td></lor<>	<lor< td=""><td><lor< td=""><td>-25.4</td><td><lor< td=""><td><lor< td=""><td>-29.2</td><td>-37.0</td></lor<></td></lor<></td></lor<></td></lor<>	<lor< td=""><td>-25.4</td><td><lor< td=""><td><lor< td=""><td>-29.2</td><td>-37.0</td></lor<></td></lor<></td></lor<>	-25.4	<lor< td=""><td><lor< td=""><td>-29.2</td><td>-37.0</td></lor<></td></lor<>	<lor< td=""><td>-29.2</td><td>-37.0</td></lor<>	-29.2	-37.0
Notes: 1. RA6-1D and RA81-D refers to "Duplicate" sample										

The intra-laboratory assessment showed acceptable reproducibility with only one sample exceeding %RPD of 50% (i.e. Sample RA6-1D for PFPeA).

# 3.2 Spiked Recovery - NMI

NMI conducted a sample spiked assessment for two samples as follows:

- 1. Sample RA8-1S (Certificate No. CARD20/130513) was spiked with an internal standard with concentration of 17 ng/g for PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFDA, PFDoA and PFOS. A concentration of 14 ng/g was used for 6:2 FtS; and
- 2. Sample RA6-1S (Certificate No. CARD20/130614) was spiked with an internal standard with concentration of 19 ng/g for PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUdA, PFDoA and PFOS. A concentration of 15 ng/g was used for 6:2 FtS.

Table 3-2 provides a summary of the spiked sample calculations as comparison with the primary and duplicate samples. Overall, the spiked analysis showed good reproducibility within the two primary and duplicate samples for the corresponding batches. However, the first batch (Certificate No. CARD20/130513) for sample RA8-1 the spiked analysis reported above 110% form most analytes with PFOS and 6:2 FtS reporting 101% and 112% for the duplicate sample respectively. The second batch (Certificate No. CARD20/130614) reported lower spiked results for most analytes with the exception of PFOS and 6:2 FtS reporting 100% and 118% respectively for primary and duplicate samples.

ID	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUdA	PFDoA	PFOS	6:2 FTS
RA6-1	0.83	<0.5	<0.5	<0.5	<0.5	<0.5	<1	<0.5	220	<0.5
RA6-1D	0.46	<0.5	<0.5	<0.5	<0.5	<0.5	<1	<0.5	220	<0.5
RA6-1S <sup>2</sup>	16	15	17	15	16	16	15	10	240	18
Primary <sup>3</sup>	81%	78%	88%	78%	83%	83%	77%	52%	100%	118%
Duplicate <sup>4</sup>	82%	78%	88%	78%	83%	83%	77%	52%	100%	118%
RA8-1	2.2	0.58	<0.5	<0.5	<0.5	0.93	<1	<0.5	380	<0.5
RA8-1D	3.2	0.88	<0.5	<0.5	<0.5	1.2	<1	<0.5	510	<0.5
RA8-1S <sup>2</sup>	24	25	23	24	24	25	26	26	530	16
Primary <sup>3</sup>	125%	142%	133%	139%	139%	139%	149%	151%	134%	112%
Duplicate <sup>4</sup>	119%	140%	133%	139%	139%	137%	149%	151%	101%	112%

# Table 3-2: Spiked Recovery Calculation – NMI (Units ng/g)

Notes:

1. RA6-1D and RA81-D refers to "Duplicate" sample.

2. RA6-1S and RA8-1S refer to Spiked sample.

3. Primary – refers to the surrogate recovery per centum comparing the Parent sample RA6-1 or RA8-1 with the Spiked sample RA6-1S or RA8-1S.

4. Duplicate - refers to the surrogate recovery per centum comparing the Duplicate sample RA6-1D or RA8-1D with the Spiked sample RA6-1S or RA8-1S



The first batch could have overestimated the concentration for some analytes with the average spiked concentrations reported at 136%, while the second batch may have underestimated the concentration for some analytes with the average spiked concentrations reported at 84%.

The compounds which reported concentrations above the laboratory limit or reporting (LOR) were PFPeA, PFHxA, PFDA and PFOS.

# 3.3 Inter-Laboratory Analysis – NMI and AQ

Cardno Eco submitted three tissue samples to NMI and AQ as part of an inter-laboratory assessment. The corresponding samples were taken from the same specimen and labelled Sample 1 and Sample 2 as shown in Table 3-3.

Sample ID	Certificate Batch No.	Laboratory
RA4-1	CARD20/130614	NMI
RA4-2	134925	AQ
RA6-1	CARD20/130614	NMI
RA6-2	134925	AQ
RA9-1	CARD20/130614	NMI
RA9-2	134925	AQ

#### Table 3-3: Intra-Laboratory Sample Identification

#### Table 3-4: %RPD summary for NMI and QA Inter-laboratory Assessment (ng/g)

Sample ID	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFDoA	PFOS	6:2 FTS
RA4-1	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	150	<0.5
RA4-2	<1.0	<1.0	<2.0	<1.0	<2.0	<2.0	140	<1.0
%RPD	<lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td>6.9</td><td><lor< td=""></lor<></td></lor<></td></lor<></td></lor<></td></lor<></td></lor<></td></lor<>	<lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td>6.9</td><td><lor< td=""></lor<></td></lor<></td></lor<></td></lor<></td></lor<></td></lor<>	<lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td>6.9</td><td><lor< td=""></lor<></td></lor<></td></lor<></td></lor<></td></lor<>	<lor< td=""><td><lor< td=""><td><lor< td=""><td>6.9</td><td><lor< td=""></lor<></td></lor<></td></lor<></td></lor<>	<lor< td=""><td><lor< td=""><td>6.9</td><td><lor< td=""></lor<></td></lor<></td></lor<>	<lor< td=""><td>6.9</td><td><lor< td=""></lor<></td></lor<>	6.9	<lor< td=""></lor<>
<u>RA6-1</u>	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	220	<0.5
RA6-2	<1.0	<1.0	<2.0	<1.0	<2.0	<2.0	190	<1.0
%RPD	<lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td>14.6</td><td><lor< td=""></lor<></td></lor<></td></lor<></td></lor<></td></lor<></td></lor<></td></lor<>	<lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td>14.6</td><td><lor< td=""></lor<></td></lor<></td></lor<></td></lor<></td></lor<></td></lor<>	<lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td>14.6</td><td><lor< td=""></lor<></td></lor<></td></lor<></td></lor<></td></lor<>	<lor< td=""><td><lor< td=""><td><lor< td=""><td>14.6</td><td><lor< td=""></lor<></td></lor<></td></lor<></td></lor<>	<lor< td=""><td><lor< td=""><td>14.6</td><td><lor< td=""></lor<></td></lor<></td></lor<>	<lor< td=""><td>14.6</td><td><lor< td=""></lor<></td></lor<>	14.6	<lor< td=""></lor<>
RA9-1	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	150	<0.5
RA9-2	<1.0	<1.0	<2.0	<1.0	<2.0	<2.0	140	<1.0
%RPD	<lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td>6.9</td><td><lor< td=""></lor<></td></lor<></td></lor<></td></lor<></td></lor<></td></lor<></td></lor<>	<lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td>6.9</td><td><lor< td=""></lor<></td></lor<></td></lor<></td></lor<></td></lor<></td></lor<>	<lor< td=""><td><lor< td=""><td><lor< td=""><td><lor< td=""><td>6.9</td><td><lor< td=""></lor<></td></lor<></td></lor<></td></lor<></td></lor<>	<lor< td=""><td><lor< td=""><td><lor< td=""><td>6.9</td><td><lor< td=""></lor<></td></lor<></td></lor<></td></lor<>	<lor< td=""><td><lor< td=""><td>6.9</td><td><lor< td=""></lor<></td></lor<></td></lor<>	<lor< td=""><td>6.9</td><td><lor< td=""></lor<></td></lor<>	6.9	<lor< td=""></lor<>

The %RPD for PFOS shows an acceptable correlation between the two laboratories (i.e. less than 15%). The remainder of the analytes reported results below the laboratory LOR or no pair coupled available for a %RPD calculation (e.g. PFHxA, PFDS and 8:2 FtS reported by AQ and not included in the NMI suite). The data is considered acceptable since the results for PFOS has a %RPD less that 20%.



# 3.4 Laboratory Blank

Three internal laboratory blank analyses was conducted, corresponding one blank per batch and summarized in Table 3-6.

Laboratory	Reference No.	Certificate No	Comments
NMI	BLK L869	CARD20/130513	
INIVII	BLK L879	CARD20/130614	All analytes reported below the laboratory LOR
AQ	134925-BL	134925	2010 II II 0 12201 2001 y 2011

## Table 3-5: Laboratory Internal Blank Analysis

# 3.5 Sample Vial – Rinsate

Cardno Eco submitted one sample container with de-ionized water as part of quality control. The de-ionized water was analysed for the CoPC to assess potential contamination due to sample jars. All analytes reported below the laboratory LOR.

# 3.6 Summary of Rabbit Muscle Results

A summary of the rabbit results is provided in Table 3-7, with analytes reporting greater than 47% detection rate highlighted with bold numbers.

## Table 3-6: Summary of Rabbit Analysis (Units ng/g)

	PFPeA	PFHxS	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFDS	PFUdA	PFDoA	PFOS	6:2 FTS	8:2 FtS
Minimum	0.46	32	0.58	<0.5	<0.5	<0.5	0.89	1.6	<1	0.6	44	<0.5	8.6
Maximum	3.7	68	0.95	<0.5	<0.5	<0.5	1.2	2.7	<1	0.6	600	<0.5	8.6
Total <sup>1</sup>	16	4	20	20	20	20	20	4	16	20	20	20	4
Detects <sup>3</sup>	7	3	4	0	0	0	3	2	0	1	15	0	1
%detects <sup>4</sup>	47	75	27	0	0	0	20	50	0	7	100	0	25
Notes:													

1. Total number of analysis, including: Blanks, Spikes and Duplicates.

2. Sample analysis reported less than laboratory LOR.

3. Does not include spiked samples.

4. Total number of reporting above the laboratory LOR – it does not include blanks or spiked sample results.

# 4 ATTACHMENTS

Attachment A

Laboratory Reports and Chain of Custody

## Attachment B

Table B1 – QA/QC Review for Rabbit Muscle Laboratory Data

#### Cardno Lane Piper March 2014



ROLECT NAME: Analysis of freshwater blot. Will cuote Number: GS1304154 CADD3 Curr Purchase Order Number: GS1304154 CADD3 Curr Purchase Order Number: TBA Curr Job Number: MAB913-034 Sample type Rabbit Muscle × PFAS including PFOS, Rabbit Muscle × PFOAS & 6:: FTS Rabbit Muscle × PFAS including PFOS, Rabbit	OLECT NAME: Analysis of freshwater biots. Li Guode Number: GS130018A_CARDO ur Purchase Order Number: GS130018A_CARDO ur Job Number: MA9913-034 ur Job Number: MA9913-034 suits due dato (as agreed with MM): ample type PPOA5 & 6.2 FFP Rabbit Muscle X PPOA5 & 6	PROJECT NAME: Analysis of freshwater blots.         PROJECT NAME: Analysis of freshwater blots.         Ising used Number: GS130413A, CARD30         Your Job Number: GS130413A, CARD30         Your Job Number: GS130413A, CARD30         Your Job Number: Massel Sector Number: TBA         Your Job Number: Name         Results due date (ns. agreed with MM):         Results due date (ns. agreed with MM):         Amalian       Sample type         Results due date (ns. agreed with MM):         Amalian       Sample type         Results due date (ns. agreed with MM):         Ambit Mussele       X Rabbit Mussele         Resolut Mussele       X Rabbi	Dure 10/0/ SEND TO: NMI (National Measurement Institute) 105 Deihi Road, North Ryde NSW 2113 Phone: 02 9449 0111 E-mail: customerservice@measurement.gov.au NMI Contact Person: Gavin Stevenson	COMMENTS	PAGE No: 1 of 1 PAGES Imultiple pages, ensure ALL pages are stapled together
	TODY FORM	N OF CUSTODY FOR Ecology Lab Ecology Lab Ecology Lab Ecology Lab LincoinSmith 2065 E LincoinSmith 205 9496 7888 0413622086 02 9496 7888 0413622086 13 / 5 / 1 3 / 5 / 5 / 1 3 / 5 / 5	OJECT NAME: II Quote Numbe ur Purchase Or ur Job Number: sults due date (	PFAS including PFOS.	Raceived at NMI taboratory by: Print Name: Date & Time: / / ; Signature:



**National Measurement Institute** 

	CERTIFICATE OF ANALYSIS # DAU13_089						
Client	Cardno Ecology Lab	Job No.	CARD20/130513				
	Cardno Ecology LabJob No.CARD20/130513L9, 203 Pacific Highway, St LeonardsSampled byClient						
	NSW 2065	Sampled by	Client				
		Date Sampled	not specified				
Contact	Marcus Lincoln-Smith	Date Received	13-May-2013				
	The results relate only to the sample(s) tested.						
Method	AUTL_07	Date Reported	24-May-2013				

**Details** The method is for determination of Perfluoroalkyl substances (PFASs) in biota samples by High Performance Liquid Chromatography tandem Mass Spectrometry (UPLC-MSMS). All results are corrected for labelled surrogates and are reported on a fresh weight basis.

Prior to extraction the sample is spiked with a range of isotopically labelled surrogate standards. Extraction is by organic solvent, with purification using activated silica gel. An aliquot of extract is injected onto the UPLC and detected using mass spectrometry.

Authorisation

Haur

Gavin Stevenson Manager Dioxin Analysis Unit

Dr Alan Yates Senior Analyst Dioxin Analysis Unit

	Sample De	tails : Job No. C	CARD20/130513		
Laboratory Reg. No.	Client Sample Ref.	Matrix	Description		
N13/012591X	RA1-1	Rabbit muscle	Rabbit Muscle May-13		
N13/012592X	RA2-1	Rabbit muscle	Rabbit Muscle May-13		
N13/012593X	RA5-1	Rabbit muscle	Rabbit Muscle May-13		
N13/012594X	RA8-1	Rabbit muscle Rabbit muscle	Rabbit Muscle May-13		
N13/012594DUP N13/012594SPK	Duplicate Spike	Rabbit muscle	Duplicate Sample Spiked sample (17 ng/g, 14 ng/g for 6:2FTS)		
BLK L869	Lab Blank	Lab Blank	Lab Blank		
Project Details					
Project Name	Fiskville Study				
Project Number	NA49913-034				
Key					
Analytes	1		Surrogate		
PFPeA	Perfluoro-n-pentanoic acid				
PFHxA	Perfluoro-n-hexanoic acid		Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]hexanoic acid Surrogate		
PFHpA	Perfluoro-n-heptanoic acid				
PFOA	Perfluoro-n-octanoic acid		Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]octanoic acid		
PFNA	Perfluoro-n-nonanoic acid		Perfluoro-n-[1,2,3,4,5- <sup>13</sup> C <sub>5</sub> ]nonanoic acid		
PFDA	Perfluoro-n-decanoic acid		Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]decanoic acid		
PFUdA	Perfluoro-n-undecanoic acid		Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]undecanoic acid		
PFDoA	Perfluoro-n-dodecanoic acid		Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]dodecanoic acid		
PFOS	Perfluoro-n-octanesulfonate		Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]octanesulfonate		
6:2 FTS	1H,1H,2H,2H-perfluoro-n-o	octane sulfonate	1H,1H,2H,2H-perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]octane sulfonate		
Units & Abbreviations					
ng/g	nanograms per gram				
<	level less than limit of reporting	· /			
þ	surrogate recovery outside norm	nal method range (25-12	25%)		

#### Laboratory Reg. No. N13/012591X

Client Sample Ref. RA1-1 Matrix Rabbit muscle Description Rabbit Muscle May-13 Extraction Date 14-May-13 Analysis Date 22-May-13

	Level ng/g	Labelled Surrogate recovery
PFPeA PFHxA	<0.5 <0.5	49
PFHpA PFOA	<0.5 <0.5	63
PFNA	<0.5	68
PFDA	<0.5	54
PFUdA	<1	64
PFDoA	<0.5	62
PFOS	44	51
6:2 FTS	<0.5	80

#### Laboratory Reg. No. N13/012592X

Client Sample Ref. RA2-1 Matrix Rabbit muscle Description Rabbit Muscle May-13 Extraction Date 14-May-13 Analysis Date 22-May-13

	Level ng/g	Labelled Surrogate recovery
PFPeA PFHxA	<0.5 <0.5	64
PFHpA PFOA	<0.5 <0.5	79
PFNA	<0.5	63
PFDA	<0.5	74
PFUdA	<1	82
PFDoA	<0.5	79
PFOS	130	47
6:2 FTS	<0.5	68

#### Laboratory Reg. No. N13/012593X

Client Sample Ref. RA5-1 Matrix Rabbit muscle Description Rabbit Muscle May-13 Extraction Date 14-May-13 Analysis Date 22-May-13

	Level ng/g	Labelled Surrogate recovery
PFPeA PFHxA	2.4 1.0	51
PFHpA PFOA	<0.5 <0.5	66
PFNA	<0.5	39
PFDA	0.89	42
PFUdA	<1	60
PFDoA	0.60	45
PFOS	350	32
6:2 FTS	<0.5	94

#### Laboratory Reg. No. N13/012594X

Client Sample Ref. RA8-1 Matrix Rabbit muscle Description Rabbit Muscle May-13 Extraction Date 14-May-13 Analysis Date 22-May-13

	Level ng/g	Labelled Surrogate recovery
PFPeA PFHxA	2.2 0.58	61
PFHpA PFOA	<0.5 <0.5	68
PFNA	<0.5	34
PFDA	0.93	53
PFUdA	<1	71
PFDoA	<0.5	71
PFOS	380	27
6:2 FTS	<0.5	54

#### Laboratory Reg. No. N13/012594DUP

Client Sample Ref. Duplicate Matrix Rabbit muscle Description Duplicate Sample Extraction Date 14-May-13 Analysis Date 22-May-13

	Level ng/g	Labelled Surrogate recovery
PFPeA PFHxA	3.2 0.88	60
PFHpA PFOA	<0.5 <0.5	74
PFNA	<0.5	42
PFDA	1.2	68
PFUdA	<1	73
PFDoA	<0.5	64
PFOS	510	30
6:2 FTS	<0.5	69

#### Laboratory Reg. No. N13/012594SPK

Client Sample Ref. Spike Matrix Rabbit muscle Description Spiked sample (17 ng/g, 14 ng/g for 6:2FTS) Extraction Date 14-May-13 Analysis Date 22-May-13

	Level ng/g	Labelled Surrogate recovery
PFPeA PFHxA	24 25	57
PFHpA PFOA	23 24	64
PFNA	24	35
PFDA	25	52
PFUdA	26	60
PFDoA	26	56
PFOS	530	28
6:2 FTS	16	72

Laboratory Reg. No. BLK L869

Client Sample Ref. Lab Blank Matrix Lab Blank Description Lab Blank Extraction Date 14-May-13 Analysis Date 22-May-13

	Level	Labelled Surrogate
	ng/g	recovery
PFPeA	<0.5	
PFHxA	<0.5	51
PFHpA	<0.5	
PFOA	<0.5	69
PFNA	<0.5	65
PFDA	<0.5	57
PFUdA	<1	58
PFDoA	<0.5	51
PFOS	<0.5	42
6:2 FTS	<0.5	69

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Mana teoretarente Mana teoretaren autoritation Mana teoretaren autoritation Sample ID / Collection information Sample	Phone:	02 9496 7888		Results due date (as	agreed with NMI ):								E-mail:	customerservice@r	neasurement.gov.au
Total Stants     Collection Information     Collection Information       Vour Sample ID / Description / Number     Collection Information     Sample ID / (10ate & Time)       PEDA. Full     Collection Information       RA3-1     7/05/20013     Rabbit Muscle       RA3-1     7/05/20013     RADbit Muscle       RA3-1     7/05/20013     RAbbit Muscle <td< th=""><th>Contact person email:</th><th>Marcus LincoinSmith@card</th><th>no.com.au</th><th></th><th></th><th></th><th></th><th></th><th></th><th></th><th></th><th></th><th>NMI Cor</th><th>tact Person : Gavin S</th><th>Stevenson</th></td<>	Contact person email:	Marcus LincoinSmith@card	no.com.au										NMI Cor	tact Person : Gavin S	Stevenson
RA3-1         7/05/2013         Rabbit Muscle         × <td>NMI SAMPLE NUMBER (NMI USE ONLY - please do not write in this column)</td> <td>Your Sample ID / Description / Number</td> <td></td> <td>Sample type</td> <td>include other PFCs as reported in previous Iab reports i.e. PFBA, PFPeA, PFDA, PFudA, PFUA, PFDA, PFudA, PFDOA and</td> <td>noitsqer9 elqms2 elsteM</td> <td>SineerA</td> <td>muimbeO</td> <td></td> <td>2001</td> <td>1/15/70246</td> <td>Nickel</td> <td>ouiZ</td> <td></td> <td>COMMENTS</td>	NMI SAMPLE NUMBER (NMI USE ONLY - please do not write in this column)	Your Sample ID / Description / Number		Sample type	include other PFCs as reported in previous Iab reports i.e. PFBA, PFPeA, PFDA, PFudA, PFUA, PFDA, PFudA, PFDOA and	noitsqer9 elqms2 elsteM	SineerA	muimbeO		2001	1/15/70246	Nickel	ouiZ		COMMENTS
RA4-1         7105/2013         Rabbit Muscle         × </td <td>N13/015963</td> <td></td> <td>7/05/2013</td> <td>Rabbit Muscle</td> <td></td> <td>×</td> <td>×</td> <td>×</td> <td></td> <td></td> <td></td> <td>×</td> <td>×</td> <td></td> <td></td>	N13/015963		7/05/2013	Rabbit Muscle		×	×	×				×	×		
Rde-1         7/06/2013         Rabbit Muscle         × <td>N13/015964</td> <td></td> <td>7/05/2013</td> <td>Rabbit Muscle</td> <td>×</td> <td>×</td> <td>×</td> <td>×</td> <td></td> <td></td> <td></td> <td>×</td> <td>×</td> <td></td> <td></td>	N13/015964		7/05/2013	Rabbit Muscle	×	×	×	×				×	×		
RA7-1         7/05/2013         Rabbit Muscle         × <td>N13/015965</td> <td></td> <td>7/05/2013</td> <td>Rabbit Muscle</td> <td>×</td> <td>×</td> <td>×</td> <td>×</td> <td></td> <td></td> <td></td> <td>×</td> <td>×</td> <td></td> <td></td>	N13/015965		7/05/2013	Rabbit Muscle	×	×	×	×				×	×		
RA9-1         7/05/2013         Rabbit Muscle         × <td>N13/015966</td> <td></td> <td>7/05/2013</td> <td>Rabbit Muscle</td> <td>×</td> <td>×</td> <td>×</td> <td>×</td> <td></td> <td>-</td> <td>200</td> <td>×</td> <td>×</td> <td></td> <td></td>	N13/015966		7/05/2013	Rabbit Muscle	×	×	×	×		-	200	×	×		
RA10-1         7/05/2013         Rabbit Muscle         X </td <td>N13/015967</td> <td>RA9-1</td> <td>7/05/2013</td> <td>Rabbit Muscle</td> <td>×</td> <td>×</td> <td>×</td> <td>×</td> <td>- 25</td> <td></td> <td></td> <td>×</td> <td>×</td> <td></td> <td></td>	N13/015967	RA9-1	7/05/2013	Rabbit Muscle	×	×	×	×	- 25			×	×		
4     1     1     1     1     1       1     1     1     1     1     1     1       1     1     1     1     1     1     1       1     1     1     1     1     1     1       1     1     1     1     1     1     1       1     1     1     1     1     1     1       1     1     1     1     1     1     1       1     1     1     1     1     1     1       1     1     1     1     1     1     1	N13/015968	RA10-1	7/05/2013	Rabbit Muscle	×	×	×	×				×	×		
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**National Measurement Institute** 

	CERTIFICATE OF A	NALYSIS # DAU	13_145
Client	Cardno Ecology Lab L9, 203 Pacific Highway, St L	Job No.	CARD20/130614
	NSW 2065	Sampled by Date Sampled	Client 7-May-2013
Contact	Marcus Lincoln-Smith	Date Received	14-Jun-2013
	The resu	ults relate only to the samp	ble(s) tested.

Method	AUTL_07	Date Reported	17-Jul-2013	

# **Details** The method is for determination of Perfluoroalkyl substances (PFASs) in biota samples by High Performance Liquid Chromatography tandem Mass Spectrometry (UPLC-MSMS). All results are corrected for labelled surrogates and are reported on a fresh weight basis.

Prior to extraction the sample is spiked with a range of isotopically labelled surrogate standards. Extraction is by organic solvent, with purification using activated silica gel. An aliquot of extract is injected onto the UPLC and detected using mass spectrometry.

Authorisation

aun

Gavin Stevenson Manager Dioxin Analysis Unit

Dr Alan Yates Senior Analyst Dioxin Analysis Unit

	Sample Deta	ails : Job No. CA	ARD20/130614
Laboratory Reg. No.	Client Sample Ref.	Matrix	Description
N13/015963X	RA3-1	Rabbit muscle	Rabbit Muscle 7-May-13
N13/015964X	RA4-1	Rabbit muscle	Rabbit Muscle 7-May-13
N13/015965X	RA6-1	Rabbit muscle	Rabbit Muscle 7-May-13
N13/015966X	RA7-1	Rabbit muscle	Rabbit Muscle 7-May-13
N13/015967X	RA9-1	Rabbit muscle	Rabbit Muscle 7-May-13
N13/015968X	RA10-1	Rabbit muscle	Rabbit Muscle 7-May-13
Sample Jar L879 N13/015965 DUP L879	Container Blank	Sample Jar	Sample Jar
N13/015965 DUP L879	Duplicate	Rabbit muscle Rabbit muscle	Duplicate Sample Spiked sample (19 ng/g, 15 ng/g for 6:2FTS)
BLK L879	Spike Lab Blank	Lab Blank	Lab Blank
DER EUro	Eab Diam	Lab Diarik	Lub Blank
Project Details			
Project Name Fiskville Study			
Project Number NA49913-034			
Кеу			
Analytes			Surrogate
PFPeA	Perfluoro-n-pentanoic acid	1	40
PFHxA	Perfluoro-n-hexanoic acid		Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]hexanoic acid Surrogate
PFHpA	Perfluoro-n-heptanoic acid	1	
PFOA	Perfluoro-n-octanoic acid		Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]octanoic acid
PFNA	Perfluoro-n-nonanoic acid		Perfluoro-n-[1,2,3,4,5- <sup>13</sup> C <sub>5</sub> ]nonanoic acid
PFDA	Perfluoro-n-decanoic acid		Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]decanoic acid
PFUdA	Perfluoro-n-undecanoic ac	bid	Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]undecanoic acid
PFDoA	Perfluoro-n-dodecanoic ac	cid	Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]dodecanoic acid
PFOS	Perfluoro-n-octanesulfona	te	Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]octanesulfonate
6:2 FTS	1H,1H,2H,2H-perfluoro-n-	octane sulfonate	1H,1H,2H,2H-perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]octane sulfonate
Units & Abbreviations			
ng/g	nanograms per gram		
<	level less than limit of reporting	(LOR)	
þ	surrogate recovery outside norm	nal method range (25-12	25%)

#### Laboratory Reg. No. N13/015963X

Client Sample Ref. RA3-1 Matrix Rabbit muscle Description Rabbit Muscle 7-May-13 Extraction Date 21-Jun-13 Analysis Date 10-Jul-13

	Level ng/g	Labelled Surroga recovery	te
PFPeA PFHxA	<0.5 <0.5	109	
PFHpA PFOA	<0.5 <0.5	109	
PFNA	<0.5	95	
PFDA	<0.5	113	
PFUdA	<1	74	
PFDoA	<0.5	31	
PFOS	110	62	
6:2 FTS	<0.5	127	þ

#### Laboratory Reg. No. N13/015964X

Client Sample Ref. RA4-1 Matrix Rabbit muscle Description Rabbit Muscle 7-May-13 Extraction Date 21-Jun-13 Analysis Date 10-Jul-13

	Level ng/g	Labelled Sur recove	
PFPeA PFHxA	0.99 <0.5	116	
PFHpA PFOA	<0.5 <0.5	122	
PFNA	<0.5	87	
PFDA	<0.5	130	þ
PFUdA	<1	79	
PFDoA	<0.5	33	
PFOS	150	60	
6:2 FTS	<0.5	133	þ

#### Laboratory Reg. No. N13/015965X

Client Sample Ref. RA6-1 Matrix Rabbit muscle Description Rabbit Muscle 7-May-13 Extraction Date 21-Jun-13 Analysis Date 10-Jul-13

	Level ng/g	Labelled Sur recover	
PFPeA PFHxA	0.83 <0.5	123	
PFHpA PFOA	<0.5 <0.5	122	
PFNA	<0.5	72	
PFDA	<0.5	98	
PFUdA	<1	59	
PFDoA	<0.5	17	þ
PFOS	220	45	
6:2 FTS	<0.5	125	

#### Laboratory Reg. No. N13/015966X

Client Sample Ref. RA7-1 Matrix Rabbit muscle Description Rabbit Muscle 7-May-13 Extraction Date 21-Jun-13 Analysis Date 10-Jul-13

	Level ng/g	Labelled Sur recover	<u> </u>
PFPeA PFHxA	3.7 0.71	119	
PFHpA PFOA	<0.5 <0.5	106	
PFNA	<0.5	40	
PFDA	<0.6	67	
PFUdA	<1	41	
PFDoA	<0.5	12	þ
PFOS	600	26	
6:2 FTS	<0.5	93	

#### Laboratory Reg. No. N13/015967X

Client Sample Ref. RA9-1 Matrix Rabbit muscle Description Rabbit Muscle 7-May-13 Extraction Date 21-Jun-13 Analysis Date 10-Jul-13

	Level ng/g	Labelled Surrogate recovery
PFPeA PFHxA	<0.5 <0.5	115
PFHpA PFOA	<0.5 <0.5	117
PFNA	<0.5	85
PFDA	<0.5	108
PFUdA	<1	69
PFDoA	<0.5	27
PFOS	150	51
6:2 FTS	<0.5	94

#### Laboratory Reg. No. N13/015968X

Client Sample Ref. RA10-1 Matrix Rabbit muscle Description Rabbit Muscle 7-May-13 Extraction Date 21-Jun-13 Analysis Date 10-Jul-13

	Level ng/g	Labelled Surrogate recovery				
PFPeA PFHxA	1.3 <0.5	122				
PFHpA PFOA	<0.5 <0.5	119				
PFNA	<0.5	92				
PFDA	<0.5	113				
PFUdA	<1	60				
PFDoA	<0.5	20	þ			
PFOS	110	58				
6:2 FTS	<0.5	112				

Laboratory Reg. No. Sample Jar L879

Client Sample Ref. Container Blank Matrix Sample Jar Description Sample Jar Extraction Date 21-Jun-13 Analysis Date 10-Jul-13

	Level	Labelled Surrogate				
	ng/g	recovery				
PFPeA	<0.5					
PFHxA	<0.5	96				
PFHpA	<0.5					
PFOA	<0.5	79				
PFNA	<0.5	82				
PFDA	<0.5	41				
PFUdA	<1	16	þ			
PFDoA	<1	4	þ			
PFOS	<1	35				
6:2 FTS	<0.5	90				

#### Laboratory Reg. No. N13/015965 DUP L879

Client Sample Ref. Duplicate Matrix Rabbit muscle Description Duplicate Sample Extraction Date 21-Jun-13 Analysis Date 10-Jul-13

	Level ng/g	Labelled Surrogate recovery					
PFPeA PFHxA	0.46 <0.5	135	þ				
PFHpA PFOA	<0.5 <0.5	125					
PFNA	<0.5	91					
PFDA	<0.5	125					
PFUdA	<1	83					
PFDoA	<0.5	28					
PFOS	220	49					
6:2 FTS	<0.5	113					

#### Laboratory Reg. No. N13/015965 SPK L879

Client Sample Ref. Spike Matrix Rabbit muscle Description Spiked sample (19 ng/g, 15 ng/g for 6:2FTS) Extraction Date 21-Jun-13 Analysis Date 10-Jul-13

	Level ng/g	Labelled Surrogate recovery				
PFPeA PFHxA	16 15	116				
PFHpA PFOA	17 15	115				
PFNA	16	75				
PFDA	16	110				
PFUdA	15	75				
PFDoA	10	34				
PFOS	240	49				
6:2 FTS	18	119				

#### Laboratory Reg. No. BLK L879

Client Sample Ref. Lab Blank Matrix Lab Blank Description Lab Blank Extraction Date 21-Jun-13 Analysis Date 10-Jul-13

	Level ng/g	Labelled Sur recove				
PFPeA PFHxA	<0.1 <0.07	99				
PFHpA PFOA	<0.02 <0.1	75				
PFNA	<0.04	49				
PFDA	<0.4	15	þ			
PFUdA	<3	6	þ			
PFDoA	<4	1	þ			
PFOS	<0.5	20	þ			
6:2 FTS	<0.1	59				

Issue Date: July 2010	Received By:	Comments:			×	-	RA9-2	RA6-2	RA4-2	Customer ID	E-maii:	Report Results To: (if different from above)	AQ Ref: (AsureQuality use only)	Page of (If more than one submission form used)	AND 70 8800 70 8176	1C Quadrant Drive Waiwhetu Lower Hutt	AsureQuality Wellington Laboratory	TO:	
	Receipt Date/Time:						Rabbit Muscle	Rabbit Muscle	Rabbit Muscle	Sample Description	- Fax:		eonly 134925	n form used)	ATTENTION:		gton Laboratory		SAMI
Page 1 of 1 QA Controlled Document	Courier Number:	a state of the second stat									Address:		Tel: 02 9496 7888 0413622086	Quote Number: 2751	NSW, 2065 Marcus LincolnSmith	Cardno Ecology Lab L9, 203 Pacific Highway, St	Customer Name/Address:	FROM:	SAMPLE SUBMISSION FORM
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1C uadrant Drive, Waiwhetu P.O. Box 31 242, Lower Hutt 5010 Wellington, New Zealand T 64 4 5708800
 F 64 4 5708176
 W www.asurequality.com

## **Certificate of Analysis**

Date Issued:	3 July 2013
Client:	Cardno LanePiper Building 2 154 Highbury Road Burwood Victoria 3125
Attention:	Marcus Lincoln Smith
AsureQuality Lab. Reference:	134925
Sample Type(s):	Rabbit Muscle
Analysis:	Perfluorinated Compounds (PFCs)
Method:	In-House LC-MS/MS Method

Results are reported as nanograms per gram (ng/g), on an as received basis to two significant figures. The LOR value is reported to two significant figures. Results have been corrected for recovery.

Unless requested, samples will be disposed of eight weeks from the date of this report.

Comments: None.

Phil Bridgen Senior Scientist AsureQuality Limited

Laboratory Reference: 134925-1

Sample Identification: RA4-2 Rabbit Muscle

Date Received: 14 Jun 2013 Date Extracted: 17 Jun 2013		Date Analyse	ed: 17 Jun 2013
Analyte <sup>1</sup>	Conc. <sup>2</sup> (ng/g)	LOR (ng/g)	Data Qualifiers
Perfluoroalkylsulfonic acids			
Perfluorobutanesulfonic acid (PFBS)	ND	1.0	
Perfluorohexanesulfonic acid (PFHxS)	68	4.0	
Perfluorooctanesulfonic acid (PFOS) <sup>3</sup>	140	8.0	
Perfluorodecanesulfonic acid (PFDS)	ND	1.0	
Perfluoroalkylcarboxylic acids			
Perfluorohexanoic acid (PFHxA)	ND	1.0	
Perfluoroheptanoic acid (PFHpA)	ND	1.0	
Perfluorooctanoic acid (PFOA)	ND	2.0	
Perfluorononanoic acid (PFNA)	ND	1.0	
Perfluorodecanoic acid (PFDA)	ND	2.0	
Perfluoroundecanoic acid (PFUnA)	ND	1.0	
Perfluorododecanoic acid (PFDoA)	ND	2.0	
Perfluorotridecanoic acid (PFTrDA)	ND	1.0	
Perfluorotetradecanoic acid (PFTeDA)	ND	1.0	
Other PFCs			
Perfluorooctanesulfonamide (PFOSA)	ND	1.0	
N-ethyl-perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	ND	1.0	
N-methyl-perfluorooctanesulfonamidoacetic acid (NMeFOSAA)	ND	1.0	
1H,1H,2H,2H-perfluorooctanesulfonic acid (6:2 FTS)	ND	1.0	
1H,1H,2H,2H-perfluorodecanesulfonic acid (8:2 FTS)	ND	2.0	
Footnotes:	Abbreviations:		
<sup>1</sup> The analytes listed represent the linear isomer.	LOR: Limit of Reportin	g	
<sup>2</sup> Results are reported on an as received basis.	ND: Not Detected		
<sup>3</sup> The result for PFOS also includes its salts and			
perfluorooctanesulfonyl fluoride (PFOSF).			

Lab Analyst: CFH/CA

Data Analyst: CFH/PB

Laboratory Reference: 134925-2

Sample Identification: RA6-2 Rabbit Muscle

Date Received: 14 Jun 2013 Date Extracted: 17 Jun 2013		Date Analyse	ed: 17 Jun 2013
Analyte <sup>1</sup>	Conc. <sup>2</sup> (ng/g)	LOR (ng/g)	Data Qualifiers
Perfluoroalkylsulfonic acids			
Perfluorobutanesulfonic acid (PFBS)	ND	1.0	
Perfluorohexanesulfonic acid (PFHxS)	32	4.0	
Perfluorooctanesulfonic acid (PFOS) <sup>3</sup>	190	8.0	
Perfluorodecanesulfonic acid (PFDS)	1.6	1.0	
Perfluoroalkylcarboxylic acids			
Perfluorohexanoic acid (PFHxA)	ND	1.0	
Perfluoroheptanoic acid (PFHpA)	ND	1.0	
Perfluorooctanoic acid (PFOA)	ND	2.0	
Perfluorononanoic acid (PFNA)	ND	1.0	
Perfluorodecanoic acid (PFDA)	ND	2.0	
Perfluoroundecanoic acid (PFUnA)	ND	1.0	
Perfluorododecanoic acid (PFDoA)	ND	2.0	
Perfluorotridecanoic acid (PFTrDA)	ND	1.0	
Perfluorotetradecanoic acid (PFTeDA)	ND	1.0	
Other PFCs			
Perfluorooctanesulfonamide (PFOSA)	ND	1.0	
N-ethyl-perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	ND	1.0	
N-methyl-perfluorooctanesulfonamidoacetic acid (NMeFOSAA)	ND	1.0	
1H,1H,2H,2H-perfluorooctanesulfonic acid (6:2 FTS)	ND	1.0	
1H,1H,2H,2H-perfluorodecanesulfonic acid (8:2 FTS)	8.6	2.0	
Footnotes:	Abbreviations:		
<sup>1</sup> The analytes listed represent the linear isomer.	LOR: Limit of Reportin	g	
<sup>2</sup> Results are reported on an as received basis.	ND: Not Detected		
<sup>3</sup> The result for PFOS also includes its salts and			
perfluorooctanesulfonyl fluoride (PFOSF).			

Lab Analyst: CFH/CA

Data Analyst: CFH/PB

Laboratory Reference: 134925-3

Sample Identification: RA9-2 - Rabbit Muscle

Date Received: 14 Jun 2013 Date Extracted: 17 Jun 2013		Date Analyse	ed: 17 Jun 2013
Analyte <sup>1</sup>	Conc. <sup>2</sup> (ng/g)	LOR (ng/g)	Data Qualifiers
Perfluoroalkylsulfonic acids			
Perfluorobutanesulfonic acid (PFBS)	ND	1.0	
Perfluorohexanesulfonic acid (PFHxS)	40	4.0	
Perfluorooctanesulfonic acid (PFOS) <sup>3</sup>	140	8.0	
Perfluorodecanesulfonic acid (PFDS)	2.7	1.0	
Perfluoroalkylcarboxylic acids			
Perfluorohexanoic acid (PFHxA)	ND	1.0	
Perfluoroheptanoic acid (PFHpA)	ND	1.0	
Perfluorooctanoic acid (PFOA)	ND	2.0	
Perfluorononanoic acid (PFNA)	ND	1.0	
Perfluorodecanoic acid (PFDA)	ND	2.0	
Perfluoroundecanoic acid (PFUnA)	ND	1.0	
Perfluorododecanoic acid (PFDoA)	ND	2.0	
Perfluorotridecanoic acid (PFTrDA)	ND	1.0	
Perfluorotetradecanoic acid (PFTeDA)	ND	1.0	
Other PFCs			
Perfluorooctanesulfonamide (PFOSA)	ND	1.0	
N-ethyl-perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	ND	1.0	
N-methyl-perfluorooctanesulfonamidoacetic acid (NMeFOSAA)	ND	1.0	
1H,1H,2H,2H-perfluorooctanesulfonic acid (6:2 FTS)	ND	1.0	
1H,1H,2H,2H-perfluorodecanesulfonic acid (8:2 FTS)	ND	2.0	
Footnotes:	Abbreviations:		
<sup>1</sup> The analytes listed represent the linear isomer.	LOR: Limit of Reportin	g	
<ul> <li><sup>2</sup> Results are reported on an as received basis.</li> <li><sup>3</sup> The result for PFOS also includes its salts and</li> </ul>	ND: Not Detected		
perfluorooctanesulfonyl fluoride (PFOSF).			

Lab Analyst: CFH/CA

Data Analyst: CFH/PB

Laboratory Reference: 134925-BL

Sample Identification: Laboratory Blank

Date Received: Not Applicable Date Extracted: 17 Jun 2013		Date Analyse	ed: 17 Jun 2013
Analyte <sup>1</sup>	$\operatorname{Conc.}^2(\operatorname{ng/g})$	LOR (ng/g)	Data Qualifiers
Perfluoroalkylsulfonic acids			
Perfluorobutanesulfonic acid (PFBS)	ND	1.0	
Perfluorohexanesulfonic acid (PFHxS)	ND	4.0	
Perfluorooctanesulfonic acid (PFOS) <sup>3</sup>	ND	8.0	
Perfluorodecanesulfonic acid (PFDS)	ND	1.0	
Perfluoroalkylcarboxylic acids			
Perfluorohexanoic acid (PFHxA)	ND	1.0	
Perfluoroheptanoic acid (PFHpA)	ND	1.0	
Perfluorooctanoic acid (PFOA)	ND	2.0	
Perfluorononanoic acid (PFNA)	ND	1.0	
Perfluorodecanoic acid (PFDA)	ND	2.0	
Perfluoroundecanoic acid (PFUnA)	ND	1.0	
Perfluorododecanoic acid (PFDoA)	ND	2.0	
Perfluorotridecanoic acid (PFTrDA)	ND	1.0	
Perfluorotetradecanoic acid (PFTeDA)	ND	1.0	
Other PFCs			
Perfluorooctanesulfonamide (PFOSA)	ND	1.0	
N-ethyl-perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	ND	1.0	
N-methyl-perfluorooctanesulfonamidoacetic acid (NMeFOSAA)	ND	1.0	
1H,1H,2H,2H-perfluorooctanesulfonic acid (6:2 FTS)	ND	1.0	
1H,1H,2H,2H-perfluorodecanesulfonic acid (8:2 FTS)	ND	2.0	
Footnotes:	Abbreviations:		
<sup>1</sup> The analytes listed represent the linear isomer	LOR: Limit of Reportin	g	
<sup>2</sup> The results are calculated using the average weight	ND: Not Detected		
of samples in this batch			
<sup>3</sup> The result for PFOS also includes its salts and			
perfluorooctanesulfonyl fluoride (PFOSF).			

Lab Analyst: CFH/CA

Data Analyst: CFH/PB

/ Data
uscle Laborator
for Rabbit M
/ QC Review
Table B1 - QA

										[	ng/g wet weight	weight					
Laboratory Reference Number	CARDNO Ref.	Sample Location	Sample Type	Sample Matrix	Certificate	PFPeA	PFHxS	PFHxA	PFHpA PI	PFOA PF	PENA PE	PFDA PFI	PFDS PFUdA	A PFDoA	A PFOS	6:2 FTS	8:2 FtS
N13/012591X	RA1-1	Fiskville	Rabbit	Muscle	CARD20/130513	<0.5		<0.5	<0.5 <	<0.5 <	<0.5 <0	<0.5	4	<0.5	44	<0.5	
N13/012592X	RA2-1	Fiskville	Rabbit	Muscle	CARD20/130513	<0.5		<0.5	<0.5 <	<0.5 <	<0.5 <0	<0.5	4	<0.5	130	<0.5	
N13/012593X	RA5-1	Fiskville	Rabbit	Muscle	CARD20/130513	2.4		0.95	<0.5 <	<0.5 <	<0.5 0.	0.89	~1	0.6	350	<0.5	
N13/012594X	RA8-1	Fiskville	Rabbit	Muscle	CARD20/130513	2.2		0.58	<0.5 <	<0.5 <	<0.5 0.	0.93	<1	<0.5	380	<0.5	
N13/012594DUP	RA8-1	Fiskville	Rabbit	Muscle	CARD20/130513	3.2		0.88	<0.5 <	<0.5 <	<0.5 1	1.2	4	<0.5	510	<0.5	
N13/012594SPK 17 ng/g, 14 ng/g for 6:2 FtS	RA8-1	Fiskville	Rabbit	Muscle	CARD20/130513	24		25	23	24 2	24 2	25	26	26	530	16	
BLK L869					CARD20/130513	<0.5		<0.5	<0.5 <	<0.5 <	<0.5 <0	<0.5	<1	<0.5	<0.5	<0.5	
N13/015963X	RA3-1	Fiskville	Rabbit	Muscle	CARD20/130614	<0.5		<0.5	<0.5 <	<0.5 <	<0.5 <0	<0.5	4	<0.5	110	<0.5	
N13/015964X	RA4-1	Fiskville	Rabbit	Muscle	CARD20/130614	66.0		<0.5	<0.5 <	<0.5 <	<0.5 <0	<0.5	7	<0.5	150	<0.5	
134925-1 (Asure Quality)	RA4-2	Fiskville	Rabbit	Muscle	134925		68	<1.0	<1.0 <	<2.0 <	<1.0 <2	<2.0 <1	<1.0	<2.0	140	<1.0	<2.0
N13/015965X	RA6-1	Fiskville	Rabbit	Muscle	CARD20/130614	0.83		<0.5	<0.5 <	<0.5 <	<0.5 <0	<0.5	4	<0.5	220	<0.5	
134925-2 (Asure Quality)	RA6-2	Fiskville	Rabbit	Muscle	134925		32	<1.0	<1.0 <	<2.0 <	<1.0 <2	<2.0 1.	1.6	<2.0	190	<1.0	8.6
N13/015966X	RA7-1	Fiskville	Rabbit	Muscle	CARD20/130614	3.7		0.71	<0.5 <	<0.5 <	<0.5 <0	<0.6	7	<0.5	600	<0.5	
N13/015967X	RA9-1	Fiskville	Rabbit	Muscle	CARD20/130614	<0.5		<0.5	<0.5 <	<0.5 <	<0.5 <0	<0.5	4	<0.5	150	<0.5	
134925-3 (Asure Quality)	RA9-2	Fiskville			134925		40	<1.0	<1.0 <	<2.0 <	<1.0 <2	<2.0 2.7	7	<2.0	140	<1.0	<2.0
N13/015968X	RA10-1	Fiskville	Rabbit	Muscle	CARD20/130614	1.3		<0.5	<0.5 <	<0.5 <	<0.5 <0	<0.5	<1	<0.5	110	<0.5	
N13/015965 DUP L879	RA6-1	Fiskville	Rabbit	Muscle	CARD20/130614	0.46		<0.5	<0.5 <	<0.5 <	<0.5 <0	<0.5	<1	<0.5	220	<0.5	
N13/015965 SPK L879 19 ng/g, 15 ng/g for 6:2 FtS	RA6-1	Fiskville	Rabbit	Muscle	CARD20/130614	16		15	17	15	16 1	16	15	10	240	18	
BLK L879					CARD20/130614	<0.1		<0.07	<0.02 <	<0.1 <0	<0.04 <c< td=""><td>&lt;0.4</td><td>&lt;3</td><td>&lt;4</td><td>&lt;0.5</td><td>&lt;0.1</td><td></td></c<>	<0.4	<3	<4	<0.5	<0.1	
134925-BL					134925		<1.0	<0.1	<0.1 <	<2.0 <	<1.0 <2	<2.0 <1	<1.0	<2.0	<8.0	<1.0	<2.0
Sample Jar L8799 (Rinsate)			Rinsate	Water	CARD20/130614	<0.5		<0.5	<0.5 <	<0.5 <	<0.5 <0	<0.5	<1	<1	~1	<0.5	
Number of Samples (total)	15																
Total Number of Analysis (Inc. Blank, Spikes and Duplicate)	d Duplicate)					16	4	20	20	20	20 2	20 4	16	20	20	20	4
Number of Blanks - NMI and AQ (Analysis)	с																
Number of Duplicates - NMI (Analysis)	2					PFPeA	PFHxS	PFHXA	PFHpA PI	PFOA PF	PFNA PF	PFDA PFI	PFDS PFUdA	IA PFDoA	A PFOS	6:2 FTS	8:2 FtS
Number of Interlaboratory (Analysis)			Analysis le	se than 1 00	Analysis less than I OO (Not inc. blanks)	4	c	11		ц Г	15 1	1 2	11	14	c	ר נו	'n

Number of Samples (rocal)         15         1         20         2																
16       Analysis less than LOQ (Not inc. blanks)       Analysis less than LOQ (Not inc. blanks)       4       Number of Detects (Not inc. spikes)       7       Sample Percentage detects (Total)       (Not including blank, spikes)	Number of Samples (total)	15														
alysis)         3         FFPeA           a)         2         Priped           a)         2         Analysis less than LOQ (Not inc. blanks)         4           a)         2         Number of Detects (Not inc. spikes)         7           QA/QC Ratio         >1:5         (Not including blank, spikes)         47%	Total Number of Analysis (Inc. Blank, Spikes and D	uplicate)		16	4	20	20	20		20	4	16	20	20	20	4
i)         2         PFPeA           3         3         Analysis less than LOQ (Not inc. blanks)         4           2         Number of Detects (Not inc. spikes)         7           QA/QC Ratio         > 1:5         (Not including blank, spikes)         47%	Number of Blanks - NMI and AQ (Analysis)	ო														
3         Analysis less than LOQ (Not inc. blanks)         4         0         11         15         15         12         2         11         14         0         15         15         12         2         11         14         0         15         15           2         Number of Detects (Not inc. splikes)         7         3         4         0         0         3         2         0         1         15         0           Sample Percentage detects (Total)         73         27%         0%         0%         50%         5%         7%         0% <t< th=""><th>Number of Duplicates - NMI (Analysis)</th><th>2</th><th></th><th></th><th>PFHxS</th><th>PFHxA</th><th>PFHpA</th><th>PFOA</th><th>PFNA</th><th>PFDA</th><th>FDS F</th><th>FUdA F</th><th>PFDoA</th><th>PFOS</th><th>6:2 FTS</th><th>8:2 FtS</th></t<>	Number of Duplicates - NMI (Analysis)	2			PFHxS	PFHxA	PFHpA	PFOA	PFNA	PFDA	FDS F	FUdA F	PFDoA	PFOS	6:2 FTS	8:2 FtS
2         Number of Detects (Not inc. spikes)         7         3         4         0         0         3         2         0         1         15         0           QA/QC Ratio         >1:5         >1:5         0%	Number of Interlaboratory (Analysis)	ĸ	Analysis less than LOQ (Not inc. blanks)	4	0	11	15			12	2	11	14	0	15	m
> 1:5 Sample Percentage detects (Total) (Not including blank, spikes) 47% 75% 27% 0% 0% 0% 20% 50% 0% 7% 100% 0%	Number of Spikes (Analysis)	2	Number of Detects (Not inc. spikes)		3	4	0	0	0	e	2	0	1	15	0	1
	QA/QC Ratio	> 1:5	Sample Percentage detects (Total) (Not including blank, spikes)		75%	27%	%0	%0	%0	20%	50%	%0	7%	100%	%0	25%



	S																				
	6:2 FTS	80	68	94	54	69	72	69	127	133	75	125	70	93	94	68	112	113	119	59	6
	PFOS	51	47	32	27	30	28	42	62	60	104	45	78	26	51	103	58	49	49	20	35
%	PFDoA	62	79	45	71	64	56	51	31	33	92	17	84	12	27	104	20	28	34	1	4
Surrogate recovery %	PFUdA	64	82	60	71	73	60	58	74	79		59		41	69		60	83	75	6	16
urrogate	PFDA	54	74	42	53	68	52	57	113	130	134	98	116	67	108	112	113	125	110	15	41
2 S	PFNA	68	63	39	34	42	35	65	95	87	118	72	113	40	85	113	92	91	75	49	82
	PFOA	63	79	66	68	74	64	69	109	122	115	122	116	106	117	110	119	125	115	75	79
	PFHxA	49	64	51	61	60	57	51	109	116	115	123	113	119	115	109	122	135	116	66	96
	PEF	7	Ŭ																		
		7				112%	112%			<lor< td=""><td></td><td><lor< td=""><td><lor< td=""><td></td><td><lor< td=""><td></td><td></td><td></td><td></td><td></td><td></td></lor<></td></lor<></td></lor<></td></lor<>		<lor< td=""><td><lor< td=""><td></td><td><lor< td=""><td></td><td></td><td></td><td></td><td></td><td></td></lor<></td></lor<></td></lor<>	<lor< td=""><td></td><td><lor< td=""><td></td><td></td><td></td><td></td><td></td><td></td></lor<></td></lor<>		<lor< td=""><td></td><td></td><td></td><td></td><td></td><td></td></lor<>						
	6:2 FTS	7				134% 112%	112% 112%			6.9 <lor< td=""><td></td><td>14.6 <lor< td=""><td>0.0 <lor< td=""><td></td><td>6.9 <lor< td=""><td></td><td></td><td>118%</td><td>118%</td><td></td><td></td></lor<></td></lor<></td></lor<></td></lor<>		14.6 <lor< td=""><td>0.0 <lor< td=""><td></td><td>6.9 <lor< td=""><td></td><td></td><td>118%</td><td>118%</td><td></td><td></td></lor<></td></lor<></td></lor<>	0.0 <lor< td=""><td></td><td>6.9 <lor< td=""><td></td><td></td><td>118%</td><td>118%</td><td></td><td></td></lor<></td></lor<>		6.9 <lor< td=""><td></td><td></td><td>118%</td><td>118%</td><td></td><td></td></lor<>			118%	118%		
		7			<lor -29.2="" -<="" td=""><td>151% 134% 112%</td><td>151% 101% 112%</td><td></td><td></td><td>-</td><td></td><td><lor 14.6="" <lor<="" td=""><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></lor></td></lor>	151% 134% 112%	151% 101% 112%			-		<lor 14.6="" <lor<="" td=""><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></lor>									
	PFOS 6:2 FTS	7				134%	101%			6.9		14.6	0.0		6.9			100% 118%	100% 118%		

PFDA PFDS

PFPEA PFHxS PFHxA PFHpA PFOA PFNA

Blanks Sum

CARDNO Ref.

Laboratory Reference Number

N13/012591X N13/012592X -25.4 139% 137%

<LOR 139%

<LOR 139%

<LOR 133%

-41.1 142% 140%

-37.0 125% 119%

% RPD Primary Duplicate

RA8-1 & 1

RA1-1 RA2-1 RA5-1 RA8-1 RA8-1 RA8-1

> N13/012593X N13/012594X N13/012594DUP N13/0125945PK 17 ng/g, 14 ng/g for 6:2 FtS

<LOR

<LOR <LOR

<LOR

<LOR

% RPD

N13/015964X 134925-1 (Asure Quality) N13/015965X

V13/015963X

3LK L869

134925-2 (Asure Quality) N13/015966X .34925-3 (Asure Quality)

113/015967X

<LOR

<LOR <LOR <LOR <LOR <LOR <LOR</pre>

<LOR <LOR

57.4

% RPD % RPD <LOR

<LOR

<LOR

<LOR

<LOR

% RPD

RA3-1 RA4-1 RA4-1 RA4-2 RA4-2 RA6-1 RA6-1 RA6-1 RA9-1 RA9-1 RA9-1 RA9-1 RA6-1 RA6-1

 78%
 83%
 83%

 78%
 83%
 83%

88% 88%

78% 78%

81% 82%

Primary Duplicate

N13/015968X N13/015965 DUP L879 N13/015965 SPK L879 19 ng/g, 15 ng/g for 6:2 FtS BLK L879

l34925-BL

<LOR <LOR

%RPD and Spike Recovery

Sample Jar L8799 (Rinsate)		<lor< th=""><th>-</th></lor<>	-
Number of Samples (total)	15		
Total Number of Analysis (Inc. Blank, Spikes and Duplicate	Juplicate)		
Number of Blanks - NMI and AQ (Analysis)	с		
Number of Duplicates - NMI (Analysis)	2		
Number of Interlaboratory (Analysis)	3		
Number of Spikes (Analysis)	2		
QA/QC Ratio	> 1:5		

	PFHXA	PFOA	PFNA	PFDA	PFUdA	PFDoA	PFOS	6:2 FTS
Minimum	49	63	34	15	9	1	20	54
Maximum	135	125	118	134	83	104	104	133

Surrogate Recovery Summary

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C Cardno

## Appendix G 69 Pages

Health Impact Assessment from Consumption of Fish from Lake Fiskville. (ToxConsult 2013).





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# Health impact assessment from consumption of fish from Lake Fiskville

Prepared by: Roger Drew, PhD, DABT Tarah Hagen, MSc, ToxConsult Pty Ltd.

Prepared for: Rob Jamieson Ashurst

> ToxConsult document ToxCR061113-RF2 1<sup>st</sup> April 2014

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Tarah Hage

**Tarah Hagen, MSc** (Environmental Toxicology)



## **Document history**

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ToxCR061113-RF1	R Jamieson, Ashurst	R.Drew	Secure PDF



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## About ToxConsult Pty Ltd

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Dr Roger Drew is one of the principal consultants of ToxConsult Pty Ltd. He has primary degrees in biochemistry and pharmacology and postgraduate degrees in toxicology. Postdoctoral training was undertaken at the National Institutes of Health, National Cancer Institute in the USA. He has more than 30 years of toxicological and risk assessment experience in academia, industry and consulting. He has provided advice to a range of industries and Government authorities and has significantly participated in developing risk assessment practice in Australia. Dr Drew is one of just a few toxicologists in Australia certified by the American Board of Toxicology.

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#### Ms Hagen

Tarah Hagen is a director and senior consultant at ToxConsult Pty Ltd providing ecotoxicology and risk assessment services to a broad range of industries and government bodies. She has a Masters degree in Environmental Toxicology and Pollution Monitoring, an honours degree in ecotoxicology, and a degree in Applied Science (Biological Sciences).



## **Executive Summary**

As a result of past training practices at Fiskville, the water and sediment of Lake Fiskville has high concentrations of perfluorochemicals (PFCs). From their extensive use in consumer products these chemicals are also ubiquitous in the general and human environment. The biota in Lake Fiskville has assimilated PFCs present in lake water and/or sediment to a much larger extent than expected from background exposure.

In particular redfin fish from the lake have very high concentrations of perfluorooctane sulphonate (PFOS) in their flesh. This is also the PFC which is at the highest concentration in water and sediment and is the PFC of concern within the lake and biota. Concentrations of PFOS in redfin were higher than those in fish considered by overseas agencies as being unfit for consumption. As soon as it became apparent to CFA management that employees were catching and consuming fish or eels from the Lake staff were advised verbally and by newsletter not to fish the lake, and prominent signs were erected at the lake to that effect. Further notices were placed in local newspapers to advise the local community.

Significant uncertainties regarding the extent and frequency that fish or eel were consumed, and lack of PFOS data in eels, precluded assessing health risk from eating fish using a traditional tolerable daily intake (TDI) approach. Because the toxicological effects of PFOS are directly related to serum concentrations, and the sensitive effects in monkeys are changes in blood biomarkers that are routinely evaluated by medical doctors for health status, persons who had eaten fish in the past were invited to voluntarily participate in a health surveillance program. This was also open to persons who may not have eaten fish but were nonetheless concerned they may have been exposed to PFCs while working at Fiskville. This 'fish consumption' health surveillance program was an extension of the health status surveillance package already in place for CFA PAD workers. Additional to the existing medical surveillance of medication examination and measurement of routine blood parameters was quantitation of heavy metals in blood and PFC concentrations in serum. Participants were asked if their de-identified results could be made available, via the CFA medical officer, to the consulting toxicologist and thence to the CFA in the form of this report. Participation in the 'fish consumption' health surveillance program was not contingent upon agreement to share de-identified information, however all participants agreed their data could be made available.

Serum PFC measurements were undertaken by a commercial laboratory that included appropriate blanks, PFC spikes and duplicate analysis of samples chosen randomly. While internal standard recoveries for some samples were lower than the range regarded as ideal by the laboratory, the data are considered reliable for assessment of potential health risk.



To preserve anonymity, PFC serum concentrations are discussed in a general sense in this report.

Twelve of the 22 participants in the 'fish consumption' health surveillance program indicated that they had eaten fish or eel from the Lake in the past. For no person in the surveillance program were there changes in blood clinical chemistry parameters that could be attributed to PFOS. While recognising the very small sample size limits confidence in the data interpretation, regression analysis of *a priori* individual blood parameters with serum PFOS levels for either the entire cohort or just those that ate fish indicated no associations. Nevertheless there were a number of individuals in both the fish eating and non-fish eating groups that had blood parameter measurements outside the population reference range. The medical officer attributed all these to life style factors (e.g. alcohol consumption), body mass index, existing disease, and/or medication (including non-compliance). Where appropriate the medical officer referred people to their own medical practitioner.

Of the 10 PFCs looked for in human serum (chosen for their presence in Lake water or fish) only two were present at measurable concentrations in the serum of program participants. These were PFOS and perfluorooctanoic acid (PFOA). All PFOA measurements were approximately an order of magnitude less than the expected background concentrations for this compound. This indicates fish consumption has not contributed to human PFOA serum concentrations; not unexpected since redfin did not have measurable concentrations of PFOA in their flesh. PFOA was therefore not considered further in the risk assessment.

Many animal studies have shown toxicological effects of PFOS are directly related to serum concentrations. The potential health impact of serum PFOS concentrations measured in participants of the health surveillance program has been assessed in a number of ways.

- Comparison with 'background' serum concentrations.
  - A review of many publications reporting PFOS serum concentration in general communities showed the majority of adults would be expected to have a concentration <0.1 mg/L.</li>
- Comparison with a human serum level considered to be without effects in humans. Three different methods were used to establish a serum no observed effect level (serum NOEL) of 2 mg/L. These were:
  - o Dose response analysis of a number of occupational epidemiology studies,
  - $\circ$   $\;$  Derivation from monkey and rat serum NOELs using standard uncertainty factors, and
  - Conversion of the TDI set by the European Food Safety Authority into an equivalent steady state serum concentration.



 Calculation of margin of exposure (MOE) is a standard risk characterisation method widely used by Australian authorities. However instead of using experimental doses applied to animals and an uncertain estimated human intake in the calculation, the animal serum NOEL from toxicological studies and serum concentrations measured in program participants were used. While an acceptable MOE based on external dose is 100, that based on serum concentrations is 25. MOEs for four different endpoints (low birth weight, blood biomarkers, liver toxicity, and hepatic adenomas) were estimated.

Four persons had serum PFOS concentrations above that identified as the higher end of the normal range expected from background (i.e. resulting from day to day living). All were below the serum NOEL, indicating low risk for adverse health effects. Available information on fishing frequency by some participants in the program suggests serum PFOS concentrations in persons who may not have been included in the cohort were unlikely to be materially different from those measured in the surveillance program.

The Margin of Exposure (MOE) estimations calculated using current measured serum PFOS concentrations and serum NOELs identified in animal toxicity experiments also indicated very low risk for adverse health effects.

When current serum concentrations were extrapolated back to theoretical levels that may have existed 5 or 10 years previously, and assuming no further fish consumption, both comparison with the human serum NOEL and the calculated MOEs indicate adverse health effects were unlikely to have arisen due to these hypothetical serum PFOS concentrations.

Overall, it is concluded existing serum PFOS concentrations, or past theoretical concentrations, are unlikely to give rise to adverse health effects.



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## 1. Introduction

The 'Joy' report (IFI 2012) made a number of recommendations concerning examining potential environmental contamination that may have arisen as a result of historical fire fighting training at the CFA Fiskville training ground. During these investigations it was discovered the sediment and water of Lake Fiskville had become contaminated with perfluorinated chemicals (PFCs). Consultation with a few long term CFA Fiskville employees revealed the Lake had in the past been stocked with Redfin Perch and some employees, over a number of years, had occasionally caught and eaten fish from the Lake.

An initial analysis of a few fish for PFCs showed they, and other organisms in the Lake, had accumulated some of the PFCs found in the water and sediment. In particular perfluorooctane sulphonate (PFOS) was present in very high concentrations in muscle and liver of Redfin. Staff were instructed not to fish the Lake and 'no fishing' signs were erected.

The initial analysis of Redfin was on just four fish, which were the largest of those caught in the sampling program undertaken. Based on recollections of a long term CFA employee for fishing frequency, the numbers of fish caught and the concentration of PFOS in muscle of these four fish, a preliminary informal risk assessment was undertaken to determine potential impact to persons who may have eaten fish from the Lake. The assessment utilised human toxicokinetic information from the scientific literature to predict potential PFOS serum concentrations. It canvassed a range of fish consumption patterns constructed around the anecdotal fishing information provided by the long term employee. The modelling of some of the assumed high consumption patterns suggested high PFOS serum concentrations may occur. At this time the Victorian Department of Health were advised of the situation and of the follow up work that was planned to address significant uncertainties in the modelling of the preliminary assessment.

Major uncertainties in the initial assessment were PFOS concentration data being limited to analysis of just four fish, and no real knowledge of how much fish a person ate or when. The former was addressed by analysis of additional Redfin flesh (in total 21) and the latter by extending the existing CFA personnel health surveillance program to include persons who may have eaten fish. Analysis of blood serum PFCs was added to the existing program for these persons.

This brief report is an updated health risk assessment (HRA) for persons who have eaten fish from Lake Fiskville. However, unlike the preliminary risk assessment it does not rely on toxicokinetic modelling of potential PFOS serum concentrations. The modelling is now redundant. The



assumptions and uncertainties inherent in such modelling are replaced by measured serum concentrations.

Cardno Lane Piper (CLP) has produced a series of reports that document the site investigation and chemical concentrations in various media at Fiskville. To enable this report to be read as a standalone document, relevant analytical data have been extracted from the CLP reports to provide contextual information. Nevertheless the reader is encouraged to consult the cited CLP reports for the complete analytical data and how it was gathered and quality assessed.

### 2. PFC concentration in water and fish

Detailed information on the concentrations of PFCs in Lake Fiskville and organisms in the Lake and the recycled water dams at Fiskville can be found in the Cardno Lane Piper reports entitled "*Surface Water and Sediment Contamination Assessment*" (CLP 2013c) and "*Ecological Assessment*" (CLP 2013b). For completeness and ease of reading a summary of the relevant data is provided herein.

#### 2.1 PFCs in Lake Fiskville

Table 2.1 summarises the PFC concentrations in Lake Fiskville. There were measureable concentrations of eight PFCs in the water column and three in sediment. Of these PFOS has the highest concentration. A glossary of PFC nomenclature and abbreviations can be found in Appendix A.

#### 2.2 PFCs in fish

The analysis of PFCs in biological matrices is not straightforward. In particular for PFOS there is potential, but inconsistent interference by unknown substances<sup>1</sup>. In addition, the literature (van Leeuwen et al. 2006, Malinsky 2009) indicates there can be marked variability within and between laboratories. The inclusion of stable isotope internal standards largely, but not completely, overcomes these issues (van Leeuwen et al. 2009). The analytical program for Redfin muscle analysis was cautiously designed by Cardno Lane Piper to include tissue duplicates, laboratory duplicates, split muscle samples for inter-laboratory comparison, and replicates. While there were instances of poor recovery of internal standard and poor replicates, Cardno Lane Piper undertook a careful quality

<sup>&</sup>lt;sup>1</sup> Personal communication with National Measurement Institute, Sydney and AsureQuality analytical services, New Zealand.



control examination of the data (CLP 2013a)<sup>2</sup> and concluded the analyses were accurate and could be relied upon.

Table 2.2 summarises the PFC concentrations in a range of organisms sampled from Lake Fiskville in December 2012. The information in the table is derived from CLP (2013d, e)<sup>3</sup>. In all organisms it is apparent that PFOS bioaccumulates to a much greater extent (by 3 – 4 orders of magnitude) than do other PFCs. This is consistent with the scientific literature (Conder et al. 2008, de Silva et al. 2011, Giesy et al. 2010, Haukås et al. 2007, Houde et al. 2011, Martin et al. 2003a, b, 2004; Morikawa et al. 2006), and that different organisms bioconcentrate PFOS to different degrees. Redfin are at the top of the aquatic food chain in Lake Fiskville and therefore biomagnify PFOS the greatest (McDowell 1996, as cited in CLP 2013b; NSW DPI 2014; Waterwatch Vic undated, Humphries and Walker 2013). While it may appear Mosquito fish and yabby have taken up a range of PFCs dissimilar to those in Redfin muscle this is probably because the former animals were analysed whole (i.e. included internal organs). Redfin liver contained the same PFCs as Mosquito fish and yabby (CLP 2013b); the redfin liver data is not replicated in this report because it is a tissue not eaten by humans.

<sup>&</sup>lt;sup>2</sup> The information contained in CLP (2013a) is also available in CLP (2014a, b).

<sup>&</sup>lt;sup>3</sup> The information contained in CLP (2013d) is also available in CLP (2014a, b).



PFC	Sediment (ng/g)	Water (ng/mL)		
PFBA	-	-		
PFPeA	-	-		
PFBS	ND	1.4 <sup>b</sup>		
PFHxS	12.6 <sup>b</sup>	4.4 <sup>b</sup>		
PFOS	225 <sup>a</sup> (57 – 785)	13.3 <sup>a</sup> (8.8 – 17.7)		
PFDS	ND	ND		
PFHxA	ND	4.8 <sup>b</sup>		
PFHpA	ND	0.7 <sup>b</sup>		
PFOA	ND <sup>c</sup>	0.58 <sup>a</sup> (0.48 – 0.76)		
PFNA	ND	0.04 <sup>b</sup>		
PFDA	ND	ND		
PFUdA	ND	ND		
PFDoA	ND	ND		
PFTrDA	ND	ND		
PFOSA	ND	ND		
NEtFOSA	ND	ND		
NEtFOSAA	-	-		
NMeFOSA	ND	ND		
NMeFOSAA	-	-		
NEtFOSE	ND	ND		
NMeFOSE	ND	ND		
4:2 FtS	-	-		
6:2 FtS	12.8 <sup>a</sup> (<5 – 24)	5 <sup>a</sup> (3.5 – 7.4)		
8:2 FtS	-	-		

#### Table 2.1: PFCs in sediment and water of Lake Fiskville.

ND = not detected;

- = not in analytical suite.

<sup>a</sup> The data are the average (range in brackets) PFC concentration measured in August 2012 at various locations/depths in the lake. It should be noted that for PFCs other than PFOS, PFOA and 6:2FtS only one sample of water and sediment (LFWE2.0/06082012 or LFSE0.1/02082012) was analysed for the complete suite of PFCs. Information in the table has been compiled from data in CLP (2013c) and ALS analysis certificates (EM1208900,

EM1208979, EM1209107) provided by CLP for the water and sediment sample that underwent full PFC analysis. b

Data are the average of the primary sample and its laboratory duplicate.

<sup>c</sup> For PFOA in sediment 4 of 5 measurements were below the LoR (0.0005 mg/kg), one measurement was marginally above the LoR (0.0007 mg/kg).



			Organism			
PFC conc (ng/g)	<b>Redfin</b> muscle n=21 <sup>b</sup>	Mosquito fish whole n= 3	Yabby whole n=4	Freshwater shrimp whole n=1	Macrophyte <sup>d</sup> n=3	
PFBA	ND	-	-	-	-	
PFPeA	ND	ND	6 (3.4 – 11)	2.1	3.3 (<2 – 6.2)	
PFOS	9,906 (4,200- 23,500) <sup>c</sup>	38,667 (30,000–50,000)	2,540 (560 – 5,000)	260	1,040 (440 – 1,440)	
PFHxA	ND	ND	<b>2.8</b> (1.2 – 6.9)	2.1	4.8 (3.2 – 5.7)	
PFHpA	ND	2 (<2 – 2.5)	2.6 (<2 - 4.4)	ND	ND	
PFOA	ND	3.3 (2.3 – 4.5)	20.5 (18.2 – 23.2)	ND	1.7 (<2 – 3.2)	
PFNA	ND	4.7 (2.3 – 6.4)	7.6 (4.5 – 10.8)	2.3	ND	
PFDA	8.1 (4.3 – 13)	9.2 (6.3 – 12.3)	7.4 (2.4 – 15.1)	2.2	ND	
PFUdA	28 (14 – 45.8)	33.6 (25 – 40.2)	26.6 (5.8 – 51.6)	2.5	2 (<2 – 2.6)	
PFDoA	1.9 (<2 – 3.5)	3.4 (2.6 – 3.7)	14.8 (<2 – 40.1)	ND	ND	
6:2 FtS	3.6 (1.3 – 5.3)	-	-	-	-	
PFBS <sup>e</sup>	ND	-	-	-	-	
PFHxS <sup>e</sup>	11.8 (6.5 – 16)	-	-	-	-	
PFDS <sup>e</sup>	16.5 (11 – 22)	-	-	-	-	
PFTrDA <sup>e</sup>	3.1 (1.6 – 4.8)	-	-	-	-	
PFTeDA <sup>e</sup>	ND	-	-	-	-	
PFOSA <sup>®</sup>	2.2 (1.7 – 2.8)	-	-	-	-	
NEtFOSAA <sup>e</sup>	ND	-	-	-	-	
NMeFOSAA <sup>e</sup>	ND	-	-	-	-	
8:2 FtS <sup>e</sup>	25.4 (20 – 32)	-	-	-	-	

#### Table 2.2: PFC concentrations in organisms sampled from Lake Fiskville <sup>a</sup>

n = number of specimens; - = not analysed; ND = Not Detected. Values are mean concentrations with the range provided in parenthesis.

<sup>a</sup> This table is compiled from a Cardno Lane Piper (CLP 2013d) file note<sup>4</sup> and spread sheet (CLP 2013e) as supplied in email from Ashurst 15/08/2013 for sampling undertaken in December 2012 at Lake Fiskville. Measurements reported as less than the detection limit were assumed to be at half the detection limit for calculation of an average. Depending on the PFC, batch run or organism type, limits of detection were 0.5, 1, 2 or 5 ng/g.

Note the units (ng/g) are as reported by the analytical laboratory, elsewhere in this report they have been converted to (mg/kg) for ease of comparison with other information.

<sup>b</sup> Redfin data is for 21 specimens, but the calculated mean value includes laboratory duplicate and replicate samples for a maximum total of 34 results for PFOS, PFOA and some other PFC's. In addition not all fish were analysed for all PFCs, so there may also be less than 21 values for calculating an average, see also Footnote 'e'.

<sup>&</sup>lt;sup>4</sup> The information in CLP (2013d) is also available in CLP (2014a, b).



<sup>c</sup> There were two fish with PFOS concentrations of about 23,000 ng/g that were analysed in the initial batch of 4 fish from Lake Fiskville, these were the largest of the redfin that were caught. These two fish were stored frozen and reanalysed by the same laboratory (NMI) some months later and returned concentrations of 15,000 ng/g; PFOS is very stable and freezing and thawing is not expected to result in degradation of PFOS, it may however change the matrix of the fish such that less interfering substances are co-extracted with PFOS. Recent developments for analysing PFCs in fish include a freezing step to enhance protein precipitation after tissue has been homogenised with extraction solvent (Malinsky 2009, Malinsky et al. 2011). In calculating the average all data has been used. The average without the additional analysis of the two fish is 8,260 ng/g. Additional information on the impact of the replicates of these fish is depicted in Figure 2.1.

<sup>d</sup> A macrophyte is an aquatic plant (*Potamogeton sp.* is a species of pondweed).

<sup>e</sup> These PFCs were only reported by AsureQuality in eight redfin samples used for inter-laboratory comparison. Averages for other PFCs include the results from both NMI (the primary analytical laboratory) and AsureQuality, with the exception of PFBA and PFPeA. The latter PFCs were only reported by NMI.

From the analysis of PFCs in water of Lake Fiskville and associated biota it is patent that PFOS is the PFC of potential concern. In comparison to concentrations of PFOS in Lake Fiskville and biota, the measured levels of other PFCs were not significant. Importantly PFOA was not detected in Redfin muscle.

Information obtained in consultation with CFA Fiskville personnel indicated anglers kept all redfin that were caught at the Lake, regardless of size. An examination of PFOS concentration in Redfin muscle with fish size shows only a weak correlation (Figure 2.1). This is consistent with other investigations which have found PFOS concentrations in fish muscle within or between species were not positively correlated with fish age or size (Becker et al. 2010, De Silva et al. 2011, Exponent 2011, Hoff et al. 2003, Martin et al. 2004, MPCA 2010, Murakami et al. 2011).

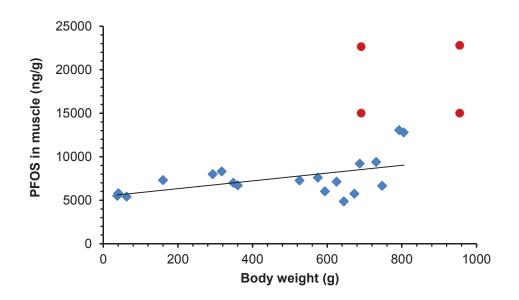
The concentration of PFOS in Redfin muscle, across a wide range of fish sizes (approximately 40 – 800g), is about 5,000 – 13,000 ng/g fish <sup>5</sup>. These fish concentrations are approximately ten times higher than levels at which a number of international authorities have made recommendations the fish should not to be eaten (Dutch VWA 2008, German FIRA 2006, Alabama DoPH undated, Minnesota MDH 2008, Ontario MoE 2013). Fish advisories set by various authorities use quite conservative assumptions about lifetime patterns of exposure. Consuming fish with higher concentrations occasionally, or for a short period, does not automatically mean unacceptable health risk for the person, or that adverse health effects will occur. The fish advisories are discussed in more detail in Appendix F.

The availability of serum PFOS concentrations in persons who have acknowledged eating fish from Lake Fiskville negates the need to undertake a 'traditional' risk assessment based on PFOS fish concentrations and assumptions about how much fish, or eel, were caught and eaten. If such an assessment were to be done, it is the average PFOS concentration in the consumed flesh that is most

<sup>&</sup>lt;sup>5</sup> This range excludes the two fish that initially analysed at approximately 23,000 ng/g but on re-analysis returned 15,000 ng/g.



appropriate to use for exposure estimations. However it is inappropriate<sup>6</sup> to use a tolerable daily intake (TDI) for risk characterisation when exposure is known to be infrequent and potentially for just a few years (i.e. a small fraction of a lifetime).



#### Figure 2.1: Correlation of PFOS concentration in Redfin muscle with fish size.

The red data points are for two fish originally returning PFOS concentrations of 22,800 and 22,650 ng/g. Reanalysis of these tissue samples sometime later gave results of 15,000 ng/g for each. Since there is uncertainty regarding these data they have not been included in the regression analysis.

The equation of the line is y = 4.4693x + 5441.2;  $R^2 = 0.273$ . Thus there is no positive association between PFOS concentrations in redfin muscle and the size of the fish. This is consistent with literature information. If the red data points are included in the regression analysis, the equation is y = 10.821x + 3734.4;  $R^2 = 0.351$ .

#### 2.3 Other substances in fish

In addition to PFCs, the initial four Redfin were also analysed for metals (arsenic, cadmium, chromium, copper, lead, mercury, nickel and zinc).

<sup>&</sup>lt;sup>6</sup> It is inappropriate to use the common risk characterisation method in these circumstances because the TDI is established on the assumption the food commodity is eaten every day for a lifetime (70 yrs). In the situation at Fiskville fish were eaten infrequently for relatively few years; averaging the total intake of PFOS over a life time dilutes the potential risk. For substances with long half-lives it is possible the total intake over a short period may increase body burden (measured as serum concentration) to levels potentially associated with changes in biomarkers of certain common diseases. This may not be recognised if intake was averaged over a life time in order to match the TDI. In addition marked uncertainty with regard to estimating intake of PFOS by persons at Fiskville via their historical fish consumption renders comparison with the TDI spurious.



Concentrations of arsenic, cadmium, chromium, lead and nickel in Redfin muscle were less than or marginally greater than the limit of reporting (0.01 or 0.05 mg/kg wet weight). Concentrations of copper (0.09 – 0.25 mg/kg ww) and zinc (3.4 – 4.3 mg/kg ww) were well within background concentrations in fish (Arellano et al. 1999, Zeynali et al. 2009, Jones et al. 2000) and below maximum residue limits (MRLs) for human consumption (APVMA 2013, EFSA 2012a).

Mercury in Redfin muscle ranged from 0.42 - 0.59 mg/kg wet weight (mean 0.48 mg/kg ww). The mean concentration in the four fish was just below the MRL of 0.5 mg/kg (FSANZ 2013). None of the participants in surveillance program had elevated blood mercury concentrations that were associated with eating fish from Lake Fiskville (Section 3.3).

## 3. Health surveillance program

#### 3.1 Overview

For some time CFA have had a health surveillance program in place for its personnel. This was extended on a voluntary basis to all persons and their families who had eaten fish from Lake Fiskville, or had concerns about other possible exposure to PFCs at Fiskville. Entry into the program was not restricted to CFA personnel. Fiskville staff were informed verbally and by newsletter of the program, and advertisements were run in the local newspaper. People who thought they knew someone who might have eaten fish from Lake Fiskville were encouraged to inform them of the program, or give CFA hygiene staff their name so they may be contacted. Where possible these persons were contacted by telephone.

In addition to obtaining a blood sample for analysis of PFCs, all persons had additional blood taken for measurement of heavy metals and, as per the existing program, for haematology parameters and clinical chemistry screening that included tests for liver, kidney and thyroid function <sup>7</sup>. A detailed list of

<sup>&</sup>lt;sup>7</sup> The blood sampling program was coordinated by the Organisational Health & Wellbeing department of the CFA. Blood was obtained by a trained phlebotomist from a pathology laboratory engaged by the medical officer. The pathology laboratory also prepared serum and organised sample shipment to the laboratory measuring PFCs. Blood chemistry parameters and heavy metals were done using standard techniques employed by the pathology laboratory with results reported against the population reference range used by the laboratory.

Serum PFC analysis was undertaken by the National Measurement Institute (NMI). The method of determination was by High Performance Liquid Chromatography tandem Mass Spectrometry (HPLC-MS-MS). Prior to extraction the sample was spiked with a range of isotopically labelled surrogate standards followed by solid phase extraction. An aliquot of extract was injected onto the HPLC and separated PFCs detected and quantitated using mass spectrometry. Results were corrected for recovery of labelled surrogates. Included in batch analysis runs were calf serum matrix blanks that had, or had not, been spiked with known amounts of PFCs.



tests and the suite of PFCs looked for in serum is in Appendix C. Furthermore individuals had their medical history obtained and a general medical examination by the contracted medical officer. At the examination the medical officer made enquiries regarding medications they may be taking and when and how much fish they may have eaten from Lake Fiskville.

All persons entering the program were adults and agreed to have the results of their tests made anonymously available for evaluation. However as explained to all participants this was not a condition of entry into the program. Only the medical officer was aware of the identity of the people in the program, he presented the de-identified data to the consulting toxicologist, who with the medical officer interpreted the information.

#### 3.2 Data interpretation

Information from the general medical screening part of the health surveillance program was evaluated as is usually done by medical practitioners. That is, an individual's blood parameters were interpreted against population reference ranges in conjunction with their medical history and condition, the concomitant medical examination, and the medical expertise of the medical officer.

An important consideration is that clear adverse effects of PFOS have only been documented in animal studies and the effects are directly related to PFOS serum concentrations in the animals. When interpreting serum PFOS concentrations in the Fiskville cohort it also needs to be remembered that the measurement represents an aggregation of several modes of potential exposure. These include background exposure, possible past consumption of fish, and perhaps also historical exposure to firefighting foams that contained PFOS. Included in the cohort were some of the PAD operators.

To interpret the PFCs measured in the serum of program participants, two 'indicator' serum concentrations were constructed as comparators (see Appendix B for details). These comparison serum concentrations are:

1. **Background serum levels** usually present in adult populations (Appendix B.1). The PFCs are ubiquitous in the human environment and are found in serum as a result of day-to-day living. The majority of people are expected to have background serum concentrations of:

PFOS is the PFC of concern. Recovery of PFOS from spiked samples ranged from 6 - 124%. Although some recoveries (5 of 22 samples i.e. 23%) were below the ideal range (25 - 125%) of the laboratory, the laboratory considered PFOS to be suitably quantitated due to the inclusion of internal standards in the analysis. Relative Percentage Difference (RPD) of duplicate analysis (n = 2 of 22 analyses) for PFOS was 4 and 10%, this is considered to be acceptable.



- PFOS <0.1 mg/L, and
- o PFOA <0.05 mg/L
- 2. A serum concentration that is without adverse effects, i.e. a serum no observed effect level (NOEL). Several lines of evidence are presented in Appendix B.2 that indicate a serum PFOS concentration of 2 mg/L (2,000 ng/mL)<sup>8</sup> is a level at which, with current knowledge, it can be confidently stated no effects are likely to be observed in adult individuals. The evidence supporting this serum NOEL comes from:
  - a) Epidemiology studies in workers making or handling PFOS who individually have serum concentrations up to 13 mg/L (i.e. about 2 – 4 orders of magnitude higher than the mean levels for non-occupationally exposed populations).
  - b) NOELs observed in monkey and rat experiments where the animals have purposefully been administered high doses of PFOS. The very high serum concentrations produced allows determination of the potential effects of PFOS, the dose response, and the NOEL for the effects. These animal serum NOELs when converted to an equivalent human serum NOEL using the standard default uncertainty (safety) factors for deriving toxicity reference values from animal information give values of 3.3 – 4.4 mg/L. The process is briefly described below and in detail in Appendix B2.2.

In a six month monkey study the most sensitive effects were decreased serum cholesterol, decreased high density lipoprotein (HDL) and slightly decreased circulating total triiodothyronine ( $T_3$ ) (Seacat et al. 2002, EFSA 2008). The lower 95% confidence limit on the benchmark dose (as serum concentration) is 35 mg/L (MDH 2008), this was divided by 2.5 to account for differences in toxicodynamics<sup>9</sup> between monkey and human and 3.2 for toxicodynamic differences between humans as per the recommendations of enHealth (2012) and WHO (2004, 2010) to yield a human serum NOEL of 4.4 mg/L.

<sup>&</sup>lt;sup>8</sup> Cross sectional epidemiology studies in communities affected by PFOA in drinking water have shown weak positive associations of relatively low PFOA serum concentrations (measured or predicted) with increased serum cholesterol and fatty acids in adults, kidney and testicular cancer and hypothyroidism in children. No such associations have been observed for PFOS. While PFOS and PFOA share a number of common toxicological properties there are also significant differences (primarily in tumourigenicity and reproductive/developmental toxicity, the latter being the most sensitive effect as determined from toxicological studies). Furthermore PFOA is not a substance of concern at Fiskville. It is therefore inadvisable to extrapolate toxicological or health information for PFOA either to PFOS or to the circumstances of PFC exposure at Fiskville.

<sup>&</sup>lt;sup>9</sup> The lower bound benchmark dose, as a serum concentration, (BMDL) is an outcome of mathematical modelling of the dose response (using either experimental serum concentrations or doses which are subsequently converted to serum concentrations). The BMDL is used in deriving guidelines and standards in a



From a variety of rodent studies the most sensitive effects of reduced pup weight at birth, neonatal weight gain and survival are found in rat two generation reproduction and developmental studies (Lau et al. 2003, Thibodeaux et al. 2003a, b; 3M Company 2003, Luebker et al. 2005a, b; Lau et al. 2007). The serum PFOS BMDL<sub>5</sub> (lower bound benchmark dose for 5% effect) for decreased neonatal weight gain was 26 - 31 mg/L and for reduced survival 83 - 100 mg/L. Applying the same toxicodynamic uncertainty factors to the lower serum concentrations (i.e. to the most sensitive effect) as for the monkey serum BMDL gives an equivalent serum NOEL for humans of 3.3 - 3.9 mg/L.

c) Conversion of the PFOS exposure guideline (the tolerable daily intake, TDI) established by the European Food Standards Authority (EFSA 2008) to a serum concentration using human toxicokinetic information (Appendix B2.3). The TDI is an intake in units of µg PFOS/kg body weight /day that is considered not to cause adverse effects to people exposed every day over their lifetime. The serum concentration associated with the TDI therefore represents a steady state concentration. Given that the half-life of PFOS in humans is 5.4 years (EFSA 2008), steady state serum concentrations will be achieved after approximately 20 – 27 years of daily exposure at the TDI (i.e. after 4 – 5 half-lives). Using standard one compartment pharmacokinetic equations for a daily dose at the EFSA TDI of 1.5 µg/kg/d yields a steady state serum concentration of 2 mg/L.

**In summary**, the interpretation of PFOS and PFOA measured in serum of persons at Fiskville has been achieved by:

- 1. Comparison with general population background serum concentrations where the majority of adults are for:
  - PFOS <0.1 mg/L.
  - PFOA <0.05 mg/L.

similar manner as the experimental NOEL but is considered to be a better estimate of the true NOEL than the experimental value (enHealth 2012, EFSA 2009, Gezondheidsraad 2003, US EPA 2012). Because the BMDLs for PFOS are expressed as serum concentrations that elicit the effect, the toxicokinetic processes that influence the serum concentrations associated with any given daily dose of PFOS are inherently incorporated into the assessment process. Thus only potential tissue responsiveness differences (i.e. toxicodynamic differences) need to be accounted for when converting an animal serum NOEL (i.e. the BMDL) to an anticipated human serum NOEL that can be used in risk assessment. This would not be the case if the BMDL's were expressed as an external dose of mg/kg/d instead of an internal dose of mg/L serum. As applied in this risk assessment the NOEL serum concentrations relate to presumed steady state concentrations.

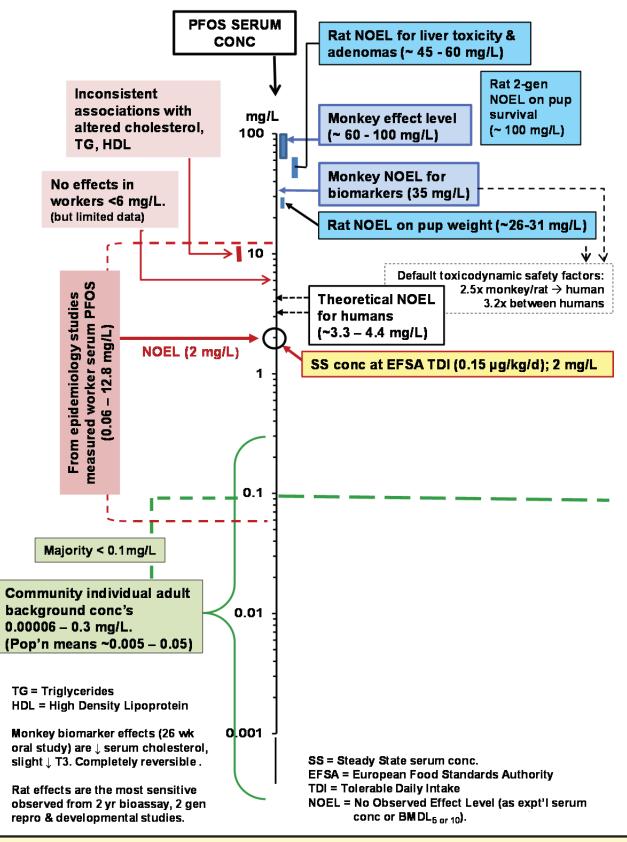


- 2. Comparison with a human NOEL of 2 mg/L for PFOS, derived from:
  - a) Occupational epidemiology studies.
  - b) NOELs in animal toxicology experiments for:
    - Reversible changes in blood cholesterol, lipids and thyroid hormone in monkeys.
    - Decreased neonatal weight gain from rat two generation and developmental studies.
  - c) Conversion of the European Food Standards Authority (EFSA 2008) tolerable daily intake to an achieved steady state serum concentration.

In addition to comparison with the above PFOS 'reference' serum concentrations, margins of exposure (MOE) were calculated (Section 4.2).

The various human and animal data discussed above and in detail in Appendix B are summarised in Figure 3.1.





**Figure 3.1: Summary of serum concentration (dose) - response for critical effects of PFOS in animals and humans.** Also shown are adult background serum concentrations and the serum NOEL of 2 mg/L for humans. The latter is derived from occupational epidemiology studies, animal toxicology investigations, and the steady state serum concentration associated with the tolerable daily intake set by the European Food Standards Authority.



#### 3.3 Health surveillance results

#### 3.3.1 General considerations

In order to preserve the privacy of persons who participated in the health surveillance program only broad précises of the data are provided in this report.

Twenty two persons availed themselves of the surveillance program; just over half indicated they had eaten fish or eel from Lake Fiskville in the past. In terms of environmental epidemiology studies this is a very small number of persons potentially exposed to PFOS through eating fish. Accordingly it is difficult to draw general conclusions from the data. Much caution should be used when weighing up the information provided in this section.

Table 3.1 summarises some of the features of the people who joined the program. PFOS serum concentrations are discussed in Section 3.3.2.

There were slightly more males and females in the group that ate fish from Lake Fiskville as in the group that did not eat fish. The age range in each group is about the same<sup>10</sup>.

However, for the people who had their body mass index (BMI) recorded, there were apparent differences in the BMI between the two groups (Figure 3.2). Overall only 2 people in the entire cohort had a BMI considered to be healthy<sup>11</sup>, one fish eater and one non-fish eater. In the group that ate fish from Lake Fiskville, 42% had BMI's considered to be in the obese range compared to 29% in the non-fish eating group. Importantly, BMI was not correlated with PFOS serum concentrations for either the whole cohort or just the fish eater group<sup>12</sup> who do have higher serum PFOS levels (Table 3.1, Section 3.3.2, Figure 3.3).

Given the small group sizes no importance can be placed on the apparent differences in BMI between the two groups; it is likely to be a random finding. However whether a person is overweight or obese has implications for interpreting their individual clinical chemistry data.

• All persons: y = -0.0068x + 30.9,  $R^2 = 0.013$ .

<sup>&</sup>lt;sup>10</sup> In the group that indicated they ate fish there was a septuagenarian person who is not a current employee of CFA. The next oldest male in this group is in his early sixties.

<sup>&</sup>lt;sup>11</sup> The following BMI based health categories are for young and middle-aged adults (Vic Govt 2013):

o 18.5 to 24.9 - healthy weight range (22-26 may be acceptable for older Australians).

o 25.0 to 29.9 - overweight.

<sup>○ &</sup>gt; 30 – obese.

<sup>&</sup>lt;sup>12</sup> The regression analysis equations for the correlation of BMI and PFOS serum concentration are:

<sup>•</sup> Fish eaters only: y = 0.0138x + 32.8,  $R^2 = 0.067$ .



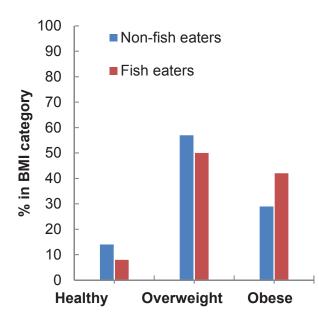


Figure 3.2: Body Mass Index (BMI) of persons enrolled in the health surveillance programme.

	Non-fish eaters <sup>a</sup>	Fish eaters <sup>a</sup>
Demographics		
Total persons	10	12
# Females (~age range) <sup>b</sup>	4 (30 - 50 yrs)	5 (35 – 60 yrs)
# Males (~age range) <sup>b</sup>	4 (45 - 50 yrs)	7 (50 – 70 yrs)
BMI: % overweight	57	50
BMI: % obese	29	42

Table 3.1:	Summary	of cohort	characteristics.
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<sup>a</sup> The 'fish eater' descriptor refers to whether or not an individual indicated they had eaten at any time, fish, eels or yabbies that were from Lake Fiskville. Personal data, including the age of the person, was inadvertently not collected for all people in each group. Consequently the numbers of males and females do not add up to the total persons.

<sup>b</sup> To preserve anonymity the age range has been rounded to the nearest 5 years. .



#### 3.3.2 PFC serum measurements

Of the 10 PFCs looked for in human serum (Appendix C) only two were present at measurable concentrations. These were PFOS and PFOA.

#### PFOA:

All PFOA measurements were approximately an order of magnitude less than the expected background concentrations of <0.05 mg/L (Section 3.2 and Appendix B.1) arising from day to day living. The data indicate fish consumption has not contributed to human PFOA serum concentrations. This is not unexpected since redfin (the fish being consumed) did not have measurable concentrations of PFOA in their flesh (Table 2.2).

#### PFOS:

A summary of the serum PFOS concentrations is at Table 3.2. Perhaps not surprisingly the average PFOS serum concentration in the group that self-reported to have eaten fish was higher than in the non-fish eating group, but not statistically significant<sup>13</sup>. Four persons had concentrations higher than the expected background concentration of <0.1 mg/L. The implications of the PFOS measurements are discussed in the risk characterisation section (Section 4).

	Non-fish eaters <sup>a</sup>	Fish eaters <sup>a</sup>
Serum PFOS (mg/L) <sup>b</sup>		
Average	0.016	0.085
Range <sup>c</sup>	<0.005 - 0.07	0.002 – 0.4
# persons > background <sup>d</sup>	0	4

#### Table 3.2: Summary of PFOS serum concentrations

<sup>a</sup> The 'fish eater' descriptor refers to whether or not an individual indicated they had eaten at any time, fish, eels or yabbies taken from Lake Fiskville.

<sup>b</sup> Of the suite of PFCs looked for in serum, only PFOS and PFOA were measureable. PFOA concentrations were all below background, consequently only PFOS serum concentrations are reported in this table. The PFC analysis method and QA/QC data are described in a footnote to Section 3.3.1. PFC concentrations are reported by the laboratory as ng/mL but for consistency within this report have been converted to mg/L.

<sup>c</sup> To preserve anonymity the values provided have been rounded to one or two significant figures. Due to matrix effects the analytical limit of reporting (LOR) differs slightly between samples. For the entire cohort the LOR's were 0.002 – 0.01 mg PFOS/L serum.

<sup>d</sup> Background PFOS serum concentrations are expected to be <0.1 mg/L (Section 3.2 & Appendix B.1).

<sup>&</sup>lt;sup>13</sup> An unpaired t-test of unequal variances showed mean PFOS concentrations in fish eaters is not statistically different from that in non-fish eaters.



#### 3.3.3 Blood chemistry results

Individual blood chemistry information was assessed by the medical officer who personally discussed them, together with the PFOS results with the person involved.

There were a number of persons, in both the fish eating and non-fish eating groups, that had some clinical chemistry parameters outside of the normal range that signalled increased risk of disease. There were also persons whose blood parameters were abnormal as a result of life style factors, existing disease, or medication. Where necessary the medical officer wrote a referral for the person to follow up with their own general practitioner.

For no individual were blood parameters related to their serum PFOS concentrations.

In addition regression analysis of blood parameters for the whole cohort, or for just the fish eaters, showed no trend association of any parameter with PFOS concentrations (Appendix D).

### 4. Risk characterisation

#### 4.1 Comparison with referent serum concentrations

The two comparator serum concentrations used for risk characterisation in this report are 0.1 mg/L (a concentration which the majority of people are expected to be below if they are only exposed to background sources) and 2 mg/L (a concentration deemed to be without adverse clinical effects) (Section 3.2 and Appendix B).

As discussed in Section 3.3.2, all except four persons had PFOS concentrations less than 0.1 mg/L. Those who returned levels above the background concentration of 0.1 mg/L were at least 5 times less than the serum NOEL of 2 mg/L (Figure 4.1).

Unfortunately information is poor on how much fish or eel was eaten and how long ago. Consequently, due to the considerable uncertainty, it is not proper to construct exposure scenarios and attempt to predict by toxicokinetic modelling serum PFOS concentrations that may have arisen from eating fish. It is however germane to consider that sometime in the past an individual may have had higher serum PFOS concentrations than has been currently measured. However, there is no indication in the consumption information provided by the four persons who have higher than background PFOS levels that they ate more fish, or more frequently, in the past 5 - 10 years than in recent years. The difference between the current serum concentrations



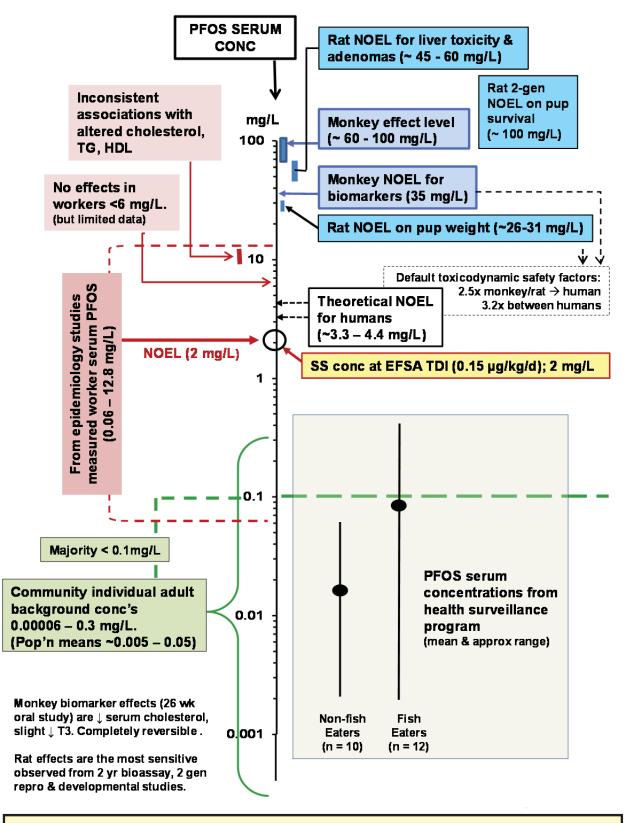


Figure 4.1: Comparison of measured serum PFOS concentrations with critical levels in animals and humans. See Figure 3.1 for explanation of abbreviations.



and the serum NOEL and the calculated MOE's (see Section 4.2) are sufficient to cater for this uncertainty. For example, if it is speculated that a person was once a consumer of fish from the Lake but stopped 5 - 6 years ago, then based on the highest current serum concentration measured in the surveillance program cohort that person's PFOS level 5 - 6 years ago would still be less than half <sup>14</sup> the serum NOEL. Similarly if a person stopped consuming fish from the Lake about 11 years ago their serum PFOS at that time, in order to give rise to the highest current serum concentration, would have been approximately 70% of the serum NOEL.

Based on these considerations there is low likelihood of adverse health effects having arisen, or arising from PFOS concentrations in these persons.

A letter from the consulting medical officer and toxicologist has been written to the CFA Chief Executive Officer expressing this opinion (Appendix E).

#### 4.2 Margin of Exposure calculations

An additional technique commonly used for judging the potential health impact of chemical exposure is to calculate a margin of exposure (MOE) against NOELs derived from well conducted animal experiments (enHealth 2012, EFSA 2012b, WHO 2004, 2010). These studies are described in Appendices B2.2 and B.3. In Australia, public health risks that may arise from use of agricultural chemicals, veterinary chemicals applied to food producing animals, or from non-occupational exposure to industrial chemicals are deemed to be acceptable if the MOE, based on exposure dose, is equal to, or greater than 100 (APVMA 2006, NICNAS 2007). This MOE is informally based on the 10 x 10 fold safety factor<sup>15</sup> widely used to account for uncertainty in intra- and inter-species differences in the effects of chemicals. It addresses toxicokinetic and toxicodynamic differences between animals and between humans. However since 'exposures' in the surveillance program are measured as serum concentration rather than the external applied dose, toxicokinetic variability between animals and humans is inherently assimilated into the MOE calculation when using serum concentrations. Hence the usual acceptable MOE of 100 needs to be adjusted to account for the inherent inclusion of toxicokinetic species differences in calculating the MOE. This is particularly the

<sup>&</sup>lt;sup>14</sup> The half-life of PFOS in humans is approximately 5.4 years (EFSA 2008). This is the time for the serum concentration to decrease by half. If the highest current PFOS serum concentration is 5 times less than the serum NOEL for humans (i.e. about 0.35 mg/L), then 5.4 years ago in the absence of further exposure the concentration would be around 0.7 mg/L (i.e. less than half the human serum NOEL). Eleven years ago the serum concentrations in this hypothetical person may have been 1.4 mg/L (70% of the serum NOEL of 2 mg/L).

 $<sup>^{15}</sup>$  The 10 x 10 safety factors (also called uncertainty factors) are firstly for interspecies differences (between animal and human) in toxicodynamics (tissue responsiveness) and toxicokinetics (chemical metabolism) respectively, these are 2.5 x 4 respectively, and secondly for interindividual differences between humans in toxicodynamics (3.2) and toxicokinetics (3.2) (enHealth 2012).



case for compounds such as PFOS which aren't metabolised and whose distribution in the body is confined to extracellular water (i.e. primarily serum) and effects are directly related to serum concentrations. Thus an acceptable MOE based on serum measurement in humans and serum NOEL in animals would be 25 (100  $\div$  4) <sup>16</sup>.

In this HRA, MOE's for a number of toxicological end points identified in animal studies have been calculated. Developmental effects in rodents are the most sensitive ones observed in animal studies (Appendix B) and patently this endpoint is only germane for females of reproductive age. These persons are also therefore the most sensitive sub-population. Thus:

For males and females  $\geq$  45 years MOEs are calculated with:

- Serum NOELs (35 mg/L) from monkey experiments for the same blood parameters as evaluated in the health surveillance program (Seacat et al. 2002).
- Serum NOELs for sensitive effects in chronic toxicity studies.
  - 60 mg/L for production of liver adenomas in a two year bioassay (Butenhoff et al. 2012b, Thomford 2002, 3M Company 2003).
  - 45 mg/L for liver toxicity in a two year bioassay (Butenhoff et al. 2012b, Thomford 2002, 3M Company 2003).

For females ≤ 45 years (i.e. considered to be of reproductive age [DFG 2005, 2013]) MOEs are calculated

- As above, plus
  - 26 mg/L in maternal serum for decreased weight gain in offspring in two generation and/or developmental rodent studies (Luebker et al. 2005a, 2005b) (Appendix B).

In order that potential reproductive risk (low birth weight) is addressed to the extent possible, females of reproductive age ( $\leq$  45 years old) have been assessed as a separate group. There are currently only 3 persons in this category, but based on the current ages of females in the cohort, five and ten years ago there were potentially 6 and 8 females in the cohort who were  $\leq$ 45 years old. Assuming these persons were eating fish from the Lake up to that time but stopped 5 or 10 years ago their serum PFOS concentrations would have to have been higher to account for the current measured concentrations. Using an approximation of the current maximum serum PFOS concentration that is

<sup>&</sup>lt;sup>16</sup> In this calculation the divisor of 4 is the toxicokinetic uncertainty factor used in risk assessments and public health guideline setting that addresses toxicokinetic differences between animals and humans (i.e. the interspecies uncertainty factor,  $AK_{UF}$ ) (enHealth 2012, WHO 2005). That is the toxicokinetic differences between humans (3.16), toxicodynamic differences between animals and humans (2.5) and toxicodynamic differences between humans have been retained (3.16) in the MOE for a total of 25.



higher than actual for females, serum concentrations for hypothetical females eating fish 5 or 10 years ago have been estimated (Table 4.1).

All MOEs, except one, are larger than the acceptable MOE of 25 (Table 4.1). This indicates low potential for health effects, either now or in the past. The MOE that is lower than the acceptable value is for a theoretical female of reproductive capacity who, ten years ago, may have had serum concentrations markedly higher than the current maximum female concentration. Given that this MOE of 22 is only marginally less than acceptable, and the approximations that have been made in the MOE calculations, this MOE of 22 is not an indication of unacceptable risk at that time.

# Table 4.1: Margin of Exposures (MOEs) for current and assumed past serum PFOS concentrations <sup>a</sup>

			<b>MOEs</b> (Calculated against serum NOELs from animal studies)			
			Animal serum NOEL (Critical effect)			
Person category <sup>b</sup>	Approx max human serum conc (mg/L) <sup>c</sup>		26 mg/L (Developmental -rodent)	35 mg/L (Serum biomarkers - monkey)	45 mg/L (Chronic liver tox - rat)	60 mg/L (Liver adenomas – rat bioassay)
Male or	Current	~ 0.35 <sup>c</sup>	N/A <sup>f</sup>	100	128	171
Female (>45 yrs)	5 yr ago	~ 0.7 <sup>d</sup>	N/A <sup>f</sup>	50	64	86
	10 yr ago	~ 1.4 <sup>d</sup>	N/A <sup>f</sup>	25	32	43
Female <sup>ь</sup> (≤ 45 yrs)	Current n = 3 ≤ 45 yrs old,	All have <<0.1 mg/L.	>>100	>>100	>>100	>>100
	5 yr ago n = 6 ≤ 45 yrs old,	Current max for this group is ~ 0.3 mg/L so 5 yr ago <b>~ 0.6 mg/L</b> <sup>d</sup>	43	58	75	100
	10 yr ago n = 8 ≤ 45 yrs old, n = 8	10 yr ago ~ <b>1.2 mg/L</b> <sup>d</sup>	22 <sup>e</sup>	29	38	50

<sup>a</sup> MOEs are calculated against a number of serum No Observed Effect Levels (NOEL) for a range of effects observed in animal toxicity studies (Appendix B.3). The acceptable MOE is ≥25 (see text).

<sup>b</sup> Females in the cohort who are currently of reproductive age (≤ 45 yrs old), or were so 5 or 10 years ago, are assessed against animal serum NOELs for reproductive effects (low birth weight of offspring) in addition to the other toxicological endpoints for males and non-reproductive capacity females. Using a high approximation of the uppermost current serum PFOS concentration in females, serum concentrations 5 or 10 years ago have been estimated using a serum half-life for PFOS of 5.4 years (EFSA 2008).

<sup>c</sup> To preserve anonymity the serum concentrations in this table are not actual measured values. They are higher than those actually measured.



- <sup>d</sup> These concentrations are estimated from the appropriate approximate maximum concentration assuming a serum PFOS half-life of 5.4 years. Because the exposure patterns are not known they do not relate to a particular individual but rather are hypothetical concentrations, but nonetheless grounded in current PFOS serum measurements.
- <sup>e</sup> This MOE is marginally lower than the critical MOE for low risk of 25. Given the approximations in the MOE calculations this does not represent an unacceptable risk of low birth weight ten years ago.
- <sup>t</sup> Because the reproductive effect of concern is low birth weight, mediated by maternal serum PFOS concentrations it is not applicable (N/A) to calculate MOEs for males or females of non-reproductive capacity using an animal serum NOEL for this endpoint.

### 5. Conclusions

Serum PFC measurements were undertaken by a commercial laboratory that included appropriate blanks, PFC spikes and duplicate analysis of samples chosen randomly. While internal standard recoveries for some samples were lower than the range regarded as ideal by the laboratory, the data are still considered reliable for assessment of potential risk.

Twelve of the 22 participants in the 'fish consumption' health surveillance program indicated that they had eaten fish or eel from the Lake in the past. For no person in the surveillance program were there changes in blood clinical chemistry parameters that could be attributed to PFOS. While recognising the very small sample size limits confidence in the data interpretation, regression analysis of *a priori* individual blood parameters with serum PFOS levels for either the entire cohort or just those that ate fish indicated no associations. Nevertheless there were a number of individuals in both the fish eating and non-fish eating groups that had blood parameter measurements outside the population reference range. All these were attributed to life style factors (e.g. alcohol consumption), body mass index, existing disease, and/or medication (including non-compliance). Where appropriate the medical officer referred people to their own medical practitioner for follow up.

Of the 10 PFCs looked for in human serum (chosen for their presence in Lake water or fish) only two were present at measurable concentrations in the serum of program participants. These were PFOS and PFOA. All PFOA measurements were approximately an order of magnitude less than the expected background concentrations for this compound. This indicates fish consumption has not contributed to human PFOA serum concentrations; not unexpected since redfin did not have measurable concentrations of PFOA in their flesh. PFOA was therefore not considered further in the risk assessment.

The potential health impact of measured serum PFOS concentrations has been assessed using two comparator serum concentrations. The first being a background concentration where it is expected the majority of the population will be below. The second is a serum concentration at which no effects in humans are expected, termed the serum NOEL.



Four persons had serum PFOS concentrations above that identified as the higher end of the normal range expected from background (i.e. resulting from day to day living). All were below the serum NOEL indicating low risk for adverse health effects. Available information on fishing frequency suggests exposure patterns were unlikely to have been materially different in the past and so serum PFOS concentrations were also unlikely to be markedly different from those measured in the surveillance program.

The Margin of Exposure (MOE) estimations calculated using current measured serum PFOS concentrations and serum NOELs for sensitive toxicological endpoints identified from animal toxicity experiments also indicated very low risk for adverse health effects.

When current serum concentrations were extrapolated back to theoretical levels that may have existed 5 or 10 years previously, and assuming no further fish consumption, both comparison with the human serum NOEL and the calculated MOEs indicate adverse health effects were unlikely to have arisen due to the hypothetical serum PFOS concentrations.

Overall, it is concluded existing serum PFOS concentrations or past theoretical concentrations are unlikely to give rise to adverse health effects.

# 6. Uncertainty analysis

As with all human health risk assessments (HHRAs) there are uncertainties in this assessment that potentially affect the conclusions. They have been addressed either by conservative assumptions or inclusion of hypothetical exposure scenarios.

#### Exposure:

The major uncertainty in HHRAs usually resides with exposure estimations. In this HHRA much of the exposure ambiguity associated with determination of external dose is negated by use of serum PFOS concentrations as a measure of internal dose. The residual exposure uncertainty lies with the analytical measurement of PFCs in human serum. Since appropriate spiked matrix samples, blanks and duplicates were included in the analytical regime which all returned consistent, expected results uncertainty in the determination of current PFOS serum concentrations is considered to be minimal.

Current measurement of PFOS serum concentrations provides information allowing assessment of health impacts at the time of measurement and, because of the long serum half in humans, also in the recent past. However there is uncertainty regarding past PFOS serum concentrations. This has been



addressed by assuming no PFOS contaminated fish consumption for the past 5 or 10 years and extrapolating the maximum measured current PFOS serum concentration back to those times. While this theoretical past serum concentration may be under or over estimated it is our opinion it is more likely an overestimate of past serum levels.

#### Toxicological reference values and risk characterisation:

HHRAs often use toxicological reference values (e.g. TDI or RfD) established by competent authorities for judging the impact of the calculated external exposures. Since the exposure metric is serum concentration rather than dose such guidance values are inappropriate. The risk characterisation has been carefully undertaken using:

- Two comparator serum concentrations developed for this assessment, and
- with MOE calculations.

The latter not being reliant on assumptions made in the development of the comparator serum concentrations.

- The first PFOS serum comparator is a maximum PFOS serum concentration that might arise due to PFOS exposure in the general human environment (i.e. background exposures). More than 40 peer reviewed papers reporting blood/serum PFOS concentrations from around the world were included in this assessment. Care was taken not to include occupational exposures, populations near PFC manufacturing/handling facilities, or communities affected by PFC ground water contamination. We have a high degree of confidence that the majority (~95%) of people should have background PFOS serum concentrations <0.1 mg/L.</li>
- The second PFOS serum comparator was the establishment of a serum concentration that would be expected to be without adverse health effects. To reduce the uncertainty in setting the human serum NOEL, three independent methods were employed (described in Appendix B). These were:
  - o a NOEL from occupational epidemiology studies,
  - application of standard techniques for setting toxicological reference values using sensitive effects observed in monkeys and rats, and
  - using human toxicokinetic data to convert the TDI set by the European Food Safety Authority to an equivalent steady state serum concentration.

We have a high degree of confidence in the robustness of the human NOEL (2 mg/L) used in this assessment.



#### Cohort sample size:

The number of people entering the PFOS health surveillance program was small (22 individuals), with just over half of these reporting they had eaten fish from Lake Fiskville. Consequently there is uncertainty in making group deductions about the relationship between serum PFOS concentrations and any particular health parameter measured in the program. Nevertheless correlations have been constructed that show no association between the health parameters and serum PFOS for the group. Due to the small sample size these need to be interpreted with caution.

Possible risk to an individual was done according to standard medical practice using the expertise of the medical officer and the consultant toxicologist. While this advice is subject to the usual uncertainties associated with medical diagnosis it has been professionally provided and we are confident it has been appropriate for the circumstance of the individual(s).



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# Appendix A: Glossary

Alkaline Phosphatase		
Alanine Aminotransferase		
Aspartate Aminotransferase		
Lower bound Benchmark Dose		
Body Mass Index		
Country Fire Authority		
European Food Safety Authority		
Gamma Glutamyl Transferase		
High Density Lipoprotein		
High Performance Liquid Chromatography Tandem Mass Spectrometry		
Health Risk Assessment		
Low Density Lipoprotein		
Margin Of Exposure		
National Measurement Institute		
No Observed Effect Level		
Perfluorinated Compound		
Relative Percentage Difference		
Steady State		
Triiodothyronine		
Thyroxine		
Tolerable Daily Intake		
Triglycerides		
Thyroid Stimulating Hormone		



#### **PFC Abbreviations**

Abbreviation	PFC
PFBA	Perfluorobutanoic acid
PFPeA	Perfluoro-n-pentanoic acid
PFBS	Perfluorobutane sulphonic acid
PFHxS	Perfluorohexanesulphonic acid
PFOS	Perfluorooctane sulphonic acid
PFDS	Perfluorodecane sulphonic acid
PFHxA	Perfluorohexanoic acid
PFHpA	Perfluoroheptanoic acid
PFOA	Perfluorooctanoic acid
PFNA	Perfluorononanoic acid
PFDA	Perfluorodecanoic acid
PFUdA	Perfluoroundecanoic acid
PFDoA	Perfluorododecanoic acid
PFTrDA	Perfluorotridecanoic acid
PFTeDA	Perfluorotetradecanoic acid
PFOSA	Perfluorooctane sulphonamide
NEtFOSA	N-ethyl-perfluorooctane sulphonamide
NEtFOSAA	N-ethyl-perfluorooctanes ulphonamidoacetic acid
NMeFOSA	N-methyl-perfluorooctane sulphonamide
NMeFOSAA	N-methyl-perfluorooctane sulphonamidoacetic acid
NEtFOSE	N-ethyl-perfluorooctane sulphonamidoethanol
NMeFOSE	N-methyl-perfluorooctane sulphonamidoethanol
4:2 FtS	1H,1H,2H,2H-perfluorohexane sulphonic acid
6:2 FtS	1H,1H,2H,2H-perfluorooctane sulphonic acid
8:2 FtS	1H,1H,2H,2H-perfluorodecane sulphonic acid



# Appendix B: Determination of serum PFC concentrations for risk characterisation.

#### B.1 Background human PFC serum concentrations

A wide range of PFCs are found in consumer and industrial products. They are used to treat leathers and paper so they repel water and grease (e.g. grease proof paper, pizza boxes, popcorn, hamburger and chip containers, fruit boxes, etc), water proof shoes and textiles, make breathable water repelling fabrics (e.g. Cortex), apply stain resistance to carpets and furniture (e.g. Scotchguard). They are in a range of cosmetics and personal care products, and in some surface coatings. PFOS has been used in certain firefighting foams. In the environment or in the body many of the PFCs in these products breakdown or are metabolised to PFOS or PFOA. These are both very stable and are persistent in the environment and long lived in the body.

To identify background serum concentrations of PFOS and PFOA a literature search was undertaken for data in populations around the world that were not occupationally exposed, did not live near PFC manufacturing sources, and were not influenced by local contamination of groundwater or soil.

Figures B.1 and B.2 summarise background PFOS and PFOA concentrations in human serum from a large number of studies, the information is consolidated in Table B.1. Individual data for the studies was not available to statistically construct a 'normal' background reference range. However inspection of Figure B.1 compellingly indicates the majority of the general population would be expected to have a PFOS serum concentration less than 0.1 mg/L. This agrees with 3M Company (2003) and Olsen et al (2003b) who statistically calculated 95% of the general population have PFOS serum concentration less than 0.1 mg/L.

Similarly, Figure B.2 indicates the majority of persons would be expected to have less than 0.05 mg/L PFOA in their serum as a result of normal day-to-day living.

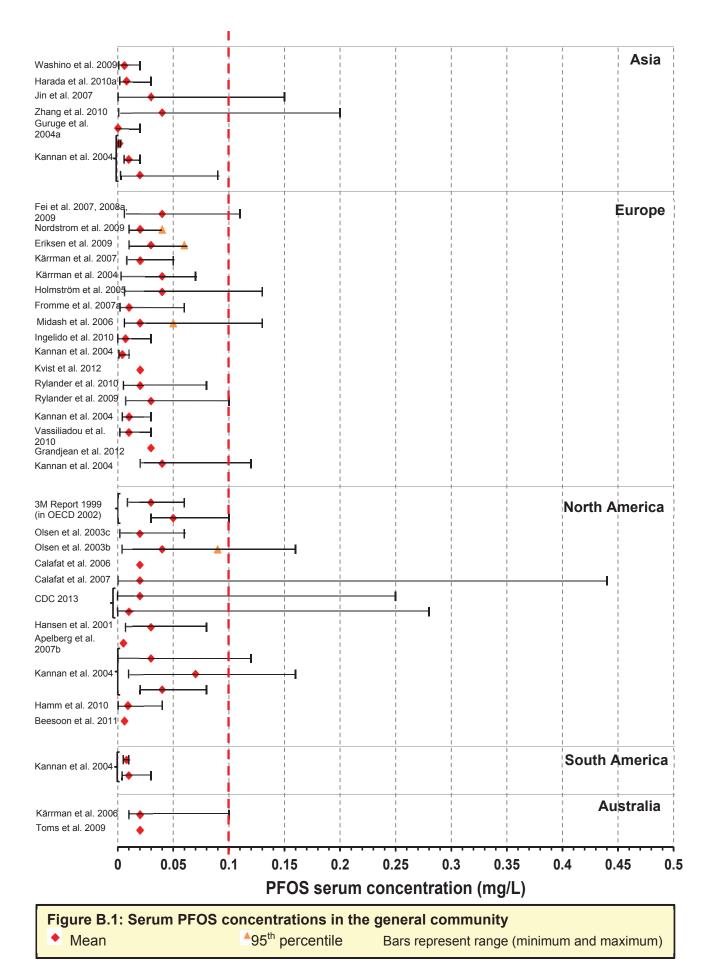
_		Population means	Range for individuals	Majority of individuals <sup>b</sup>
	PFOS	0.005 - 0.05	0 - 0.3	<0.1
	PFOA	0.0002 - 0.06	0 – 0.09	<0.05

Table B.1: Summary of background PFC serum concentrations <sup>a</sup>

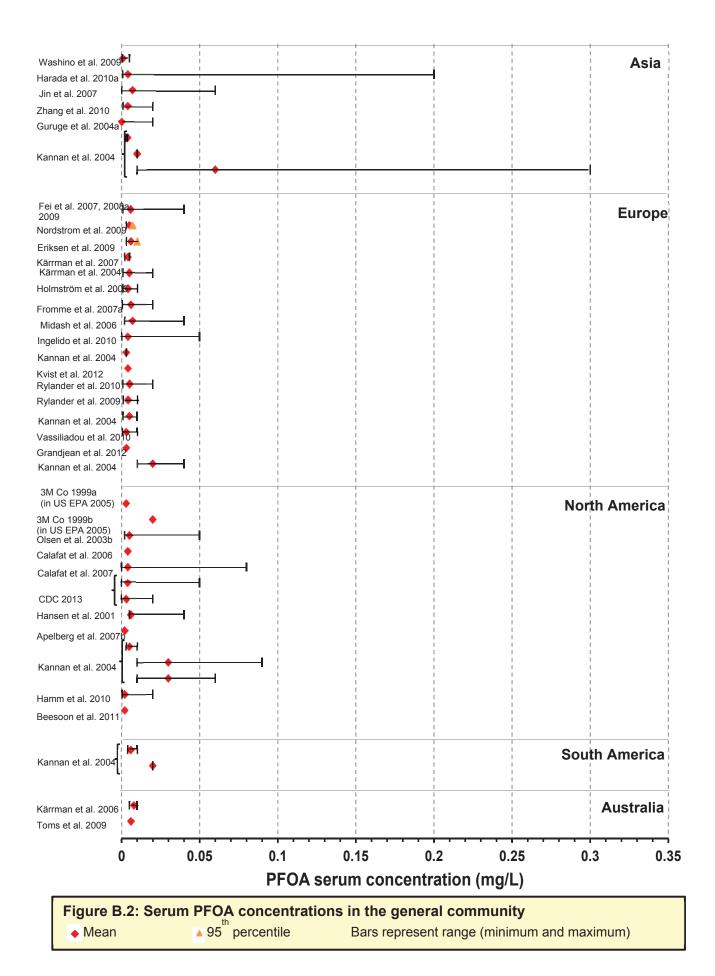
<sup>a</sup> Information in the table is a summary of that visually presented in Figures B.1 and B.2.

<sup>&</sup>lt;sup>b</sup> It is expected from Figures B.1 and B.2 that the majority of individuals would have serum concentrations less than these values. These concentrations are therefore used as the upper end of 'background' serum concentrations for PFOS and PFOA.











#### **B.2 Human no effect serum concentration (PFOS)**

#### **B2.1 Occupational epidemiology studies**

Workers who handle or make PFCs have much higher serum PFOS concentrations than the general population; they also tend to have higher PFOA levels. In such workers PFOS concentrations may be as high as 12 - 13 mg/L, but the majority are <6 mg/L (Olsen et al. 1999a, 2003a, 2003f) (Figure AB.1). These levels of exposure are primarily confined to three manufacturing plants in the US and Belgium. Over more than a decade several occupational epidemiology studies have been undertaken on this cohort. The studies have primarily focussed on the *a posteriori* toxicological knowledge gained from monkey and rodent studies; the most sensitive effects being decreased cholesterol and circulating thyroid hormones which are totally reversible when serum concentrations decrease (Section B2.2). At higher serum concentrations (BMDL<sub>10</sub> 60 mg/L) in 2 year rat experiments liver adenomas are observed (PFOS is not genotoxic) and in developmental and multi-generation studies PFOS causes decreased pup weight and neonatal survival (BMDL<sub>5</sub> pup weight 31 mg/L, BMDL<sub>5</sub> perinatal mortality 83 mg/L) (Butenhoff et al. 2012b, Thomford 2002, 3M Company 2003). Potential effects investigated in the epidemiology studies included thyroid and lipid metabolism disorders, mortality, cancer incidence, liver, cardiovascular and gastrointestinal diseases, and pregnancy outcomes (Alexander et al. 2003; Alexander and Olsen 2007; Olsen 1999a, 2003a, 2004c; Grice et al. 2007).

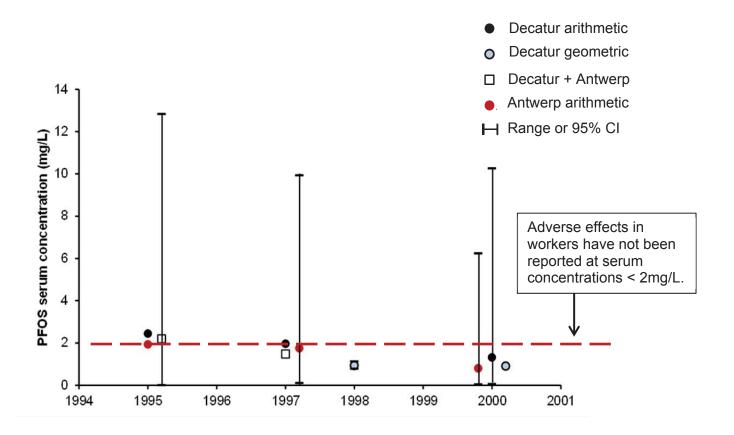
There were no changes in haematological, lipid, hepatic, thyroid, or urinary parameters consistent with the known toxicological effects of PFOS in cross-sectional or longitudinal analyses of workers who had PFOS serum levels < 2 mg/L. At concentrations higher than 6 mg/L slight positive associations with altered cholesterol, triglyceride and high density lipoprotein have been reported but these are inconsistent with the known biochemistry of PFOS and the effects observed in animals, including monkeys. Consequently these associations should be interpreted with care, they may be random findings, or due to a different variable other than PFOS.

Although an initial study reported an association between PFOS and urinary bladder cancer (Alexander et al. 2003) this was based on just three cases, when the study was expanded with more accurate exposure measures and confounders controlled, no association between PFOS and bladder cancer was apparent <sup>17</sup> (Alexander and Olsen 2007). No changes in other endpoints investigated have been reported.

<sup>&</sup>lt;sup>17</sup> A chemical or biological basis for induction of bladder cancer by PFOS is obscure. It does not appear to have the properties of known bladder carcinogens and has not shown any bladder effects in toxicology studies. It is neither genotoxic nor insoluble in urine at room temperature.



The overall size of the occupational cohort is greater than 3,500 however it was smaller subgroups that were investigated in the epidemiology studies, with 100 – 300 persons in any particular exposure strata. As with many cross sectional epidemiology investigations, the individual studies are open to criticism. Some of these are study design, lack of control for certain confounders, participation being



# Figure AB.1: Serum PFOS concentration in workers at two manufacturing plants (Decatur, Alabama and Antwerp, Belgium).

Production of perfluorinated sulphonated compounds began in Decatur in 1961 and Antwerp in 1976. PFOS measurements started in the early 1990's when specific analytical techniques became available. The total number of persons who have been exposed and studied in these factories is greater than 3,500. Adverse effects in workers have not been reported at serum concentrations < 2mg/L, this is taken to be a No Observed Effect Level (NOEL) for adults.

The data in the figure has been compiled from the following publications which have studied various sectors of the worker population. The PFOS serum concentrations are the geometric or arithmetic means of the study population, with either the 95% confidence limit (CL) of the mean or the range of serum concentrations when reported.

Gilliland & Mandel (1996), Olsen et al. (1999a, 2000, 2003a, 2003c), Alexander et al. 2003, Alexander & Olsen 2007, Grice et al. 2007, Olsen and Zobel 2007.



voluntary rather than random recruitment, uncertainty in assignment to an exposure group based on job description and years of service with serum PFOS bands allocated by measurement of workers with similar job task profiles. Nevertheless the cohort represents the most highly exposed humans in the world. Overall PFOS serum levels in individual workers are up to 4 orders magnitude greater than the population means of the general public, the lowest occupational sub-cohort is approximately 1 - 2 orders greater. Thus, if humans are susceptible to the adverse effects observed to be induced by high serum PFOS in animals, they would be expected to be detected in this occupational cohort. As per the philosophy of administering high doses of chemical to small groups of animals to identify hazards, the high serum concentrations in workers counters the less than ideal number of subjects in the occupational epidemiology studies.

#### Conclusion:

From the occupational epidemiology information it is concluded that a serum PFOS concentration of 2 mg/L represents a level at which no effects have been observed in adults. The actual no effect level may be higher than this but there are insufficient numbers of persons with concentrations around this level for implications to be drawn.



#### B2.2 Animal serum PFOS no observed effect level (NOEL)

The procedures employed in this section of Appendix B for deriving human serum NOELs are part of standard risk assessment methodologies for setting toxicity reference guideline values used by WHO, the EC and recommended in Australia (WHO 2004, 2010; enHealth 2012).

In general, observations from toxicological studies with PFOS include reductions in body-weight and weight gain, increases in liver weight (characterised by increased centrilobular hepatocellular hypertrophy), mild-to-moderate peroxisome proliferation in rats, increased incidence of hepatocellular adenoma<sup>18</sup> in rats, and hypo-cholesterolemia. Effects appear to be related to a threshold body burden and often are associated with a steep dose–response.

The mechanisms of PFOS induced toxicity are not fully understood but may include effects on fatty acid transport and metabolism, membrane function, and/or mitochondrial bioenergetics. Cumulative toxicity, occurring at high serum concentrations, is expressed as metabolic wasting in adult experimental animals, decreased neonatal survival and weight gain in offspring. Sensitive effects are observed in monkey studies which provide serum concentrations for changes in blood biomarkers for potential effects on lipid metabolism and energy production. Developmental and 2-generation reproduction studies in rats deliver benchmark doses (BMD and BMDL) for conversion to serum concentrations for the sensitive effects of neonatal survival and weight gain.

#### Monkey:

The pivotal study for PFOS is a 28 week oral (0, 0.03, 0.15 & 0.75 mg/kg/d via capsule) study in cynomolgus monkeys (Seacat et al. 2002). A range of blood parameters and serum PFOS concentrations were monitored throughout the study and during a one year recovery period. At serum concentrations not causing overt toxicity (approximately 60 – 100 mg/L) the primary findings are changes in biochemical parameters associated with lipid metabolism. The animals show increased liver weight and decreases in body weight, together with decreased cholesterol and high density lipoprotein (HDL), decreased triglycerides and thyroid hormone (T3) (without marked compensatory increase in TSH). These changes have been shown to be readily and completely reversible within 30 weeks of treatment cessation as serum concentrations decrease.

For each of the doses and sampling times serum PFOS was measured. Dose response modelling gave a BMDL serum concentration of 35 mg/L for no or minimal impact on sensitive effects in the liver

<sup>&</sup>lt;sup>18</sup> Hepatocellular hypertrophy and liver adenomas induced in rats by PFOS are mediated through the nongenotoxic mechanisms of PPARα and CAR activation and are considered irrelevant modes of action for human risk assessment (Klaunig et al. 2003, Elcombe et al. 2012a, 2013).



(decreased cholesterol) (MDH 2008). The fact that this serum concentration is the low 95th confidence limit estimate means it is conservative and is taken as the no observed effect level (NOEL).

Supporting use of the serum concentration in monkeys as a surrogate for the human internal dose to the target tissue is the liver:serum ratios being similar in monkeys and humans. These ratios are 1.4 and 1.3 respectively (Olsen et al. 2003c, Seacat et al. 2002). There is however undefined uncertainty with regard to the responsiveness of monkey and human liver to the same internal dose (serum concentration) of PFOS. This is despite the majority of hepatic effects being mediated via the PPARα receptor, and humans and monkeys being approximately equally sensitive to its activation by peroxisome proliferators (Cariello et al. 2005, FDA 2005, Kane et al. 2006). To account for human liver possibly being more sensitive than that of monkeys (i.e. for interspecies toxicodynamic differences), the standard default uncertainty factor of 2.5x has been applied to the NOEL of 35 mg/L. In addition the usual default for response variability (toxicodynamic) between humans (3.2x) has been added.

The total uncertainty factor applied to extrapolate the monkey NOEL serum concentration is therefore 8x and the equivalent human serum NOEL derived from the monkey BMDL<sub>10</sub> of 35 mg/L is 4.4 mg/L.

#### Rat:

In rat toxicity studies the most sensitive effect is decreased pup weight gain observed in two generation reproduction experiments (Lau et al. 2012; 3M Company 2003; Luebker 2005a, 2005b; Thomford 2002).

BMDL<sub>5</sub> on pup weight gain is 26 mg/L – 31 mg/L (3M Company 2003) and pup survival 83 mg/L (Lau et al. 2007). The 26 mg/L is derived from data from the two-generation reproduction/developmental study (pup weight gain through lactation) (Luebker et al. 2005a) and the serum PFOS concentration measurements made in a separate toxicokinetic study during pregnancy at the same dose levels (Luebker et al. 2005b). The 31 mg/L is for in reduced pup weight gain during lactation using the mean of gestation day 21 and pre-gestational serum levels in dams (Luebker et al. 2005b).

Lau et al. (2007) is a review of the toxicology of perflouroalkyl acids, primarily PFOS and PFOA. In this review 'no effect' doses [i.e. the BMD and BMDL as reported by Luebker et al. (2005b) and Lau et al. (2003)] were translated into equivalent no effect serum concentrations using linear relationships between dose (mg/kg) and serum concentration (mg/L). Thus Lau et al. (2007) converted the BMD<sub>5</sub> and BMDL<sub>5</sub> of:

1.06 and 0.89 mg/kg/d from Luebker et al. (2005b) into serum concentrations of 67 and 59 mg/L for postnatal survival at lactation day (LD) 5, and



 1.07 and 0.58 mg/kg/d from Lau et al. (2003) for postnatal survival to day 8 were translated into serum concentrations<sup>19</sup> of 25 and 16 mg/L.

Unfortunately Lau et al. (2007) did not fully consider the serum data reported in these studies and the animal serum <u>BMDs derived by this author are not the most appropriate</u> for defining serum concentrations for deriving human equivalent serum NOELs for PFOS. It is also noted that Lau et al. (2007) only considered neonatal survival and not the more sensitive endpoint of decreased birth weight and weight gain. The studies and derivation of suitable human serum NOELS are described below.

#### The Luebker et al. (2005b) study:

Luebker et al. (2005b) dosed rats at 0.4, 0.8, 1.0, 1.2, 1.6, and 2.0 mg PFOS/kg/d for 42d prior to mating and through to gestation day 20, or LD 4 depending on the study phase. The BMDL<sub>5</sub> based on decreased gestation length, birth weight, pup weight at LD 5, pup weight gain through LD 5, and pup survival through LD 5 were relatively tight at 0.31, 0.39, 0.27, 0.28, and 0.89 mg/kg/day, respectively. There is a steep dose–response relationship that begins to appear between 0.8 and 1.2 mg/kg before becoming statistically significant at 1.6 mg/kg. According to Luebker et al. (2005b) this observation, together with other reports in the literature (Lau et al. 2003; Luebker et al. 2005a), suggests a critical body burden in dams is required to influence viability in neonates. In the Luebker et al. (2005b) study maternal serum concentrations on gestation days 1, 7 and 15 were relatively constant indicating the animals were at steady state after 42 days of dosing prior to mating. However there was a 40 - 60% decrease in maternal serum concentrations at gestation day 21. The decline may have been the result of increased volume expansion and other physiological changes during the last trimester, including changes in serum protein content. Patently, post gestational serum concentrations do not reflect the potential extent of foetal exposure during pregnancy.

Lau et al. (2007) converted the  $BMD_5$  and  $BMDL_5$  of 1.06 and 0.89 mg/kg/d as determined by Luebker et al. (2005b) for pup survival at LD 5 into equivalent serum concentrations using the linear association between dose and maternal serum concentration at gestation day 21. As noted above there is a substantial decrease in serum concentrations between the steady state concentrations up to gestation day 15 and concentrations measured on gestation day 21. It would appear that the dose-

<sup>&</sup>lt;sup>19</sup> Although both Luebker et al. (2005b) and Lau et al. (2003) have modelled similar BMD's from their data (1.06 and 1.07 mg/kg/d respectively), Lau et al. (2007) derived corresponding serum concentrations that are very different from each other, i.e. 59 and 16 mg/L respectively.



serum concentration relationship at steady state is a better indication of foetal exposure. This relationship yields a regression equation of y = 85.656x + 6.8086 (r<sup>2</sup> = 0.9949)<sup>20</sup>; and at a BMDL<sub>5</sub> of:

- 0.89 mg/kg/d for pup survival at LD 5, the maternal steady state serum concentration is 83.1 mg/L.
- 0.28 mg/kg/d for pup weight gain through to LD 5, the maternal steady state serum concentration is 30.9 mg/L. Thus pup weight gain is the more sensitive indicator.

#### The Lau et al. (2003) study:

Lau et al. (2003) treated rats with 1, 2, 3, 5 and 10 mg PFOS/kg/d on gestation days 2 to 21. In this study there was decreased pup survival and in survivors decreased weight gain. While serum and liver concentrations of pups after birth were measured, serum PFOS in the dams was not. There was a decrease in pup survival at and above 2 mg/kg. The BMD<sub>5</sub> and BMDL<sub>5</sub> were 1.07 and 0.58 mg/kg/d for postnatal survival to day 8. Since Lau et al. (2003) did not report maternal serum PFOS concentrations, Lau et al. (2007) used the maternal serum concentrations at gestation day 21 from Thibodeaux et al. (2003a, b) to convert the Lau et al. (2003) BMDs to equivalent serum maternal concentrations. Rats in Lau et al. (2003) and Thibodeaux et al. (2003a, b) were given the same PFOS dose regime.

Thibodeaux et al. (2003a, b) is a developmental investigation in which skeletal variations occurred in the presence of decreased maternal weight gain. The graphical data in Thibodeaux et al. (2003a) indicates the maternal PFOS serum concentrations are steeply rising for most doses at gestation days 7 and 14 when serum was drawn. This indicates serum PFOS concentrations were not at steady state. Indeed the serum concentrations were markedly less than reported in Luebker et al. (2005b) despite the fact the doses were approximately 5 times higher. Nevertheless, as observed in Luebker et al. (2005b) maternal serum concentrations were somewhat lower at gestation day 21 than at day 15, particularly for the top three doses. Although Lau et al. (2003) and Thibodeaux et al. (2003a, b) are reporting different aspects of the same study, because the dose regime was short and there were marked changes in maternal serum PFOS concentrations between days 14 and 21 it is very difficult to determine the serum concentrations that may be associated with the effects observed in Lau et al. (2003).

<sup>&</sup>lt;sup>20</sup> This correlation is stronger than the R<sup>2</sup> of 0.862 reported by Lau et al. (2005b) using the gestation day 21 serum data of Luebker et al. (2005b). The data for the correlation is provided in table below:

Premating dose (mg/kg/d)	15-d serum concentration (mg/L)	
0.1	8.81	
0.4	41.4	
1.6	156	
3.2	275	



#### Conclusions:

For pup survival and weight gain the data of Luebker et al. (2005b) is preferred over Lau et al. (2003) because of the longer dose time (42d vs 19d), lower doses employed, serum concentrations are reported as values that can be used in independent analysis, and the serum and effects data are consistent with the two generation reproduction study (Luebker et al. 2005a). Thus the favoured serum NOELs (as BMDL<sub>5</sub>) for rat neonatal survival and decreased neonatal weight gain are 83 and 31 mg/L respectively.

The appropriate BMDLs for deriving a human serum PFOS no observed effect level from 2-generation and developmental studies are:

- 26 31 mg/L for reduced pup weight gain.
- 83 100 mg/L for reduced neonatal survival.

Applying the same uncertainty factors (i.e. 2.5x for interspecies toxicodynamic differences and 3.2x for toxicodynamic variability between humans) to the most sensitive reduced pup weight gain BMDLs of 26 - 31 mg/L as for the monkey serum BMDL gives an equivalent NOEL for humans of 3.25 - 3.9 mg/L.

#### In summary:

- The equivalent human serum NOEL from the monkey investigation of Seacat et al. (2002) is 4.4.mg/L.
- The human serum LOEL from rat reproduction and developmental studies in which the most sensitive effect was decreased weight gain of neonates is 3.25 3.9 mg/L.



#### B2.3 Conversion of TDI to serum concentration

Four TDIs for PFOS have been established by international authorities:

- ο The UK Committee on Toxicity (COT 2006): 0.3 µg/kg/d.
- ο The European Food Standards Authority (EFSA 2008): 0.15 μg/kg/d.
- ο The Minnesota Department of Health (MDH 2008): 0.08 μg/kg/d.
- o US Environmental Protection Authority (US EPA 2009): 0.08 μg/kg/d.

They have all based their deliberations on the 26 week oral monkey study by Seacat et al. (2002) described in Appendix B2.2 but have arrived at different TDI values as a result of different methodologies, different uncertainty factors and/or different science policy.

Generally Australian authorities have a preference for World Health Organisation and European deliberations because these tend to match science policy and risk assessment methods used in Australia more closely than those in North America. Thus Food Standards Australia New Zealand (FSANZ 2011) refer to the ESFA TDI when they reported the results of a survey of chemical migration, including PFCs, from food contact packaging materials into Australian food. In this assessment the TDI of 0.15 µg/kg/d from EFSA (2008) has been adopted. Furthermore it is noted that the average of all the above TDIs is 0.15 µg/kg/d.

The TDI is an estimate of the amount of a contaminant or natural toxicant, expressed on a body weight basis that can be ingested daily over a lifetime without appreciable risk. Thus the long term serum concentrations associated with this dose are steady state concentrations.

The standard pharmacokinetic equation (Birkett 1999) used in medicine to calculate steady state blood concentrations is:

 $C_{SS} = (DR \times t_{\frac{1}{2}}) \div (0.693 \times Vd)...$ Equation B1 Where:

 $C_{SS}$  = Steady state serum concentration. DR = Dose Rate. In this case 0.15 µg/kg/d (0.00015 mg/kg/d)  $t_{\frac{1}{2}}$  = Serum half-life (1971days, (EFSA 2008, Olsen et al. 2007, DFG 2010). Vd = Apparent volume of distribution is extracellular water (0.2 L/kg bw, [Olsen et al. 2007, DFG 2010, Chang et al. 2012]).

Substituting values into Equation B1  $C_{SS} = (0.00015 \text{ mg/kg/d x 1971d}) \div (0.693 \text{ x } 0.2 \text{ L/kg}) = 2.13 \text{ mg/L}$ 

Thus the steady state serum concentration of PFOS associated with a TDI of 0.15  $\mu$ g/kg/d is 2 mg/L (rounded).



#### **B.3 Studies supporting margin of exposure calculations**

The serum NOELs used in the calculation of MOEs in Section 4.2 are:

- 26 mg/L in maternal serum for decreased weight gain in offspring in two generation and/or developmental rodent studies (Luebker 2005a, 2005b).
- 35 mg/L from monkey experiments for the same blood parameters as evaluated in the health surveillance program (Seacat et al. 2002).
- 45 mg/L for liver toxicity in a two year bioassay (Thomford 2002, 3M Company 2003, Butenhoff et al. 2012b).
- 60 mg/L for production of liver adenomas in a two year bioassay (Thomford 2002, 3M Company 2003, Butenhoff et al. 2012b).

The Luebker (2005a, 2005b) and Seacat et al. (2002) studies, with the identification of the serum NOELs, are described in Appendix B2.2.

The two year bioassay supporting serum NOELs for chronic liver toxicity and induction of liver adenomas is described below. The study was sponsored by 3M, conducted at Covance Laboratories Ltd under good laboratory (GLP) standards, with the report authored by Thomford (2002). The laboratory report is not publically available but was submitted to EFSA as part of a data package for the PFOS/PFOA review that was being undertaken. EFSA (2008) describes the essential features of the study. The terminal pathology obtained in the study was reported at a toxicology science conference (Seacat et al. 2002b). The 3M Company (2003), in consultation with independent toxicologists, used data from the study to model serum concentration and effects, with the objective of determining serum PFOS concentrations equivalent in status to the lower confidence limit of a benchmark dose for 5% response for liver toxicity (i.e. a serum BMDL<sub>5</sub>) or 10% incidence of liver adenomas (i.e. a serum BMDL<sub>10</sub>). Sometime after this work was completed, Butenhoff et al. (2012b), with Thomford as co-author, published the study in a peer reviewed journal. The description of the study below is primarily derived from Butenhoff et al. (2012b).

The two-year dietary toxicity and cancer bioassay was conducted with potassium PFOS in male and female Sprague Dawley rats. Dietary concentrations were 0, 0.5, 2, 5, and 20 µg/g (ppm). Included in the study was a recovery group that was fed 20 ppm for the first 52 weeks, after which they were fed control diet through to study termination. Scheduled interim sacrifices occurred on Weeks 4, 14, and 53, with terminal sacrifice between Weeks 103 and 106. The PFOS dietary treatment appeared to be well-tolerated, however there were sporadic decreases in body weight during the treatment period that were not clearly dose related. Interestingly male rats had a statistically significant decreased mortality with significantly increased survival to term at the two highest treatment levels. Decreased



serum total cholesterol, especially in males, and increased serum urea nitrogen were consistent clinical chemistry observations that were clearly related to treatment. The reduced serum total cholesterol, seen at earlier time points, was no longer apparent after 104 weeks of treatment. This may have been due to lower liver PFOS concentrations compared to earlier time points.

The principal non-neoplastic effect included liver hypertrophy, with proliferation of endoplasmic reticulum. The effect was dose related from 5 ppm upward. This was also evident in the 20 ppm recovery group, probably as a result of sufficient PFOS being retained in the liver to stimulate PPAR and CAR receptors. In males there were also increased serum enzymes indicative of liver toxicity. Statistically significant increases in benign hepatocellular adenoma<sup>21</sup> were observed in surviving males and females of the 20 ppm treatment group. There were no treatment-related findings for thyroid tissue and no evidence of kidney or bladder effects.

Butenhoff et al. (2012b) determined dietary doses corresponding to the estimated  $BMDL_{10}$  for liver adenomas was 7.9 ppm for male rats and 8.0 ppm for female rats. Aging of animals, characterised by progressive nephritis, resulted in high variability in PFOS serum and liver concentrations of PFOS beyond week 53, PFOS concentrations were somewhat less at week 105. At week 53 serum concentration data was only obtained for the controls and high dose (20 ppm) group.  $BMDL_{10}$  values expressed as serum PFOS concentration after 14 weeks of dosing were 62 µg/mL and 92 µg/mL respectively for male and female.

Butenhoff et al. (2012b) did not determine serum BMDL for liver toxicity, however 3M Company (2003) report a serum  $BMDL_5$  of 44 mg/L in male rats for non-neoplastic liver effects, and  $BMDL_{10}$  of 62 mg/L for liver tumours. These values have been used in calculation of MOEs for these endpoints.

<sup>&</sup>lt;sup>21</sup> Hepatocellular hypertrophy and liver adenomas induced in rats by PFOS are mediated through the nongenotoxic mechanisms of PPARα and CAR activation and are considered irrelevant modes of action for human risk assessment (Klaunig et al. 2003, Elcombe et al. 2012a, 2013).



# Appendix C: Program surveillance tests

- 1. Full blood examination:
  - a. Haemoglobin
  - b. Packed cell volume (PCV)
  - c. Red cell count (RCC)
  - d. Mean cell volume (MCV)
  - e. Mean cell haemoglobin (MCH)
  - f. Red cell distribution width (RDW)
  - g. White cell count (WCC)
  - h. Platelets
- 2. Blood lipids:
  - a. Total cholesterol
  - b. Triglyceride
  - c. HDL cholesterol
  - d. LDL cholesterol
- 3. General biochemistry (serum):
  - a. Sodium
  - b. Potassium
  - c. Chloride
  - d. Bicarbonate
  - e. Urea
  - f. Estimated glomerular filtration rate (GFR)
  - g. Creatinine
  - h. Total bilirubin
  - i. Alanine aminotransferase (ALT)
  - j. Aspartate aminotransferase (AST)
  - k. Alkaline phosphatase (ALP)
  - I. Gamma glutamyl transferase (GGT)
  - m. Total protein
  - n. Albumin
  - o. Globulin
  - p. Urate
- 4. Thyroid function (serum):
  - a. Free thyroxine (FT4)
  - b. Thyroid stimulating hormone (TSH)



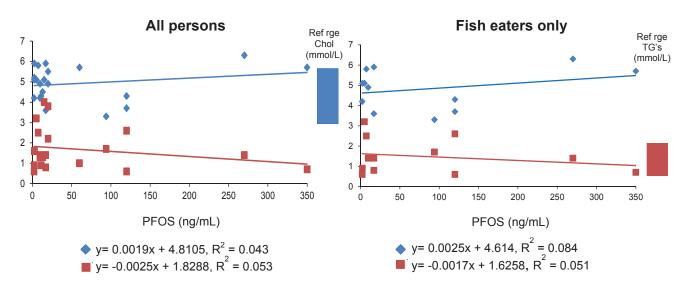
- c. Free triiodothyronine (FT3)
- 5. Other (serum):
  - a. Glucose
  - b. Creatine kinase (CK)
  - c. Prostate specific antigen (PSA)
- 6. Metals (blood):
  - a. Mercury
  - b. Cadmium
  - c. Lead
  - d. Copper
  - e. Arsenic
- 7. Physical examination:
  - a. Height
  - b. Weight
- 8. PFCs in serum (see Table C.1 for suite of PFCs)

#### Table C.1: Suite of PFCs that were analysed in serum

PFC	Abbreviation
Perfluoro-n-pentanoic acid	PFPeA
Perfluorohexanoic acid	PFHxA
Perfluoroheptanoic acid	PFHpA
Perfluorooctanoic acid	PFOA
Perfluorononanoic acid	PFNA
Perfluorodecanoic acid	PFDA
Perfluoroundecanoic acid	PFUdA
Perfluorododecanoic acid	PFDoA
Perfluorooctanesulphonic acid	PFOS
1H,1H,2H,2H-perfluorooctanesulphonic acid	6:2 FtS



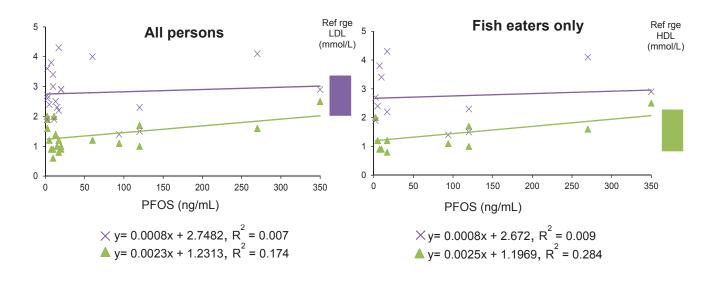
# Appendix D: Regression analysis of blood parameters with PFOS levels.



Cholesterol (♦) & Triglycerides (■)

Lipids

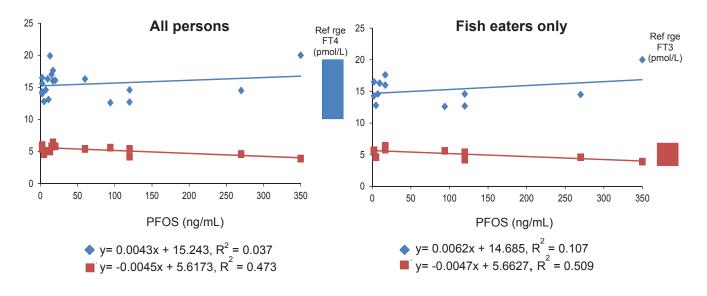
LDL ( × ) & HDL (▲ )



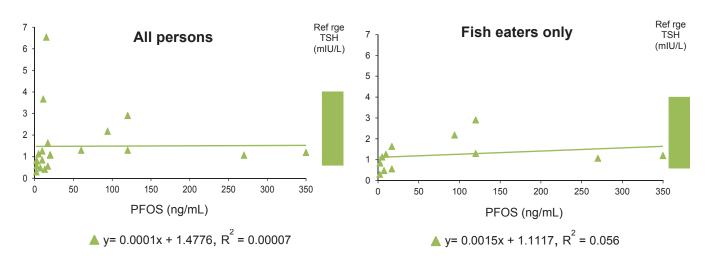


#### Thyroid function

#### Free T4 ( ◆ ) & Free T3 ( ■ )



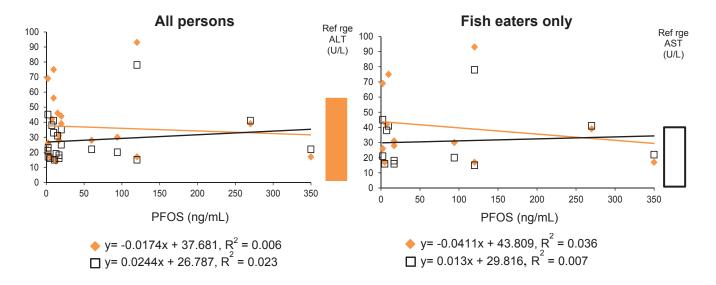
TSH ( )



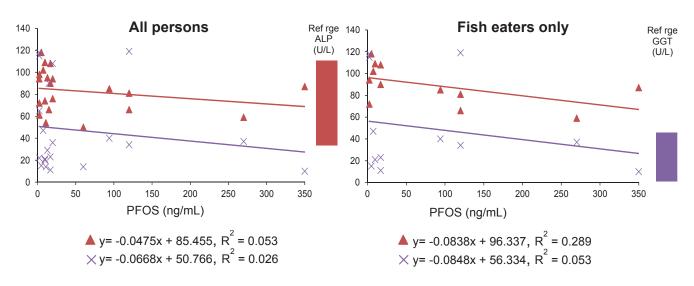


#### Liver function

### ALT ( 🔶 ) & AST ( 🗆 )



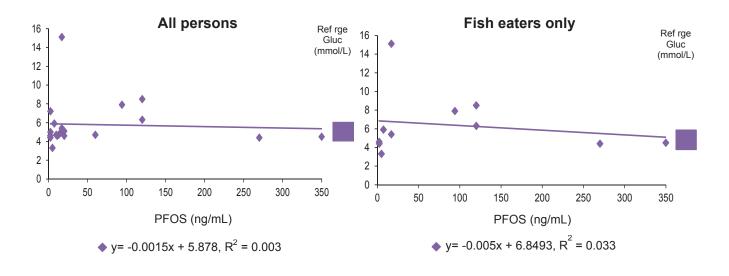
ALP (▲) & GGT (×)



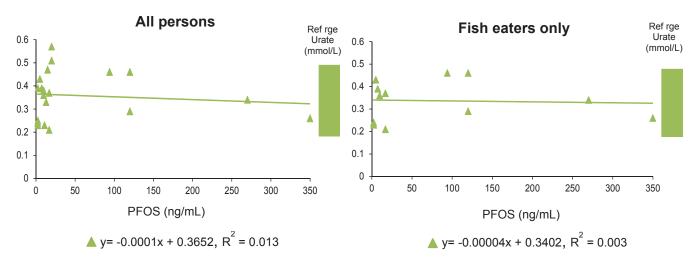


#### <u>Other</u>

#### Glucose ( > )



Urate (A)



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## Appendix E: Letter to CFA CEO



ABN: 55 158 303 167 PO Box 316, Darling South, VIC 3145 Tel: 03 9569 3918/03 9572 1448 Fax: 03 9563 5330

Mr Mick Bourke, Country Fire Authority 8 Lakeside Drive, Burwood East, Vic, 3151

> ToxConsult document: ToxCL281013-R 28<sup>th</sup> October 2013

#### Re: PFOS blood tests

Dear Mr Bourke,

To date twenty four persons have volunteered to have blood samples taken for measurement of perfluorinated chemicals (PFCs) in their serum. Approximately 50% have indicated that in the past they have eaten fish from Lake Fiskville. Included in the overall group are people who are not involved with training operations at Fiskville, and some who are not employees of CFA. All persons have had additional blood taken for measurement of heavy metals, haematology parameters, and clinical chemistry screening that included tests for liver, kidney and thyroid function. Furthermore all CFA personnel in the group have had a general medical examination given by the CFA medical officer. All persons have agreed to have the results of their tests made anonymously available for evaluation.

Only two of the eight PFCs looked for in serum were measurable. These were PFOA and PFOS. The PFOA concentrations for all individuals were well within what is expected for the general population. The majority of the PFOS measurements were also comfortably within the values for the general population. A few individuals had PFOS concentrations at, or slightly above, the upper edge of the background range. These results are higher than what is expected for the majority (95%) of the general population. Nevertheless they were still markedly less than serum concentrations in factory workers making PFOS, and for whom there are no PFOS associated changes in blood parameters or demonstrable illness.

None of the individuals examined had changes in their blood parameters characteristic of PFOS, or which correlated with their PFOS serum concentration. Some persons had blood parameters

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outside the reference ranges but these were associated with existing health conditions, medication or admitted lifestyle factors.

The CFA medical doctor has discussed the results of their medical examination and testing with each person. Where necessary he has encouraged them to follow up their health condition with their GP and has supplied a facilitating letter.

In conclusion, we do not expect there to be any health implications arising from the concentrations of PFOS measured in the serum of the persons investigated.

Yours faithfully,

Roger Drew, PhD, DABT, Toxicologist & Health Risk Assessor, ToxConsult Pty Ltd.

Adjunct Associate Professor, Department of Epidemiology & Preventative Medicine, Monash University

Dr Michael Sargeant, CFA Medical Officar, Public Health Management Pty Ltd.

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## Appendix F: International fish advisories

A number of authorities have provided advice regarding consumption of fish containing PFOS (Dutch VWA 2008, German FIRA 2006, Alabama DoPH undated, Minnesota MDH 2008, Ontario MoE 2013). These fish advisories are not regulatory standards. The technical derivation of many could not be found (Dutch VWA 2008, Alabama DoPH undated, Minnesota MDH 2008, Ontario MoE 2013). However when the basis of the fish advisories was available it is apparent they are very conservative, primarily because large amounts of fish are assumed to be eaten every day of a person's life (this is patently not the case for fish consumed from Lake Fiskville). In addition, despite the fact that fish are by far the greatest contributors to PFOS intake by humans, only a small fraction of the TDI is assigned by some agencies to fish. The resulting fish advisories are precautionary, occasional consumption of fish with higher PFOS concentrations does not necessarily indicate an unacceptable health risk or that adverse health effects are likely.

Information on the derivation of guidance concentrations for PFOS in fish from some countries is below.

#### Netherlands:

A maximum permissible concentration (MPC) for PFOS in fish has been calculated by RIVM (2010) based on the European Food Safety Authority TDI of  $1.5 \times 10^{-4}$  mg/kg bw/d (EFSA 2008), assuming a body weight of 70 kg, a daily intake of 115 g fish, and a maximum contribution to the TDI from fish of 10%. The math are  $(0.1 \times 1.5 \times 10^{-4} \times 70) / 0.115 = 9.1 \times 10^{-3}$  mg/kg = 9.1 µg/kg (9.1 ng/g) fish wet weight.

If more realistic assumptions are made (e.g. 90% of the TDI for fish and 30 g fish eaten on average per day) the resulting MPC is 315 ng/g fish.

RIVM (2010) indicates that after a fire fighting foam incident at Schipol airport in 2008 in which foam containing PFOS was washed into a nearby canal, the Dutch Food and Consumer Product Authority ("Voedsel en Warenautoriteit", VWA) concluded that PFOS concentrations in fish from the canal were high (400-1,500  $\mu$ g/kg as compared to 30  $\mu$ g/kg in fish caught upstream from the incident location) and consumption was advised against. The advice was for the particular incident and was not underpinned by quantitative considerations of risk to health.



## Germany:

In order to evaluate the significance of high PFOS concentrations measured in fish from an aquaculture pond in North Rhine Westphalia, Germany, the German Federal Institute for Risk Assessment (German FIRA 2006) used a TDI of 0.1 µg/kg /day to derive a theoretical tolerable intake of 6 µg PFOS per day for a 60 kg individual.

At an assumed fish consumption rate of 300 g/day, it was determined 100% of the TDI would be exhausted at a PFOS fish concentration of 0.02  $\mu$ g/g fish (6  $\mu$ g PFOS/day ÷ 300 g fish/day. However FIRA reasoned it was unlikely for a person to continually eat this amount of fish each day for their lifetime. It was therefore concluded that PFOS concentrations under 0.02  $\mu$ g/g (i.e. 20 ng/g) in fish are tolerable.

## Alabama:

The Alabama Department of Public Health (Alabama DoPH, undated) combined the RfD for PFOS derived by the US EPA (2009) of 0.08  $\mu$ g/kg/day with standard information for national body weight and food consumption patterns to determine the following advisories for PFOS in fish:

- No restriction: 0 40 µg/kg
- 1 meal/week: >40 200 µg/kg
- 1 meal/month: >200 800 µg/kg
- Do Not Eat: >800 µg/kg

Details on how the calculations were performed and the values used were not provided.

- However assuming 100% of the RfD was assigned to fish and 70 kg body weight, the amount of fish assumed by Alabama DoPH to be consumed per day can be calculated from the maximum value of the "no restriction" range:
  - ο A TDI of 0.08  $\mu$ g/kg/day equates to 5.6  $\mu$ g/d PFOS for a 70 kg individual. Therefore 5.6  $\mu$ g/d PFOS ÷ 40  $\mu$ g PFOS/kg fish = 0.14 kg/d fish (i.e. 140 g/d).



## Minnesota:

The Minnesota Department of Health (MDH 2008) have the same PFOS fish advisories as Alabama. The scientific derivation of the Minnesota fish advisories could not be found.

## Ontario:

The Ontario Ministry for the Environment (Ontario MoE 2013) provides consumption guidelines for various contaminants in sporting fish. Included is PFOS. Details for the derivation of the guidelines are not provided. However, it is stated that consumption guidelines are based on tolerable daily intakes provided by the Food Directorate of Health Canada. There are five areas in Ontario where consumption of fish is restricted due to concentrations of PFOS they contain. The restrictions are attributed to PFOS released from historic use of firefighting foams.

In Ontario consumption restrictions for PFOS begin at 80 ng/g fish, with complete restriction on consumption advised for levels above 160 ng/g for the sensitive population and 640 ng/g for the general population. The 'sensitive population' is defined by Ontario MoE (2013) to include women of child-bearing age and children less than 15 years. Other agencies do not sub-categorise the population, presumably because the TDI is set to include the sensitive sub-populations.

Details for the derivation of the Ontario PFOS fish guidelines are not provided in Ontario MoE 2013.

## Appendix H 24 Pages

# Toxicity Profiles for Perflourinated compounds (PFCs)



## HUMAN HEALTH RISK ASSESSMENT – FISKVILLE COMMUNITY

## 4549 GEELONG-BALLAN RD, FISKVILLE VICTORIA

## **APPENDIX H**

## TOXICITY PROFILES- PERFLOURINATED COMPOUNDS, MARCH 2014



## HUMAN HEALTH RISK ASSESSMENT – FISKVILLE COMMUNITY APPENDIX H - TOXICITY PROFILES-PERFLOURINATED COMPOUNDS, MARCH 2014

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## APPENDIX H - TOXICITY PROFILES- PERFLOURINATED COMPOUNDS -

## **1** INTRODUCTION

Perfluorinated compounds (PFCs) area class of chemicals that are ubiquitous in the environment as a result of anthropogenic activities. They have been used in a variety of industrial processes and products including carpets, cooking utensils, clothing and non-stick coatings. PFOA and PFOS are PFCs that are known to have been used in alcohol resistant aqueous film forming foams (AR-AFFF). These compounds are predominantly used in "B class foams" formulations as they are able to form a protective film that contain vapours while fighting flammable liquid fires. Other PFCs such as fluorotelomer sulphonic acid (6:2 FTS) have been developed as replacements since restrictions on the use of PFOA and PFOS have been put in place.

PFCs have been identified in water, sediments, plants, foodstuffs and animals (in particular fish). In humans, PFCs have been found predominantly in blood as some are known to bind strongly to plasma proteins. PFOS is known as a 'persistent organic pollutants'. (ATSDR 2009). There are hundreds of chemicals that are classed as PFCs. They can be described simply as those compounds with fluorine atoms bound to carbon atoms typically in chains up to C20 in length.

Little is known about the toxicology of many PFCs. Therefore in toxicological reviews they are usually split into different classes (DME 2012, Perforce 2006, NICNAS 2011). Classes considered in the literature include but may not be limited to:

- Perfluoalkyl sulfonic acids (PFAS),
- Perfluoroalkyl carboxylic acids (PFAA),
- Fluorotelomers,
- Fluoropolymers, and
- Perfluoroalkanamides.

Perfluorooctane sulfonic acid (PFOS) and perfluorooctanoic acid (PFOA) are two PFCs that have been studied extensively. They are two of the four "Indicator B6" PFCs that are the most commonly detected PFC in humans (USEPA 2013). The other two PFCs are perfluorohexane sulfonic acid (PFHxS) and perfluorononanoic acid (PFNA). The toxicity of these substances and their potential to bioaccumulate in the environment increases with alkyl chain length. There is uncertainty due to insufficient toxicological data for many PFCs therefore Cardno Lane Piper has grouped PFCs by functional group and using the most commonly detected PFC identified in humans as surrogates (see Section 2.1). A toxicology profile for PFCs has been provided by Dr Roger Drew of ToxConsult. This was peer reviewed by Dr Brian Priestly of Priestly Consulting

This is similar in some respects to how NICNAS (2007 and 2009) has placed restrictions on the use of PFAS based on the toxicity of PFOS. According to NICNAS (2007) "*PFOS-based and related PFAS-based chemicals continue to be restricted to only essential uses, for which no suitable and less hazardous alternatives are available*" (*NICNAS 2007*).



Further, many other Perfluorinated Compounds (OPC) degrade to PFAS or PFAA compounds including PFOA and PFOS which are stable and highly resistant to metabolic and environmental degradation.

## 2 PERFLUOROALKYL COMPOUNDS IN THE ENVIRONMENT

PFCs have been released during manufacturing processes to air, water or soil. They have been measured in urban air up to 0.05 ng/m<sup>3</sup> (PFOS) and 0.9 ng/m<sup>3</sup> (PFOA) however they are generally present at below 0.001 ng/m<sup>3</sup> (ATSDR 2009). The degradation of PFCs is considered slow where they typically remain suspended as particulate matter for a few days before partitioning to water or soil. PFC are known to have been transported over thousands of kilometres from their source and have been identified in water. The background level in water (due to anthropogenic activities) of PFC is considered to be less than 50 ng/L (ATSDR 2009). PFCs generally do not degrade in water, those that do degrade do so to smaller PFCs such as PFOS and PFOA. PFCs also do not degrade in soils where they are potentially carried down in to groundwater.

## 2.1 Range of Perfluoroalkyl Compounds (PFCs)

PFCs are sometimes referred to as fluorosurfactants. Their molecular structure is consistent with that of typical surfactants (lipophilic hydrocarbon backbone with a polar functional group) however fluorine atoms replace hydrogen atoms on the hydrocarbon backbone. PFCs are synthetic chemicals that typically have two components:

- An alkyl group which consists of a chain of carbon atoms surrounded by fluorine atoms; and
- One of a number of different hydrophilic functional groups such as a carboxylic acid, amide, alcohol or sulphonate group.

The chemical structure of the PFCs gives them the "*unique property of being able to repel oil, grease, and water*" (ATSDR 2009) hence their 'non-stick properties'. The PFCs that are most often discussed in literature reviews on PFCs are those that have a hydrocarbon backbone (chain length) between 4 and 12 carbons long and either a sulfonic acid or a carboxylic acid as the hydrophilic functional group.

Various PFC are used in Class B fire-fighting foam products (referred to as "foams" in this appendix) however they are not typically identified in product material safety datasheets. Foams used on-site are known to comprise of PFAS, PFAA and fluorotelomers (6:2 FTS). A limited number of PFC from other classes (Perfluorooctane sulfonamide, N-alkyl Perfluorooctane sulfonamide and N-alkyl Perfluorooctane sulfonamidoethanol) have also been included in analytical suite however their inclusion is based the analytical suite offered by testing laboratories. Some of these other PFCs have also been detected in water at Fiskville Training College. There are many other PFC from various classes<sup>1</sup> that are not included in the analytical suite.

A list of the various PFCs classes routinely included in laboratory analytical suites and potentially used in AFFF foams is shown in Table 2-1. This list includes the relevant acronym, the number of carbon atoms in the alkyl chain and the relevant hydrophilic functional group.

<sup>&</sup>lt;sup>1</sup> Perfluoroalkyl sulphinate (PFASi), Fluorotelomer alcohol (FTOH), Fluorotelomer acid (FTA), Fluorotelomer unsaturated acid (FTUA), Perfluoroalkyl phosphonic acid (PFAPA), Perfluoroalkyl phosphinate (PFPi), Perfluoroalkyl phosphate ester (PAP), di-Perfluoroalkyl phosphinate (diPAP) and N-alkyl Perfluoroctane sulfonamidoacetic acid (N-Alkyl FOSAA).





#### Table 2-1: Classes of Perfluoroalkyls Compounds (PFCs) in the Analytical Suite and their Chemical Makeup

Family Name	Acronym	Carbons in chain	Aliphatic chain	Functional Group
Perfluoroalkyl sulphonic acid	PFAS	4 to 20	CF <sub>3</sub> (CF <sub>2</sub> ) <sub>n</sub> <sup>a</sup>	SO₃H
Perfluoroalkyl carboxylic acid	PFAA	4 to 20	$CF_3(CF_2)_n^{\mathbf{b}}$	CO₂H
Fluorotelomer sulphonic acid	X:2 FTS <sup>c</sup>	4 to 15	$CF_3(CF_2)_n(CH_2)_2^d$	SO₃H
Perfluoroalkyl sulfonamide	FOSA		CF <sub>3</sub> (CF <sub>2</sub> ) <sub>n</sub>	SO <sub>2</sub> NH <sub>2</sub>
N-alkyl Perfluoroalkyl sulfonamide	N-Alkyl FOSA	Typically 8	CF <sub>3</sub> (CF <sub>2</sub> ) <sub>n</sub>	SO <sub>2</sub> NH(Me or Et)
N-alkyl Perfluoroaklyl sulfonamidoethanol	N-Alkyl FOSE	(n = 7)	CF <sub>3</sub> (CF <sub>2</sub> ) <sub>n</sub>	SO <sub>2</sub> N(CH <sub>2</sub> CH <sub>2O</sub> H) (Me or Et)
Notes:				

a.

n = 7 for PFOS n =7 for PFOA b

X in the acronym X:2 FTS equals n+1, e.g. n= 5 for 6:2 FTS. C.

n = 5 for 6:2 FTS. d.

> The approach used in this HHRA is to assess toxicity of the PFCs according to their respective hydrophilic functional groups. PFC were placed in their respective classes due to the large variety of PFCs that may be present as contaminants. Two distinct classes plus a third broad generic class are outlined below including the surrogate assigned to represent each class. The PFC classes are:

- **PFAS:** This PFC class is assessed using PFOS as a surrogate. PFOS was selected by Cardno as the toxicological database for this compound is extensive. PFOS was until recently the main PFC used in fire-fighting foams. It has been detected in water at CFA Fiskville Training Ground.
- **PFAA**: PFOA is the PFC used as a surrogate for this class. PFOA was selected by Cardno for the same reasons outlined above for the PFAS class. PFCs from this class (not including PFOA) are still used as foams in portable fire extinguishers at CFA Fiskville Training Ground.
- OPC: All other PFCs not belonging to the other classes (PFAS and PFAA) identified in water at CFA Fiskville Training Ground were assessed using 6:2 FTS as a surrogate. 6:2 FTS is believed to be the main PFC ingredient used in the foams used at CFA Fiskville Training Ground.

The PFCs assessed in this HRA and their respective classes are shown below in Table 2-2.



Perfluorinated Compound (PFC)	Acronym				
Perfluoroalkyl sulfonic acids (PFAS)					
Perfluorobutane Sulfonic Acid	PFBS				
Perfluorohexane Sulfonic Acid	PFHxS				
Perfluoroheptane Sulfonic Acid	PFHpS				
Perfluorooctane Sulfonic Acid	PFOS				
Perfluorodecane Sulfonic Acid	PFDS				
Perfluoroalkanoic acids (PFAA)					
Perfluorobutanoic Acid	PFBA				
Perfluoropentanoic Acid	PFPA				
Perfluorohexanoic Acid	PFHxA				
Perfluoroheptanoic Acid	PFHpA				
Perfluorooctanoic Acid	PFOA				
Perfluorononanoic Acid	PFNA				
Perfluorodecanoic Acid	PFDA				
Perfluoroundecanoic Acid	PFUnA				
Perfluorododecanoic Acid	PFDoA				
Perfluorotridecanoic Acid	PFTrA				
Perfluorotetradecanoic Acid	PFTeA				
Other fluorinated Compounds (OFC)					
6:2 Fluorotelomer Sulfonate	6:2 FTS				
Heptadecafluorooctane sulphonamide	FOSA				
N-Methylheptadecafluorooctane sulphonamide	NMeFOSA				
N-Ethylheptadecafluorooctane sulphonamide	NEtFOSA				
N-Methylheptadecafluorooctane sulphonamidoethanol	NMeFOSE				
N-Ethylheptadecafluorooctane sulphonamidoethanol	NEtFOSE				

# Table 2-2: Perfluoroalkyls compounds (PFCs) included in laboratory analytical suites in the surface water monitoring events conducted at CFA Fiskville Training College.

## 2.2 Perfluoroalkyl compounds (PFC) and their production

PFC are synthesised using 2 main processes:

- *Electrochemical fluorination (ECF):* ECF has historically been used to synthesise PFCs such as those used in AFFF fire-fighting foams including PFOS and PFOA. The purity of PFCs synthesised by ECF are typically considered a "technical mixture" as multiple analogues of the target PFC may be produced, i.e. molecules with the same molecular formula.
- *Telomerisation*: Telomerisation is a more recent process used to synthesise PFCs. It does so by first preparing an intermediate, perfluoroalkyl iodide, which is then used to produce a variety of PFCs including some of those listed in Table 2-1 (e.g. 6:2FTS). The benefit of telomerisation is that more control of the synthesis process is gained and the purity of the PFC produced is improved.

Advantages of PFC manufactured using telomerisation over the ECF process include:

 Preparation of straight chain PFCs is possible thus avoiding the preparation of "technical mixtures"; and



• PFC can be prepared without fluoride atoms on every carbon atom in the alkyl chain. This is associated with a reduction in toxicity and bioaccumulation (Dupont 2008) however peer-reviewed technical literature is not currently available to confirm this.

## 2.3 Properties of surrogate PFCs

A summary of the key properties for the surrogate PFC, i.e. PFOS, PFOA and 6:2FTS, is shown below in Table 2-3.

# Table 2-3: General Properties of the Surrogates Used to Classes of Represent Perfluoroalkyl Compound (PFC).

Property		PFC Class				
		PFAS <sup>a</sup>	PFAA <sup>a</sup>	OPC <sup>c</sup>		
Surrogate	Compound	PFOS	PFOA (PFO <sup>b</sup> )	6:2 FTS		
Name		Perfluorooctane sulphonate	Perfluorooctanoic acid (Perfluorooctanoate)	1H,1H,2H,2H- Perfluorooctane sulphonate		
IUPAC Name		<u>1,1,2,2,3,3,4,4,5,5,6,6,7,7,</u> <u>8,8,8-</u> <u>heptadecafluorooctane-1-</u> <u>sulfonic acid</u>	2,2,3,3,4,4,5,5,6,6,7,7,8,8,8 -pentadecafluorooctanoic acid	<u>3,3,4,4,5,5,6,6,7,7,8,8,8-</u> tridecafluorooctane-1- sulfonic acid		
Family		Perfluoroalkyl sulphonates	Perfluoroalkyl carboxylates	Fluorotelomer sulphonates		
Process		ECF	ECF	Telomerisation		
CAS No		(2795-39-3	335-67-1	27619-97-2		
Molecular I	Formula	F(CF <sub>2</sub> ) <sub>8</sub> SO <sub>3</sub> H	F(CF <sub>2</sub> ) <sub>8</sub> CO <sub>2</sub> H	F(CF <sub>2</sub> ) <sub>6</sub> CH <sub>2</sub> CH <sub>2</sub> SO <sub>3</sub> H		
Molecular \	<u> </u>	538 (Potassium Salt)	414	427		
Melting Po		>400	45 to 50	NI		
Vapour Pre at 20°C)	essure (mmHg	2.48 x 10 <sup>-6</sup>	0.017	NI		
Water Solu	ibility (mg/L)	570	9500	NI		
L L LA LA C	Atmospheric	114 days	90 days	NI. Assumed to be		
Half-life <sup>c</sup>	Water (25 <sup>°</sup> )	41 years	> 92 years	persistent.		
Biodegradable		Does not degrade chemically or biologically	Yes, under sulphur limiting and aerobic conditions.	No biodegradation products identified in sludge.		
Persistent Organic Pollutant (POP)		Yes	No	No		
BMF		22 – 160 <sup>d</sup>	1.3 to 13 (dolphin)	Low		
BCF		1000-4000 <sup>d</sup> (fish 2796 – 3100 <sup>d</sup> )	4 (rainbow trout)	<502		
Bioaccumulative		Yes	No <sup>e</sup>	No		

NI = no value identified in literature, PFAS = Perfluoroalkyl sulfonic acids, PFAA = Perfluoroalkyl Carboxylic Acid, OPC = Other Perfluorinated Compounds, PFOS = Perfluorooctane Sulfonic Acid, PFOA = Perfluorooctaneic Acid, 6:2FTS = 6:2 Fluorotelomer Sulfonate.

a. unless specified the property has been sourced from USEPA (2012a).

b. PFOA dissociates to perfluorooctanoate (PFO) in the environment

c. Based on 3<sup>rd</sup> party details from Dupont (2008 and 2012). Data not published in peer reviewed journal

d. PFOS fulfils the criteria for bioaccumulation based on the high concentrations that have been measured in top predators at various locations such as the Arctic, the US and Sweden (KemI 2004).

e. Longer chain PFAA are potentially bioaccumulative.



## 3 EXPOSURE TO PERFLUOROALKYL COMPOUNDS

## **3.1 Background Exposure to Perfluoralkyl compounds (PFCs)**

For the general population the predominant route of exposure is from oral exposure, i.e. from consumption of food and water that are contaminated with PFC (EFSA 2008) The intake from fish was considered more significant than intake from water (EFSA 2008, 2012). Higher PFOS levels were typically quantified in freshwater fish than marine or diadromous fish (ATSDR 2009), i.e. fish that live at sea and breed in freshwater or vice versa (e.g. salmon, trout, etc.). Dermal exposures are considered minor routes of exposures as PFCs in general have poor dermal absorption. The exposure pathway from air is also considered minor (unless a person resides in the vicinity of a manufacturing facility) as in general intake from this pathway would make up only a small portion of background intake.

The upper intakes of PFOS (0.030  $\mu$ g/kg/day) and PFOA (0.047  $\mu$ g/kg/day) from dietary exposure have been estimated for the general populations in North America and Europe according to ATSDR (2009). Extensive review of dietary intakes has also been conducted by EFSA (2008, 2012). The total intake from water was considered insignificant as it was estimated to be 0.00019 $\mu$ g/kg/day (0.014 $\mu$ g/person) for PFOS and 0.000024 $\mu$ g/kg/day (0.018 $\mu$ g/person) for PFOA (EFSA 2008). The total intake from the diet varied and depended on a person's geographical location, the type of food they eat and its source.

Background exposure in this HHRA is based on a survey conducted by Food Standards Australia New Zealand which looked at concentrations of PFCs in foods packaged in glass, paper, plastic or cans (FSANZ 2011). A summary is provided in the HHRA (See Section 5.4). The assumed background dietary intake for adults in the Australian population exposed to PFAS and PFAA are 0.01 and 0.02  $\mu$ g/kg/day respectively. The dietary exposure to PFCs for Australian Adults exposures is lower than for adults in North America and Europe.

Estimates of the background concentrations of OPC from surveys in 13 European countries were not quantifiable, i.e. very low, therefore the background dietary intake of OPC is therefore assumed to be negligible (i.e. set to zero).

## 3.2 Intake from Other Sources

The intake from other sources such as contact with materials on cooking utensils, air and dusts is considered negligible compared to levels from diet. PFC may be present in contact materials used in cooking (e.g. non-stick coatings on frypans, paper as used in bags for microwaving popcorn) and therefore potentially contaminate foods. The intake from this source was considered negligible, however the available data is insufficient to discount contribution from food contact materials such as non-stick coatings on cookware and paper food packaging (EFSA 2008). The intake of PFAS from air (indoor and outdoor) was also considered negligible as it was determined to be <0.001 $\mu$ g/kg/day (EFSA 2008). The level of PFOS in indoor air on dust was based on dust collected in studies of residential properties from Japan (Moriwaki 2003, mean of 0.2 $\mu$ g/g, range of 0.011 to 2.5 $\mu$ g/g, n=11) and Canada (Kubwabo, 2005, mean, 0.443 $\mu$ g/g, range of <0.0046 to 5.065 $\mu$ g/g, n = 67). The level of PFOS in outdoor air ranged from 0.000001 to 0.00001 $\mu$ g/m<sup>3</sup> and for outdoor dusts (0.03 to 0.1 $\mu$ g/g) (EFSA 2008).



## 4 ABBREVIATED TOXICOLOGICAL PROFILE FOR PFOS & PFOA

The abbreviated toxicological profile provided here in Section 4 has been prepared by Dr Roger Drew of Toxconsult.

The toxicological literature on the PFCs is large and complex. The toxicological profile below is not intended to be comprehensive, rather it is an easy to read (note format) compilation of information relevant for this occupational exposure risk assessment. Emerging community epidemiology studies have not been reviewed herein as they are more relevant for other risk assessments being undertaken within the overall Fiskville project. Much of the information below has been gleaned from agency reviews but key research papers have also been accessed. The reference list contains a large number of papers that are not cited in the toxicological profile, they nevertheless have been used to formulate the summaries in the information below.

## 4.1 Absorption, Distribution, Metabolism and Excretion:

- Well absorbed orally, subject to enterohepatic recirculation.
- Not metabolised.
- Urine is the major route of elimination but is poor (significant species differences).
- Marked differences in serum elimination half-lives between species and PFCs.

#### PFOS:

Rat	~ 40 -100 days
Monkey	~200 days
Human	5.4 years (95%CL 3.9 – 6.9)

#### PFOA:

Rat <sup>a</sup>	Female 1.9 to 24 hours	
και	Male 4.4 to 9 days	
Monkey <sup>b</sup>	21 to 30 days	
Human <sup>b</sup>	3.8 years (95% CI 3.1–4.4)	
rats are usu	ifference in elimination, experimental NOAELs in male ally lower than females. ally lower than females.	

• Blood (serum) PFOS levels are the best indicator of exposure and for determining margins of exposure when assessing risk (3MCompany 2003, MDH 2008, DFG 2011).

## 4.2 General Distribution:

- Not accumulated in fat.
- Primarily confined to extracellular water, i.e. primarily in serum (Vd 0.2L/kg).
- High protein (albumin) binding, including to fatty acid-binding protein.

The distribution of PFOS is summarised as follows:

- The liver concentration in humans, monkeys, hamster, cows and chickens is approximately the same as in serum or slightly higher. However in rats, mice, sheep and seals it is 4 – 5 times higher.
- In all species PFOS kidney concentrations are about the same as in serum.
- In all species PFOS muscle concentrations are 10 times lower than in serum, other tissues are lower still.



The distribution of PFOA is summarised as follows

- A similar distribution profile to PFOS. Liver and kidney concentrations are the same or less than in serum, muscle concentrations are more than 10 times lower than serum.
- May be dose dependent with greater distribution into liver at low compared to high doses.

## 4.3 **Biochemical Effects**:

Both PFOS and PFOA are agonists of PPAR $\alpha$  in rodents and produce the typical effects as observed with other peroxisome proliferator substances (see below). PFOA is a stronger agonist for these effects than is PFOS. Humans and monkeys are equally refractory to the effects of PPAR $\alpha$  activation but rats and mice are very sensitive. This species difference is largely due to lower number of receptors. However not all the effects of PFOS and PFOA are necessarily mediated by PPAR $\alpha$ . The mechanisms of toxicity are not fully understood but may include effects on fatty acid transport and metabolism, membrane function, and/or mitochondrial bioenergetics.

Experiments in animals (rats and monkeys) show PFOS and PFOA may affect the transport and metabolism of cholesterol and fatty acids. Clinical chemistry parameters indicate potential for liver toxicity but histopathology is only evident with very high doses. Also observed is a tendency for lower circulating  $T_3$  and at high doses in rodents, hypothyroidism is evident and likely contributes to the low neonatal survival in these species. It should be noted the physiological stability of thyroid hormones in rats is different to that of humans and primates; this renders rodents more susceptible to agents that affect the utility, catabolism and production of thyroid hormones. Monkeys are the most relevant species for humans.

All the above effects are dependent upon the PFC concentration in blood serum.

Thyroid hormones seem to start to be altered when serum PFOS level reaches the 70–90 mg/L range, regardless of animal species (rat or monkey) or route of administration (diet, gavage, or drinking water) (Lau 2012).

In the 6 month monkey PFOS gavage study used by agencies for TDI setting (Seacat et al. 2002) the following is observed:

- At high serum concentrations hypolipidemia and metabolic wasting, with signs of liver toxicity.
- At serum concentrations not causing overt toxicity (approximately 60 100 mg/L) the primary findings are changes in biochemical parameters associated with lipid metabolism. The animals show increased liver weight and decreases in body weight, together with decreased cholesterol and HDL, decreased triglycerides and T<sub>3</sub>. These changes have been shown to be readily reversible as serum concentrations decrease.
- Serum NOAEL (as BMDL<sub>10</sub>) 35 mg/L.

For humans there are no substantial findings in serum hepatic enzymes, cholesterol or lipoproteins in persons occupationally exposed during manufacture of PFOS when serum PFOS concentrations are less than approximately 2 - 6 mg/L. Although firm conclusions at higher concentrations are difficult to make, in worker groups with the highest serum PFOS there is a trend for lower blood cholesterol and HDL, increased serum triglyceride and ALT, and increased  $T_3$ .

Animal studies show reduced synthesis and esterification of cholesterol and enhanced oxidation of fatty acids in the liver. Overall the data suggests high serum PFC may be associated with changed metabolic status, altered serum lipoprotein profile, and therefore may



influence risk factors for cardiovascular disease. Some of the biochemical effects are similar to the fibrate and statin therapeutic agents.

## 4.4 Genotoxicity:

In a large range of tests PFOS & PFOA are negative for genotoxicity.

## 4.5 Acute Toxicity:

- PFOS Moderately toxic, rat oral LD<sub>50</sub> ~250 mg/kg.
- PFOA Moderately toxic, rat oral LD<sub>50</sub> ~400 1,800 mg/kg (M >500 & F 250 – 500 mg/kg).
- In life symptoms include decreased body weight, decreased limb tone, anorexia, and accompanying hypoactivity.
- PFOA is a weak skin irritant, PFOS not an irritant.
- PPARα agonists (PFOA >>PFOS): ↑ liver weight (hepatocyte hypertrophy), ↓ serum glucose, ↓ cholesterol, ↑β-oxidation fatty acids.

## 4.6 Sub-chronic & Chronic Oral Toxicity

#### PFOS:

- ↓ total cholesterol an early consistent finding, cumulative toxicity expressed as metabolic wasting.
- 2 year rat dietary study with PFOS (~ 0.04, 0.14, 0.4 &1.5 mg/kg/d) (Thomford 2002) showed:
  - Trend for increased survival in males at two highest doses but not females.
  - Centrilobular hypertrophy ( $\uparrow$  SER but  $\leftrightarrow$  peroxisomal proliferation).
  - NOAEL 0.14 mg/kg/d.

  - Evidence for induction of thyroid and mammary tumours in F was limited (no dose response).
- BMCL<sub>5</sub> (equivalent to serum concentration NOAEL) (3MCompany 2003, Olsen et al. 2003b):
  - 31 mg/L for rat pup weight gain in multigeneration reproduction studies.
  - 44 mg/L for rat liver toxicity.
  - 62 mg/L for rat liver adenomas.
  - 35 mg/L for monkey  $\downarrow$  cholesterol & T<sub>3</sub> (Seacat et al. 2002, MDH 2008).

#### PFOA:

- Sub-chronic rat studies consistently show ↓ weight gain, ↑ liver weight (hepatocellular hypertrophy, peroxisome proliferation), high doses hepatocellular necrosis & ↑ mortality (preceded by wasting).
  - NOAEL (M) 0.6 mg/kg/d based on increased liver weight at higher doses (Goldenthal 1978) but this dose has shown ↑peroxisome proliferation & ↑ liver weight (Perkins et al. 2004).
- 2 year dietary rat study at ~ 1.5 and 15 mg/kg/d (Sibinski 1987).
  - Dose related ↓ body weight gain, and at top dose ↑ serum ALT, AST, AP & CPK.



NOAEL (M) 1.3 mg/kg/d based on  $\uparrow$  liver weight.

- Hepatocellular and Leydig cell adenomas, and pancreatic acinar cell hyperplasia in males (Sibinski 1987, Biegel et al. 2001).
- Tumour pattern is typical of PPARα agonists (Klaunig et al. 2003, 2012, Lau 2012).

In monkeys doses of 0, 3, 10 or 30 mg/kg/d for 6 months showed dose dependent ↑ liver weight (mitochondrial proliferation) in all treatment groups. No histopathological evidence of liver injury at 3 or 10 mg/kg/d. No changes in clinical chemistry, hormones, urine composition or haematological effects (Butenhoff et al. 2002). NOAEL <3 mg/kg/d based on ↑ liver weight.

- BMCL<sub>10</sub> (equivalent to serum concentration NOAEL) (Butenhoff et al. 2004a):
  - 23 mg/L ↑ liver weight (monkey), 34 mg/L (rat).
  - 29 mg/L Post-natal effects 2-generation rat.
  - 60 mg/L  $\downarrow$  Body weight (monkey)
  - 125 mg/L ↑ Leydig cell tumours (rat). Questionable significance to humans.

## 4.7 Developmental and Reproductive Toxicity

#### PFOS:

- Developmental and 1 & 2 generation rat studies show foetal toxicity and neonatal effects at doses similar to, or below those causing maternal toxicity.
  - ↓ foetal weight, cleft palate, anasarca (oedema), delayed ossification (sternebrae and phalanges) and cardiac abnormalities (ventricular septal defects and enlargement of the right atrium).
  - Dose response curves are steep, with high mortality observed early after birth.
  - In surviving pups delays in growth and development accompanied by hypothyroxinemia.
- Late gestational age seems to be a very vulnerable period.
- Neonatal deaths are hypothesised to be due to delayed lung development but more likely to be hypothyroxinemia in the pups (Lau 2012).
- Two-generation reproduction studies give a LOAEL of 0.4 mg/kg/d and NOAEL of 0.1 mg/kg/d.

#### PFOA:

- Teratology studies at 100–150 mg/kg/d for rats and 50 mg/kg/d for rabbits are negative (Lau et al. 2004).
  - In rats, NOAEL for maternal and developmental toxicity were 5 and 150 mg/kg/d.
- In mice post natal survival  $\downarrow$  at >5 mg/kg/d & dose dependent growth deficits  $\ge$  3 mg/kg/d.
  - NOAEL 1 mg/kg/d (Lau et al. 2006).
  - In addition to gestational exposure, abnormal lactational development of dams may play a role in the early growth retardation. PPARα may have a role in delayed weight gain, but other mechanisms may also contribute.
- In a two generation reproduction study in rats at 1, 3, 10 or 30 mg/kg/d by gavage (Butenhoff et al. 2004b); ↓ body weight, ↑liver & kidney weight in F<sub>0</sub> & F<sub>1</sub>; ↓pup weight at top dose.
  - NOAELs 30 mg/kg/d for reproductive function, 10 mg/kg/d. for sexual maturation, and < 1 mg/kg/d for body weight and increased liver weight.



## 4.8 Summary of the Relevant Human Data

In general, no consistent association between serum fluorochemical levels and adverse health effects in worker populations has been observed (Lau et al. 2007).

#### PFOS:

- Long half-life (ave 5.4 yr), therefore will tend to accumulate.
- Liver:serum ~2:1
- Breast milk:serum ~ 0.01:1 (same in rodents).
- Crosses placenta but neonate:mother serum ~0.5:1 (or less).
- General community population mean serum concentrations: 0.005 0.05 mg/L.
  - Reliable range for individuals in the populations: 0.00006 0.3 mg/L.
- PFOS worker serum concentration range: 0.06 12.8 mg/L.
  - NOEL (for possible  $\downarrow$  serum cholesterol & lipoprotein changes)  $\sim 2 6$  mg/L.
  - Medical surveillance of PFOS production employees has not been associated with adverse clinical chemistry, haematology results or illness (Olsen et al. 1999, 2003a).

#### PFOA:

- Long half-life (ave 3.8 yr), therefore will tend to accumulate.
- Concentration neonate:mother plasma ~1.2 1.9:1
- Some occupational studies have found a positive association with cholesterol and triglycerides whereas others found no such association. Overall there is no consistent pattern of changes, but HDL may be negatively associated and triglycerides positively associated with serum PFOA (effects marginal). No significant increased risk of ischaemic heart disease or cancer.
- PFOA serum levels appear inversely associated with birth weight but not low birth weight or small gestational age.
- Hepatic toxicity, hypolipidemia, and abnormal hormone levels have not been associated with serum PFOA concentrations in workers whose serum levels have averaged 5 mg/L (0.1-114 mg/L) (Gilliland and Mandel 1996; Olsen et al. 1998, 2000).



## 5 AVAILABLE PUBLIC HEALTH TOXICITY REFERENCE VALUES

## 5.1 Toxicity Reference Values for Perfluorinated Compounds

Australian toxicity reference values (TRVs) for the PFCs are not available. Many of the international agencies that have developed a toxicity reference value (TRV) for PFCs have done so as part of setting a drinking water guideline for the general population. Often these are provisional or interim (German DWC 2006, NCDENR 2007, RIVM 2010, US EPA 2009) and with limited support documentation explaining the basis of the TRV. These guidelines assume a chronic (lifetime) exposure and are conservative (i.e. are precautionary) in order to provide adequate protection for sections of the general population that are thought to be especially vulnerable to chemicals. These are traditionally considered to be the foetus, young children, the old and infirmed, and persons who, for some reason or other (e.g. genetic constitution or acquired disease) are less able to cope with the effects that the chemical may be able to cause.

A summary of the public health TRV, i.e. equivalent to Tolerable Daily Intakes (TDI), that have been derived by various International Agencies is shown in Table 5-1 for PFOS and Table 5-2 for PFOA. This includes the critical study, the point of departure and the uncertainty factors (UF) used derive the TDI.

	Critical Effect		TDI				
Agency	Critical Study	POD (µg/kg/day)	UF <sup>a</sup>	(µg/kg/day)			
International Age	encies	·					
COT (2006a)	Sub-chronic oral study in Cynomolgus monkey <sup>b</sup>	30 (NOEAL)	100	0.3			
EFSA (2008)	Sub-chronic oral study in Cynomolgus monkey <sup>b</sup>	30 (NOEAL)	200	0.15			
FEA (2006) EWG (2002)	2 year dietary study in rat	25 (NOAEL)	300	0.1			
USEPA (2009)	SEPA (2009) Sub-chronic oral study in Cynomolgus monkey <sup>b, c</sup> 30 (NOEAL)		390	0.08			
Other agencies							
MDH (2008) Sub-chronic oral study in Cynomolgus monkey <sup>b</sup> 2.5 (BMD) <sup>d</sup>		30	0.08				
Notes: POD = Point of departure from toxicological study, UF = Uncertainty factor, TDI = Tolerable Daily Intake.							

## Table 5-1: Summary of public health toxicity reference values for PFOS.

COT = Committee on Toxicology, EFSA = European Food Safety Authority, USEPA = United States Environmental Protection Authority. MDH = Minnesota Department of Health, FEA = German Ministry of Health at the Federal Environment Agency.

a. The uncertainty factor is made up of factors to account for interspecies differences (10x) and human variability (10x). An additional UF was applied by some agencies to account for long half-life of PFOS in humans.

b. Seacat et al. (2002). Steady state blood concentration was not achieved however this is not a concern if serum concentrations at the dose associated with NOEL is used.

c. The TDI from the USEPA is based on their derivation of provisional health advisory levels (drinking water guidelines).

d. The POD is based on PBPK modelling required to convert a serum bench mark concentration to a dose. The serum BMDL of 35mg/L was converted to a human equivalent taking into account physiological differences between monkey and humans and PFOS long half-life in humans.



	Critical Effect		Reference				
Agency	Selected Health Endpoint POD (µg/kg/day)		UF <sup>a</sup>	Value (µg/kg/day)			
International agen	cies						
COT (2006b)	2 generation reproductive study in mice.	300 (BMDL <sub>10</sub> )	100	3			
EFSA (2008)	2 generation reproductive study in mice. 300 (BMDL <sub>10</sub> )		200	1.5			
USEPA (2009) b	EPA (2009) <sup>b</sup> Developmental toxicity study in mice. 460 (BMDL		2400	0.2			
FEA (2006)			3000	0.3			
Other agencies	Other agencies						
NC (MDH 2008)	Sub-chronic oral study in cynomolgus monkey.	2.9 (BMDL <sub>10</sub> ) <sup>c</sup>	30	0.09			
MDH (2008)	Sub-chronic oral study in cynomolgus monkey.	2.3 (BMDL <sub>10</sub> )	30	0.077			
Notes: POD = Point of departure from toxicological study, UF = Uncertainty factor, TDI = Tolerable Daily Intake.							

#### Table 5-2: Summary of public health toxicity reference values for PFOA

y

COT = Committee on Toxicology, EFSA = European Food Safety Authority, USEPA = United States Environmental Protection Authority, FEA = German Ministry of Health at the Federal Environment Agency, MDH = Minnesota Department of Health, NC = State of North Carolina.

The uncertainty factor is made up of factors to account for interspecies differences (10x) and human variability (10x). An additional UF was applied by some agencies to account for long half-life of PFOA in humans.

b The TDI from the USEPA is based on their derivation of provisional health advisory levels (drinking water guidelines).

The POD is based on PBPK modelling required to convert a serum bench mark concentration to a dose. The serum BMDL was 23mg/L C. was converted to a human equivalent taking in to account physiological differences between rat and humans and PFOA long half-life in humans.

The TDI derived for PFOS ranged from 0.08 to 0.3 µg/kg/day mainly due to the differences in the uncertainty factors (UF) applied, see Table 5-3. In the US an UF of 3x is applied for intraspecies differences compared to 10x by the European agencies. However, large UF are applied by US EPA (2009) for PFOS (13x) and PFOA (81x) or accounted for in bench mark dose modelling (MDH 2008) to account for the long-half-life of PFOS and PFOA in humans.

#### Table 5-3: Uncertainty Factors used in derivation of the Public Health Toxicity **Reference Values for PFOA and PFOS**

Agency	Intraspecies Differences	Human Variability	Long human half-life	Use of LOAEL	Total
PFOS					
COT (2006a)	10	10	Nil	-	100
EFSA (2008)	10	10	2	-	200
FEA (2006), EWG (2002)	10	10	3	-	300
USEPA (2009)	3	10	13	-	390
MDH (2008)	3	10	n/a <sup>a</sup>	-	30
PFOA					
COT (2006b)	10	10	Nil	-	100
EFSA (2008)	10	10	2	-	200
USEPA (2009)	3	10	81	-	2400
FEA (2006)	10	10	3	10	3000
NC and MDH (MDH 2008)	3	10	n/a <sup>a</sup>		30

Notes: COT = Committee of toxicology, EFSA = European Food Safety Authority, USEPA = United States Environmental Protection Authority, FEA = German Ministry of Health at the Federal Environment agency, MDH = Minnesota Department of Health, NC = State of North Carolina

Clearance was taken into account in benchmark dose modelling used to derive the point of departure



## 5.2 Human Health Criteria for Perfluorinated Compounds

A summary of criteria suitable for screening the PFCs relevant to the current investigation is provided below in Table 6-1. Drinking water criteria from USEPA (2009) are primarily used for screening human health impacts. In the absence of a specific value for 6:2 FTS, the value for PFOS is substituted as a conservative approach for screening risks associated with 6:2 FTS.

Compound	Criteria Name	Criterion Value	Source	Media					
Drinking Water									
PFOS, 6:2 FTS	PHA	0.2 µg/L							
PFOA	PHA	0.4 µg/L	USEPA (2009a)	Matar					
PFOS	MPC <sub>DW,Water</sub>	0.53 µg/L	RIVM (2010)	Water					
PFOS and PFOA									
Recreational Guideline	Recreational Guidelines (Water)								
A factor of at least 10x could be applied to drinking water guidelines for primary contact recreation as dermal exposure to PFC is considered an incomplete/insignificant exposure pathway compared to the oral pathway (NHMRC 2011). This is because PFCs in general have low rates of dermal absorption. (ASTDR 2009), e.g. PFOS criterion = $0.2 \times 10 = 2 \mu g/L$ .									
Direct Contact With So	oil								
PFOS,	SSL	6 mg/kg		Soil/Sodimont					
PFOA	USEPA (20090) Soll/Sediment								
6:FTS SSL 6 mg/kg Assumes same as PFOS <sup>a</sup> Soil/Sediment									
PHA = Provisional Health Advisory, GV = guideline value, SSL = Soil Screening Level, MPC <sub>DW,Water</sub> = Maximum Permissible Concentration in drinking water, a. Note no criteria was identified for 6:2FtS, as result Cardno Lane Piper adopted PFOS criteria value as a screening level only.									

#### Table 6-4: Summary of human health criteria for PFC



References

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Note: The references with asterisks (\*) below have been used to compile the abbreviated toxicity profile however they have not been cited within the profile.

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