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AFFF Decontamination of Aircraft Rescue and Firefighting Vehicles

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AFFF Decontamination of Aircraft Rescue and Firefighting Vehicles

Yoon Ko, Ph.D

Executive Summary

Samples of AFFF (Aqueous Film-Forming Foam) and rinse water effluents were obtained from the trial decontamination procedures conducted by the Department of National Defence (DND) at the Canadian Airforce Base in Greenwood, Nova Scotia in Nov. 2020. The samples were analysed to evaluate the effectiveness of the decontamination procedures mainly for environment and for human health concerns. The ecotoxicity was analysed in collaboration with FireTox LCC.

In the assessment for the ecotoxicity of the foam effluent samples containing various PFAS components, many of which are ecotoxic with potential long-term chronic effects, the Global Harmonized System (GHS) method was used with pre-Total Oxidizable Precursor (TOP) and post-TOP test data. The GHS method found the final sample (2nd Rinse) to be neither an acute nor chronic aquatic hazard while highly ecotoxic fluorinated compounds remained in the final sample (2nd Rinse).

The existing guides and standards for human health consider concentrations of only few PFAS components (e.g. PFOS and PFOA) while foam mixtures/rinse water contain multiple different fluorinated compounds with potential human health impacts. In the final sample (2nd Rinse), the concentration measured for PFOA was much greater than the Australian drinking/recreational water and the Canadian drinking water thresholds.

The lab data resulted from the final sample (2nd Rinse) were also above the threshold values of total PFAS concentrations or PFOA concentrations that are suggested by some stakeholders (i.e. not justified threshold values).

It is concluded that the effectiveness of the trial decontamination procedures was limited. Thus, it is recommended to evaluate potential rebound of PFAS in the tank when replaced with new fluorine free foams or others. The efficiency of decontamination cannot be fully evaluated without the assessment for environment and human health impacts.

1 Introduction

AFFF (Aqueous Film-Forming Foam) is used to extinguish high-hazard flammable liquid fires, such as in chemical plants; flammable liquid storage and processing facilities; aviation operations (aircraft rescue and firefighting, hangars); and military facilities. When applied to a hydrocarbon pool fire, with its ability to form foam blanket and thin film (a layer of surfactant), AFFF effectively suppresses the fire by cooling off and limiting fuel vapour coming through the fuel surface [1].

The surfactant used in AFFF are per- and polyfluoroalkyl substances, such as perfluorooctylsulfonate (PFOS, i.e. one of the most used fluorinated surfactants in foams), perfluorooctanoic acid (PFOA) and perfluorocarboxylic acids (PFCA). These fluorinated surfactants are chemically inert and resistant to degradation. Thus, in 2006 the European Union banned the use of PFOS due to the low biodegradability. In addition, the U.S. Environmental Protection Agency (EPA) classified that PFOS is “likely to be carcinogenic to humans” [2]. In May 2016, EPA also established drinking water health advisories of 70 parts per trillion (70 nanograms per liter (ng/L)) for the combined concentrations of PFOS and PFOA [3].

Due to the environmental and toxicological concerns, most of the fluorinated surfactants have been gradually phasing out of the market since 2009. The Department of National Defence (DND) is also developing a plan to phase-out the use of AFFF in their fire-fighting equipment, and to replace it with an alternative foam (considered to be less harmful). As a first step, DND developed a trial decontamination protocol to remove AFFF concentrate from their aircraft rescue and firefighting (ARFF) vehicles.

The trial decontamination protocol (see Appendix A in Supplement A) that DND developed outlines key cleaning procedures including foam drain; multiple rinse cycles; and rinse water collection as well as disposal. The protocol also defines requirements for sampling of the collected rinse water and effluent analysis of the samples, in order to validate the effectiveness of the decontamination procedures. Samples of the collected effluent would be tested mainly for residual PFAS.

In support of this effort, NRC (National Research Council) has been requested to perform the following three tasks:

- Task 1: Review the DND trial protocol for the decontamination of ARFF vehicles to ensure that it adequately addresses the safety of the personnel and environment.
- Task 2: Review the sampling details and lab analysis plan associated with the testing of the blank sample and effluents collected during the decontamination process.
- Task 3: Review the lab test results, assess the efficacy of the decontamination process and evaluate the ecotoxicity of the decontamination effluents.

NRC submitted a letter report (see Supplement B), which provided reviews of the trial protocol for the decontamination of ARFF vehicles (Task 1) and recommendations for the sampling and lab analysis plan (Task 2). Following the protocol, DND conducted a trial decontamination at the Canadian Airforce Base in Greenwood, Nova Scotia.

This report reviews the lab test results of the collected samples from the DND trial decontamination and discusses the ecotoxicity analysis conducted with the lab data of the samples. Also, it discusses the efficacy of the decontamination process.

2 A summary of the trial decontamination procedures and effluent sampling

To validate the AFFF decontamination protocol for ARFF vehicles, DND conducted the decontamination trial at the Canadian Forces Base Greenwood facility in Nova Scotia, over 4 days from November 23 to November 26, 2020.

2.1 Materials

2.1.1 AFFF

The decontamination trial was conducted on an ARFF vehicle (i.e. E-ONE P1 series, equipped with one 800-liter foam tank and associated foam distribution piping), which contained AFFF concentrate (Ansulite AFC-3DC 3%). The SDS of the AFFF concentrate indicates that the foam concentrate contains Polyfluorinated alkyl betaine up to 10%, which is harmful to humans and the environment for its low degradability. It is also indicated that the foam can cause “serious eye damage” (GHS Health Hazard Code: H318, Category 1) and “mild skin irritation.”

2.1.2 Cleaning agent and water solutions

A cleaning agent, Arcadis V171 was used since unaided water flushing is known to be ineffective to remove residual fluorinated compounds coated on the surfaces of tanks and pipelines. The V171 is transparent liquid solvent containing primarily d-Limonene (less than 65%), acetic acid (less than 40%) and critic acid (less than 30%). D-Limonene is toxic to aquatic life. According to the SDS for the Arcadis V171 Cleaning Fluid, the chemical can cause “serious eye irritation” (GHS Health Hazard Code: H319, Category 2).

Arcadis V171 was mixed with water and used in multiple rinse cycles in Day 2 and Day 3. The DND reported that a total amount of 1100 liters of V171 was used in the trial, but the report does not provide the total amount of water used. It is also noted that the foam tank was pressure-washed two times in Day 1, prior to the use of the cleaning agent.

2.2 Cleaning and sampling procedures overview

The procedures utilized by DND for the trial at Greenwood are provided in Appendix A in Supplement A. It should be noted that the procedures were modified from initial protocol to accommodate specific conditions and constraints at the site. Table 1 summarizes the procedures of rinse cycles, 1 blank (AFFF) sampling and 6 effluent samplings.

Table 1. Overview of the on-site decontamination procedures

Procedures	Note	Sampling	Schedule
Preparation	<ul style="list-style-type: none"> Positioned a vehicle over berm for accidental foam release Collected a foam sample directly from the vehicle tank for a laboratory analysis testing. 	AFFF	Day 1
AFFF drain	<ul style="list-style-type: none"> Drained the AFFF from the foam tank and the pipelines into totes. 		
Pre-water rinse	<ul style="list-style-type: none"> Pressure-washed with water Collected a water rinse sample 	Water Rinse (pre-V171)	
Cleaning 1	<ul style="list-style-type: none"> Pumped cleaning agent and water solution into the foam tank with 650 L of V171 and 150 L of water (note: dilution was not suggested by the manufacturer). Circulated the solutions for 2.5 hours (Note: the manufacture recommended 4 hours) Collected an effluent sample for a lab analysis Drained partially and refilled the foam tank. 	First Flush (V171)	Day 2
Soak	<ul style="list-style-type: none"> Commenced an overnight soak 		
Cleaning 2	<ul style="list-style-type: none"> Drained after an overnight soak Collected an effluent sample Partially drained and refilled with V171 (200 L) Rinsed the foam tank with vehicle water (Note: the vehicle water appeared cross-contaminated from the foam). Thus, filled and drained with mixed solution of clean and used V171 two times. 	20-hour Soak (V171)	Day 3
Water Rinse 1	<ul style="list-style-type: none"> Rinsed the foam tank with water 		
Water Rinse 2	<ul style="list-style-type: none"> After the first water flush, collected an effluent sample 	1st Rinse (Water)	
Soak	<ul style="list-style-type: none"> Refilled the foam tank and let it sit over night 		
Water Rinse 3	<ul style="list-style-type: none"> Drained the tank of water Collect an effluent sample for a lab analysis. Power-washed the foam tank Collect an effluent sample for a lab analysis. Cleaned-up 	2nd Rinse (Water) (i.e. the final sample) ¹ Final Rinse (Water) ²	Day 4

¹ DND indicated that the 2nd Rinse sample is the final sample and need to be used for Ecotoxicity analysis.

² DND indicated that the Final Rinse sample was mislabeled and is more likely to be the final rinse sample taken after the V171 application, rather than the final rinse sample taken at the end of the test cycle.

3 Limonene analysis

The cleaning agent/solvent V171 was used in Day 2 and Day 3 for cleaning and decomposing PFAS that was presumed to coat the surfaces of the tank and piping. To ensure the AFFF decontamination procedure was environmentally safe, selected effluent samples were analysed for D-limonene, which was identified as a potentially ecotoxic chemical component of the cleaning agent V171.

3.1 Method

D-limonene is identified as a primary chemical component of the cleaning agent V171. To ensure that the decontamination procedures was not environmentally harmful, the selected effluent samples were collected and sent to a testing lab (ALS Environmental) for d-limonene concentrations. D-Limonene concentrations were evaluated for the following three samples;

- Pure AFFF sample,
- 1st Rinse (water) sample and
- Final Rinse sample.

Following the testing lab's standard procedures, extracted d-limonene from the samples were analysed using Gas Chromatography/Mass Spectrometry (GC/MS) to quantify the concentrations.

3.1.1 Limonene eco-toxicity

The Lethal Concentration 50 (LC50) value for fish and Effective Concentration 50 (EC50)³ values for water fleas and freshwater algae are less than 1 mg/L (1,000,000 ng/L) for d-limonene (Section 12 of the SDS). These values are measures of the aquatic ecotoxic potency of d-limonene. A component with an L(E)C50 less than or equal to 1 mg/L (1,000,000 ng/L) for either fish, water fleas, or algae, is classified as Acute 1 under the Global Harmonized System (GHS). Acute 1 classification means that the substance can be "Very Toxic to aquatic life" at certain concentrations.

3.2 Limonene Results

Figure 1 presents the concentrations of limonene in the three investigated samples. The results show that the AFFF blank sample contained less than 10,000 ng/L (or < 10 ppb) of d-limonene and that the Final rinse sample contained less than the blank. Moreover, the concentration of d-limonene present in the Final rinse⁴ was significantly less than the L(E)C50 value. Based upon the above findings, it can be concluded that the highest possible concentration of d-limonene remaining inside the tank and piping does not present an aquatic ecotoxic risk.

³ The concentration of a chemical substance in water which results in death in 50% of the fish or algae exposed.

⁴ DND indicated that the Final Rinse sample was mislabeled and is more likely to be the final rinse sample taken after the V171 application.

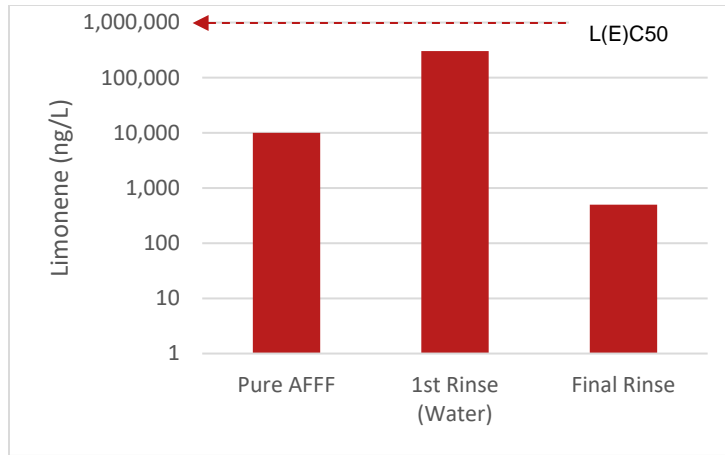


Figure 1 Limonene results

4 PFAS analysis

As per the protocol, the collected rinse water and effluent samples were tested for the level of PFAS to validate the effectiveness of the decontamination procedures.

4.1 Methods

As suggested in NRC review letter (see Supplement B), the following EPA methods and the TOP assay are used in the analysis of fluoridated substances.

- Method 533: “Determination of Per- and Polyfluoroalkyl Substances in Drinking Water by Isotope Dilution Anion Exchange Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)” [4]
- Method 537.1: “Determination of Selected Per- and Polyfluorinated Alkyl Substances in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)” [5]
- Method of the Total Oxidizable Precursor (TOP) assay: To fully detect and measure PFAS compounds not measurable by the EPA methods of 533 and 537.1.

The samples were analyzed by SGS AXYS Analytical Services following their MLA-110 method, which measures “40 PFAS compounds to cover all EPA 537.1, EPA 533, and several additional precursors”, according to their website. To detect PFAS substances including precursor compounds that are not measurable by regular analytical methodology of LC-MS/MS, the samples were re-analyzed by the TOP assay (following SGS AXYS MLA-111 method). The oxidation step (i.e. hydroxyl radical-based) in the TOP assay converts the precursor compounds (i.e. Polyfluorinated compounds) to detectable perfluoroalkyl acids (PFAA). The TOP assay provides a quantitative estimate for the amount of total PFAS in a sample. Thus, the pre-TOP and post-TOP analyses were conducted for the following seven samples;

- o Pure AFFF sample,
- o Water Rinse (pre-V171)
- o First Flush (V171)
- o 20-hour Soak (V171)
- o 1st Rinse (Water)
- o 2nd Rinse (Water) (i.e. the final sample)

4.2 Results

The pre-TOP analysis was conducted with the six samples for 40 fluorinated compounds, and, among them, 16 components were detected, which include PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFNA and PFOS. Among the 16 detected components, 11 components are analysed by the TOP assay. The TOP assay increased the concentrations of all component significantly. As such, the post-TOP concentrations (Figure 3) are found to be higher than the pre-TOP concentrations (Figure 2) for all of the PFAS substances measured.

The concentration of each fluorinated compound is plotted for each rinse cycle in Figure 2 (pre-TOP results) and Figure 3 (post-TOP results). In both pre- and post-TOP results, the concentrations in the AFFF sample were initially high, yet they dropped significantly after the first water rinse. Since PFAS substances tend to adhere to the tank and distribution piping surfaces, the cleaning agent (the V171) was used to assist in breaking down and removing the PFAS from these surfaces.

As shown in the lab analysis results, the PFAS concentrations in the effluent samples of First Flush (V171) and 20-hour Soak (V171) were higher than those in the Water Rinse (pre-V171) sample. The results imply that the cleaning agent was more effective than water in dissolving the PFAS adhered on the surfaces, but not to the

extent of significant removal of the PFAS. The PFAS concentrations decreased in the following cycles (1st Rinse (water) and 2nd Rinse (water) samples).

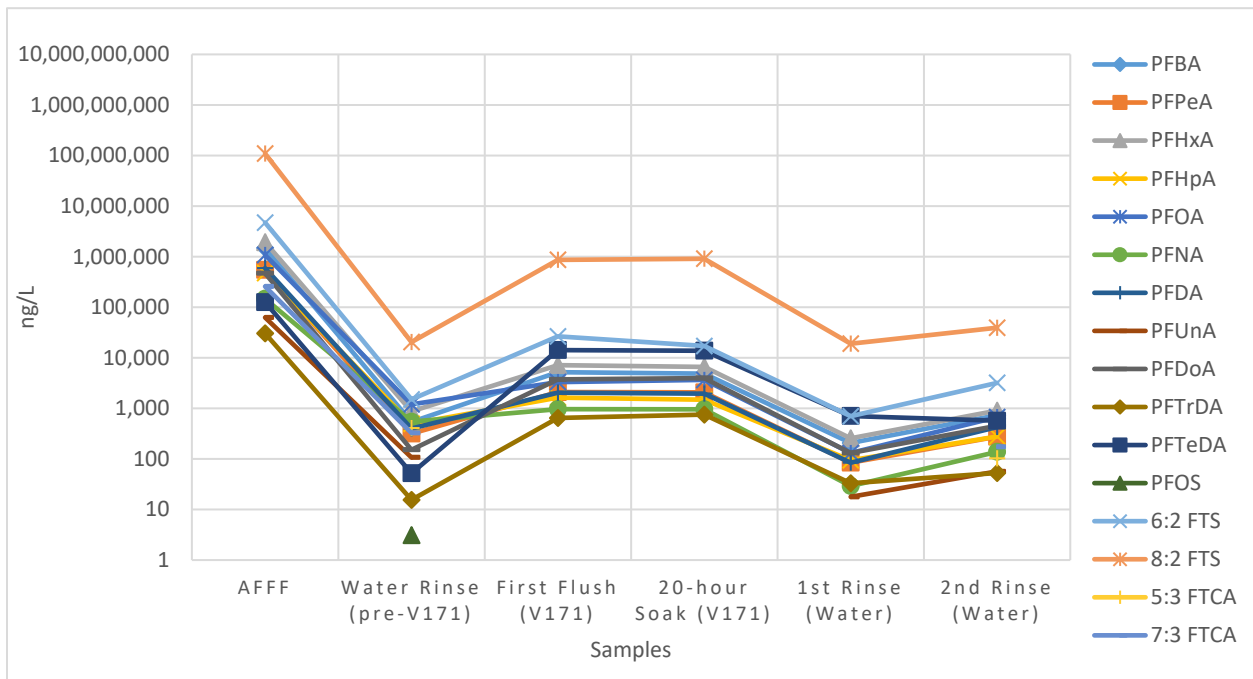


Figure 2 Pre-TOP results

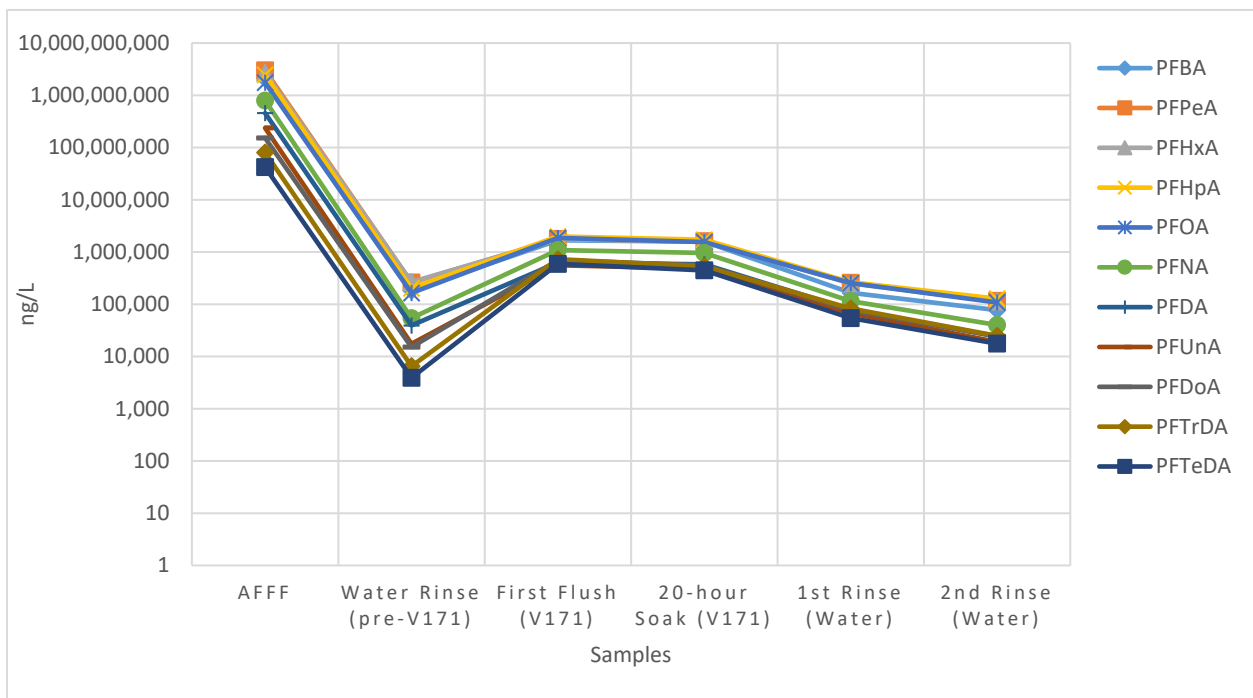


Figure 3 Post-TOP results

Figure 4 compares the compositions of the fluorinated compounds analysed by the post-TOP assay for the samples taken at each rinse cycle. While the AFFF sample and Water Rinse (pre-V171) sample show similar composition, considerable change is observed in the compositions of the samples obtained after applying the cleaning agent V171. For unknown reasons, the concentrations of DFTeDA, DFTTrDA and DFTDoA increased after rinsing with the cleaning agent, and these fluorinated compounds are identified as highly ecotoxic compounds (for details, see Section 5.1.1).

Figure 5 compares the total PFAS concentrations measured for the samples taken at various rinse cycle, as analyzed by post- and pre-TOP assays. For the AFFF sample, it should be noted that the post-TOP PFAS evaluated concentration seems relatively low in comparison to the manufacturer's info on the chemical compositions of the AFFF. The SDS of the AFFF concentrate indicates that the foam concentrate contains Polyfluorinated alkyl betaine up to 10% while the post-TOP assay resulted in 1.3% (13,341,900,000 ng/L) of the total PFAS in the AFFF⁵.

While the cleaning procedures reduced the total PFAS significantly, the total PFAS in the final sample (2nd Rinse) was over 690,000 ng/L (for post-TOP). Assuming that residual PFAS levels in the tank and pipelines are similar to the measured total PFAS concentration in the Final sample (2nd Rinse), it is concluded that the effectiveness of the decontamination procedure was limited.

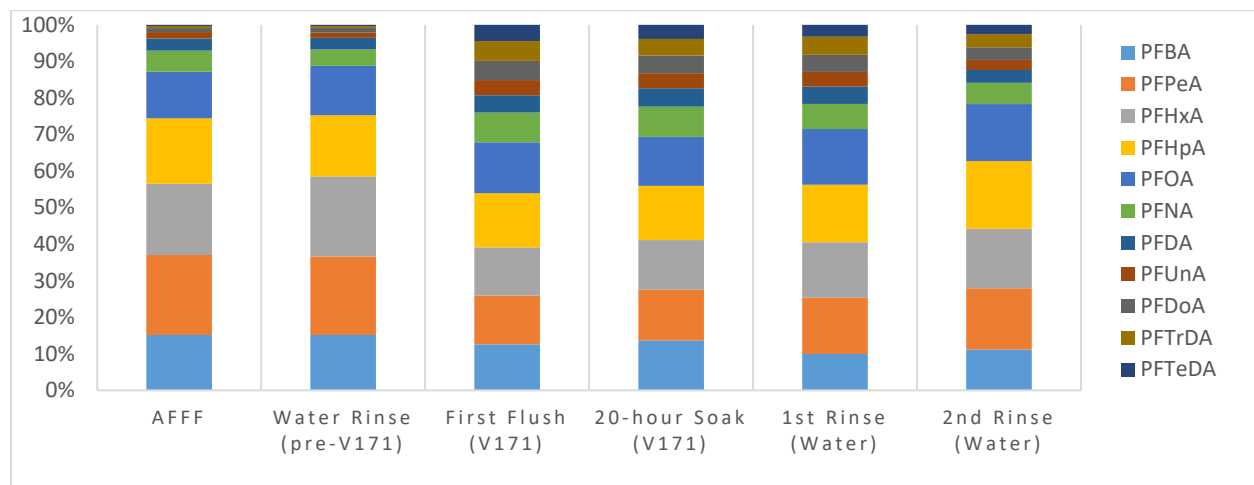


Figure 4 Compositions of fluorinated compounds (post-TOP)

⁵ The AFFF sample was directly obtained from the tank and might have been diluted. The DND decontamination procedures (provided in Appendix A in Supplement A) indicates potential cross contamination between the water tank and foam tank in the truck.

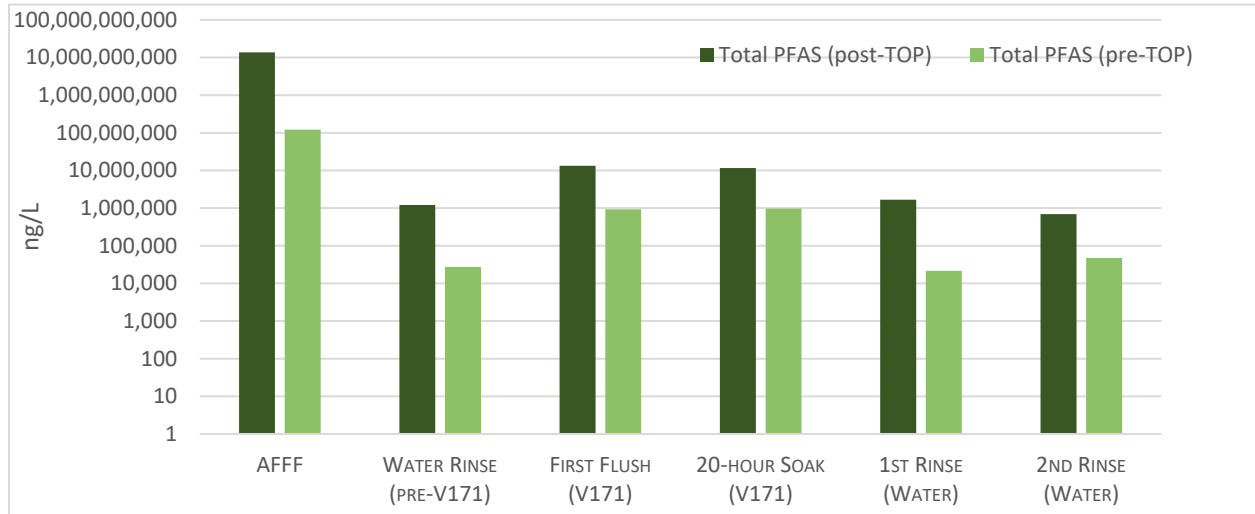


Figure 5 Total PFAS concentrations

5 Ecotoxicity Analysis

With the pre-TOP and post-TOP concentrations data, the collected samples are analysed in collaboration with FireTox, LLC. for the ecotoxicity. This section summarizes the analysis methods and results. Detailed analysis is provided in Supplement A: Ecotoxicity analysis reported by FireTox, LLC.

5.1 Methods

In order to understand the ecotoxic potency of the effluents collected from the decontamination trial, the lab data of the effluent samples are analyzed following the GHS (Globally Harmonized System) environmental hazard classification scheme [6]. The GHS hazard classification methodology was adopted by the United Nations in 2003 to unify the approach used across the world to classify the environmental, physical and health hazards of chemicals. The Canadian Workplace Hazardous Materials Information Systems (WHMIS) adopted the GHS in 2015 for physical and health hazard. The GHS environmental hazard classification scheme focuses on the aquatic impact of chemicals since aquatic organisms, as in the final receiving environment, are impacted by many harmful substances. In addition, ecotoxicity data other than aquatic toxicity data are rarely available.

To compare the ecotoxicity of different types of foams, the NRC study conducted in 2020 [1] used the GHS methodology since the method enabled systematic comparisons of different AFFF and fluorine free foams, taking into account various ecotoxic aspects of firefighting foams, such as acute, chronic, and potential for bioaccumulation and degradation.

The GHS method classifies substances and chemical mixtures into 3 acute hazards and 4 chronic hazard categories [6] based on the collected ecotoxicity data, as shown in Table 1.

Table 1 GHS hazard category, code, and statement [6]

Category	Hazard Code	Hazard Statement
Acute 1	H400	"Very toxic to aquatic life"
Acute 2	H401	"Toxic to aquatic life"
Acute 3	H402	"Harmful to aquatic life"
Chronic 1	H410	"Very toxic to aquatic life with long lasting effects"
Chronic 2	H411	"Toxic to aquatic life with long lasting effects"
Chronic 3	H412	"Harmful to aquatic life with long lasting effects"
Chronic 4	H413	"May cause long lasting harmful effects to aquatic life"

To classify the hazards of the effluent samples, which were mixtures of many chemical substances including toxic fluorinated compounds, ecotoxicity analysis was conducted following the steps below;

- First, classify each fluorinated component following the GHS hazard classification method. The GHS acute hazard category of a chemical is determined using the corresponding lethal concentration and/or effective concentration (e.g. LC50, EC50)⁶. The GHS chronic hazard category for each is determined mainly using its Lowest Observed Effect Concentration (LOEC) or No Observed Effect Concentration (NOEC), or ChV value (i.e. the geometric mean of the NOEC and LOEC)⁷.
- Second, classify the hazard of a sample (i.e. mixture of toxic fluorinated compounds) following the GHS summation method.

5.1.1 Toxicity of each fluorinated compound

As the first step, for each of the 16 fluorinated compounds detected, LC50 or LE50 values were searched from ECOSAR⁸ or ECOTOX⁹ database. To identify the concentration level of each fluorinated compound relative to the LC50 or LE50, the percent concentration of each component relative to the corresponding lowest value of LC50 or LE50 is calculated. The post-TOP concentration data are used, except for the compounds without post-TOP data.

As shown in Figure 6, the concentrations of most fluorinated compounds detected in the AFFF sample are significantly above the lowest L(E)C50 (lowest value of LC50/LE50).

Also in the final sample (2nd Rinse), the concentrations of PFTeDA, PFTrDA and PFOA remained higher than or close to their lowest L(E)C50 values. These concentrations of a chemical substance in water which results in death in 50% of the exposed fish/algae. Therefore, the level of ecotoxicity of the final sample (2nd Rinse) effluent calls for cautions.

⁶ The ecotoxicity for fish and algae are reported as Lethal Concentration (LC) and Effective Concentration (EC), respectively. The LC50 value represents the concentration of a chemical substance in water which results in death in 50% of the fish exposed for typically 96 hours. The EC50 value is the concentration of a substance that results in crustacea immobilization or a 50% reduction in algae growth rate [6], for 48-96 hour exposure. These species are considered as surrogate for all aquatic organisms.

⁷ Chronic toxicity is reported as the Lowest Observed Effect Concentration (LOEC) or the No Observed Effect Concentration (NOEC). The LOEC is the lowest concentration tested that results in a statistically significant adverse impact when compared to the control. The NOEC represents the highest concentration, immediately below the LOEC, showing no statistically significant adverse impact when compared to the control. In some cases, a Chronic Value (ChV) may be reported [6].

⁸ Ecological Structure Activity Relationships

⁹ The ECOTOXicology Knowledgebase

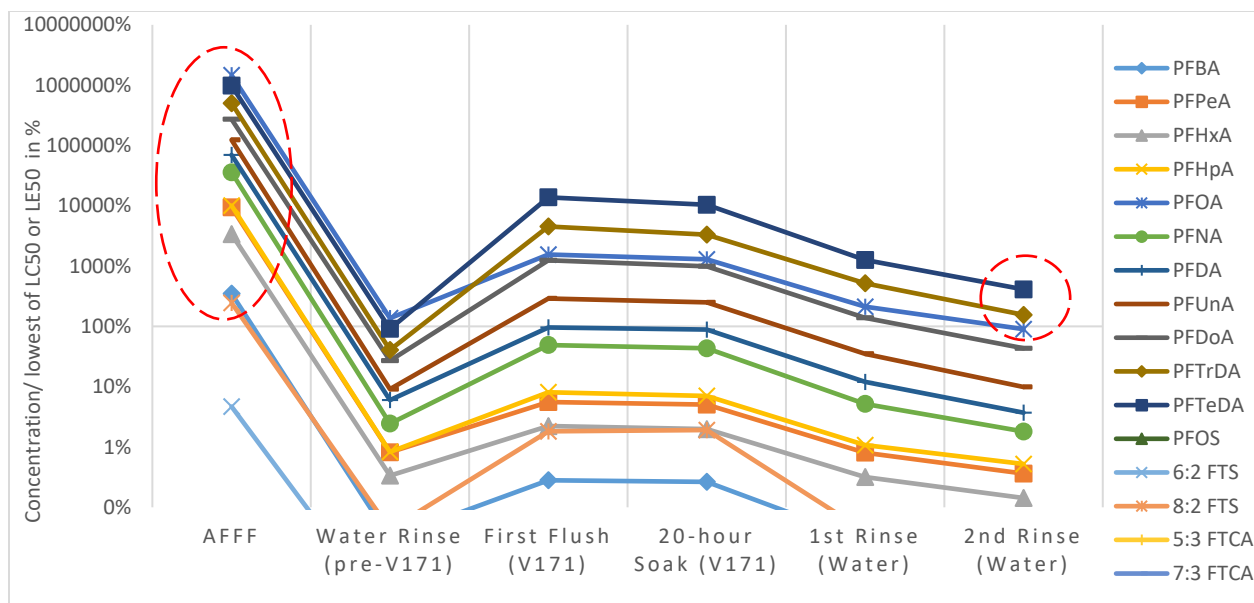


Figure 6 Percent concentrations of compound relative to the corresponding lowest LC50 or LE 50 (Post-TOP concentration data are used except for PFOS, 6:2 FTS, 8:2 FTS, 5:3FTCA and 7:3 FTCA)

Acute Toxicity

The acute toxicity of each fluorinated compound was classified to understand the potential impact on an organism in a short-term aquatic exposure. Based on the lowest value of LC50/LE50, each compound was classified for its acute toxicity as per the GHS method (see Table 4.1.1(a) in Appendix C of Supplement A). The classified hazard category for each fluorinated compound is summarized in Table 2. Among the detected 16 compounds, 14 compounds fall into 'Acute' categories, and 6 compounds including PFOA are identified as 'Acute 1 category' (i.e. very toxic to aquatic life). It should be noted that those (i.e. PFTeDA, PFTrDA and PFOA) identified with relatively high concentrations in the final sample (2nd Rinse) are classified into 'Acute 1'.

In addition, the L(E)C50 value of PFTeDA is notably low, the order of magnitude of which in ng/L is 3 while that of PFOA, PFDA and PFUnA is 5. The low L(E)C50 value indicates highly toxic potency even with a low concentration. To account the effect of highly toxic components, the GHS summation method to classify hazard categories of a mixture assigns a M-factor (Multiplication factor) for each component reflecting the L(E)C50 value. Thus, high M-factors are identified for PFDoA, PFTrDa and PFTeDa, as in Table 2.

Chronic Toxicity

To classify the chronic toxicity of each fluorinated compound, the NOEC and ChV values were searched from ECOSAR and ECOTOX database. The NOEC values represent the highest concentration with no statistically significant adverse impact when compared to the control, values above which are considered to negatively affect aquatic organism in relation to the life cycle following long-term exposure in the aquatic environment. As compared in Table 2, the concentrations of PFOA, PFNA, PFDoA, PFTrDa and PFTeDA measured in the final sample (2nd Rinse) are much higher than the corresponding lowest values of NOEC or ChV (i.e. These compounds are also very toxic to aquatic life with long lasting effect). Thus, the final sample (2nd Rinse) effluent poses chronic ecotoxic potency.

Based on the lowest value of NOEC or ChV, each compound was classified for its chronic toxicity as per the GHS method (see Table 4.1.1(b)(i) in Appendix C of Supplement A). As summarized in Table 2, among the 16

compounds, 10 compounds fall into 'Chronic' categories, and 8 compounds including PFOA are identified as 'Chronic 1 category' (i.e. Very toxic to aquatic life with long lasting effects). Those (i.e. PFTeDA, PFTrDA, and PFOA) identified for their relatively high concentrations in the final sample (2nd Rinse) are classified into 'Chronic 1' as well as 'Acute 1'.

Also, following the GHS method, M-factors are assigned based on the magnitude of NOEC or ChV values of each component, as shown in Table 2. High M-factors are identified for PFNA, PFTrDa and PFTeDa, indicating high chronic toxic potency. In addition, as presented in Figure 4, compositional changes in the effluent samples were observed with increased concentrations of the highly chronic and acute components (e.g. PFTeDA, PFTrDA and PFTDoA) after applying the cleaning agent.

Table 2 Hazard category of fluorinated compounds (Post-TOP assay)

Substance	AFFF** (ng/L)	Final sample (2nd Rinse) (ng/L)	GHS Acute			GHS Chronic		
			Lowest L(E)50 (ng/L)	GHS Acute Category	M-factor*	Lowest NOEC or ChV (ng/L)	GHS Chronic Category	M-factor*
PFBA	2,080,600,000	77,200	597,000,000	NAT		76,800,000	NCT	
PFPeA	3,007,600,000	116,000	250,000,000	Acute 3		30,000,000	NCT	
PFHxA	2,678,000,000	113,000	79,300,000	Acute 3		6,280,000	NCT	
PFHpA	2,461,700,000	128,000	24,500,000	Acute 3		4,150,000	NCT	
PFOA	1,751,000,000	108,000	7,440,000	Acute 1	1	100,000	Chronic 1	1
PFNA	795,160,000	40,100	2,220,000	Acute 2		8,000	Chronic 1	10
PFDA	456,290,000	24,300	656,000	Acute 1	1	100,000	Chronic 1	1
PFUnA	236,900,000	19,000	192,000	Acute 1	1	36,000	Chronic 1	1
PFDoA	152,440,000	24,200	56,000	Acute 1	10	11,000	Chronic 1	1
PFTrDA	80,340,000	24,800	16,000	Acute 1	10	3,100	Chronic 1	10
PFTeDA	42,127,000	17,600	4,300	Acute 1	100	890	Chronic 1	100
PFOS*	ND	ND	16,900,000	Acute 3		5,000	Chronic 1	
6:2 FTS*	(5,051,740)	(3,190)	521,000,000	NAT		62,800,000	NCT	
8:2 FTS*	(116,918,000)	(39,300)	47,700,000	Acute 3		7,770,000	NCT	
5:3 FTCA*	ND	(103)	NR	Acute 3		-	Chronic 3	
7:3 FTCA*	ND	(107)	NR	Acute 2		-	Chronic 2	
Total PFAS	13,742,157,000	692,200						

**The values for the AFFF samples are converted from the lab data reported in ng/g to ng/L (using a relative density 1.03). The SDS for the Ansilite 3% AFFF (Product ID: 431441) does not report a density. A similar Ansilite 3% AFFF composition (Product ID: 446894) had a reported density of 1.03 g/ml.

ND = Not Detected, NAT= No Acute Toxicity, NCT = No Chronic Toxicity, NR=Not Reported, M-factors for Acute 1 category substances were determined as outlined in the GHS method.

The test data for the samples are obtained by the post-TOP assay, except the components marked with*.

Total PFAS concentrations were calculated with the post-TOP data only.

5.1.2 Toxicity summation to classify effluent samples

To assess the toxicity of the effluent samples collected from the decontamination trial, the GHS summation method [6] is used. Based on the classified category of each component, the toxic effects of the fluorinated components are summed up using the GHS additive calculation method shown in Table 3. The calculation method takes into account each component's concentration (in %), hazard category, and M-factors (i.e. determined based on the magnitude of its toxic potency). Applying M-factor gives an increased weight to the highly toxic components of PFNA, PFDoA, PFTrDa and PFTeDa.

Table 3 GHS Summation Method for Category Classification of Mixtures [6].

Category	Summation of Ingredients
Acute 1	If Acute 1 % x M \geq 25%
Acute 2	If (M x 10 x Acute 1 %) + Acute 2% \geq 25%
Acute 3	If (M x 100 x Acute 1 %) + (10 x Acute 2 %) + Acute 3 % \geq 25%
Chronic 1	If Chronic 1 % x M \geq 25%
Chronic 2	If (M x 10 x Chronic 1 %) + Chronic 2% \geq 25%
Chronic 3	If (M x 100 x Chronic 1 %) + (10 x Chronic 2 %) + Chronic 3 % \geq 25%
Chronic 4	If Chronic 1 % + Chronic 2 % + Chronic 3 % + Chronic 4 % \geq 25%

The post-TOP PFAS concentrations are used in the acute and chronic ecotoxicity analysis for two samples of the AFFF and final sample (2nd Rinse). The results are summarized in Table 4. The AFFF sample (Ansulite 3% AFFF foam mixture) would be classified as Acute 3 (“Harmful to aquatic life”) and Chronic 3 (“Harmful to aquatic life with long lasting effects”). The GHS method [6] requires a calculated concentration equal to or greater than 25% to fall into each level of hazard category (see Table 3). Given that the total PFAS was only 1.37% (13,742,157,000 ng/L) in the AFFF sample, it can be understood that the PFAS components in the AFFF are highly ecotoxic.

However, the final sample (2nd Rinse) was found to be neither an acute nor chronic aquatic hazard.

Table 4 Acute and chronic ecotoxicity of the AFFF and Final sample (2nd Rinse)

Acute Ecotoxicity Category of Mixture		Chronic Ecotoxicity Category of Mixture	
AFFF sample	Final sample (2nd Rinse)	AFFF sample	Final sample (2nd Rinse)
Acute 3	NAT	Chronic 3	NCT
Summation 91.46%	Summation 0.0241%	Summation 155.64 %	Summation 0.0258%

NAT = No acute toxicity, NCT = No chronic toxicity

6 Discussion

6.1 Success criteria

The goal of the decontamination procedure was to remove aqueous film forming foam (AFFF) and associated per- and polyfluoroalkyl substances (PFAS) from an aircraft rescue and firefighting vehicle (ARFF). The effectiveness of the decontamination procedure should be evaluated with measurable indicators for the following goals;

- Effectively remove environmentally harmful PFAS contaminants from the ARFF vehicle and apparatus to the extent that ensures no acute and chronic ecotoxicity.
- Effectively remove health hazardous contaminants from the ARFF apparatus to ensure that residual PFAS levels in the ARFF apparatus do not pose health hazards.

6.1.1 Assessment for environmental impacts

The GHS method was used in assessing the ecotoxicity of the final sample (2nd Rinse), and the final sample was found to be neither an acute nor chronic aquatic hazard. However, the ecotoxic analysis with PFAS concentration data brought attentions to the following key findings;

- The final sample (2nd Rinse) contained all of the 16 fluorinated compounds detected in the AFFF sample, which include highly toxic fluorinated compounds (e.g. PFTeDA, and PFTrDA) with their concentrations in the final sample (2nd Rinse) remained greater than their L(E)C50 values. It should be also pointed out that the compositions of those highly toxic fluorinated compounds increased in the effluent samples after applying the cleaning agent of V171.
- The post-TOP assay provided a quantitative estimate for the amount of total PFAS in a sample by converting the precursor compounds to detectable compounds.
- The total PFAS evaluated for the AFFF sample was only 1.3%, which appears relatively low compared to the chemical compositions of the AFFF (i.e. The SDS of the AFFF concentrate indicates that the foam concentrate contains Polyfluorinated alkyl betaine up to 10%).
- The results of the ecotoxic analysis in this study is specific to the trial decontamination since the results are affected by many variables, such as the chemical composition of the AFFF (i.e. vary depending on manufacturer and batch), the amounts of the cleaning agent applied, and the amounts of water used in each rinse cycle as well as the residual build-up of PFAS in a ARFF vehicle and piping.

6.1.2 Assessment for health effects

Since the effect of PFAS on human is different from that on aquatic organisms, the ecotoxicity analysis does not address the human health hazards of PFAS substances. While studies demonstrated possible PFAS effects on human health (e.g. potential damage to the liver and the immune systems as well as birth defect, newborn death [2]), the adverse effect of PFAS on human health is not yet fully understood.

This study found that the concentrations of highly toxic fluorinated compounds in the final sample (2nd Rinse) remained greater than their L(E)C50 and NOEC/ChV values, which are in general significantly greater than PFAS human health hazard thresholds. Therefore, the human health hazards of the final sample (2nd Rinse) cannot be ruled out while the PFAS in the final sample (2nd Rinse) was not found to present a hazard to aquatic organisms. Given that the majority of the PFAS substances found in the Post-TOP analysis are not rapidly degradable, these chemicals could make their way into water and food sources for humans.

In particular to ARFF vehicles, firefighters can be exposed to residual PFAS components in the tank and pipelines, which could be present even in new replacement fluorine free foams.

For the thresholds of concern in protecting firefighters and workers who operate contaminated ARFF vehicles, consideration is given to existing PFAS health standards and guides with threshold concentrations of PFAS for drinking water and recreational water.

- For drinking water, Canada has limits of 200 ng/L for PFOA and 600 ng/L for PFOS [7]. Canadian local water source is reported to have PFOA concentration of 0.799 ng/L. US EPA established in 2016 health advisories of 70 ng/L for the combined concentrations of PFOS and PFOA [3]. Australian PFAS Health Based Guidance values for drinking water are 560 ng/L for the concentration of PFOA and 70 ng/L for the combined concentrations of PFOS and PFHxS [8].
- For recreational water, Australian PFAS Health Based Guidance values are 10,000 ng/L for the concentration of PFOA and 2,000 ng/L for the combined concentrations of PFOS and PFHxS [8].
- These threshold values for human health are much lower than those used in evaluating ecological hazards. As such, the post-TOP results from the final sample (2nd Rinse) surpassed all of the drinking water and recreational water thresholds. In Figure 7, the post-TOP data from the final sample (2nd Rinse) are plotted to compare with the threshold values for human health. While PFOS was not detected from the final sample (2nd Rinse), the concentration measured for PFOA is approximately 11 and 540 times greater than the Australian recreational water and the Canadian drinking water thresholds, respectively.

6.1.3 Assessment for cleaning procedure efficiency

The cleaning efficiency is suggested to be assessed during the decontamination process and after the process. Thus, two goal standards might be identified;

- During the decontamination process, the cleaning efficiency can be measured by reduction in PFAS concentration in the final sample (2nd Rinse) effluent or recovery rate of PFAS throughout the cleaning process.
- After the process, the cleaning efficiency should be confirmed by reaching tolerable maximum residual PFAS concentrations in the replacement foam.

The efficiency of the decontamination cleaning procedure can be generally assessed by monitoring total PFAS concentrations measured by the post-TOP assay. The GHD report [9] suggests the use of the recreational water limits for ARFF vehicle cleaning criteria since primary types of exposure to a firefighter would be dermal and not through ingestion. In addition to the recreation water limits of 2,000 ng/L for PFOS and PFHxS; and 10,000 ng/L for PFOA, GHD suggests that all other components excluding PFOS, PFHxS and PFOA should not exceed 5000 ng/L. GHD further suggests a re-cleaning trigger criterion of 15,000 ng/L for a total PFAS concentration (presumably including PFOS, PFHxS, and PFOA) [9].

As shown in Figure 7, the measured PFOA and total PFAS concentrations in the final sample (2nd Rinse) were much higher than these values. The post-TOP PFOA concentration was 108,000 ng/L in the final rinse, and the Post-TOP total PFAS concentration (all other components excluding PFOS, PFHxS and PFOA) was 584,200 ng/L in the final sample (2nd Rinse). The post-TOP total PFAS concentration was 692,200 ng/L.

The Wood report published in 2018 [10] includes the discussions made during a stakeholder workshop, and it noted that achieving a total PFAS concentration lower than 100,000 ng/L might be possible if numerous cleaning cycles were adopted in decontamination processes (e.g. 32 stages of cleaning achieved PFAS levels below 1000 ng/L).

The DND trial decontamination procedure resulted in a post-TOP Total PFAS concentration of 692,200 ng/L for the final sample (2nd Rinse), which is slightly lower than the level (10,000,000 ng/L) that one stakeholder reported to use as a criterion for replacing with fluorine-free foams in the Wood report. To gauge the level of contamination in replacement fluorine free foams, Queensland (Australia) regulations, allows up to 10,000,000 ng/L for a combined concentrations of PFOA/PFHxS and 50,000,000 ng/L for a concentration of PFOA related precursors and higher homologues in non-fluorinated foam. The PFOA concentration of 108,000 ng/L resulted from the DND trial decontamination procedures for the final sample (2nd Rinse) sits below the Queensland PFOA limit for replacement fluorine free foams.

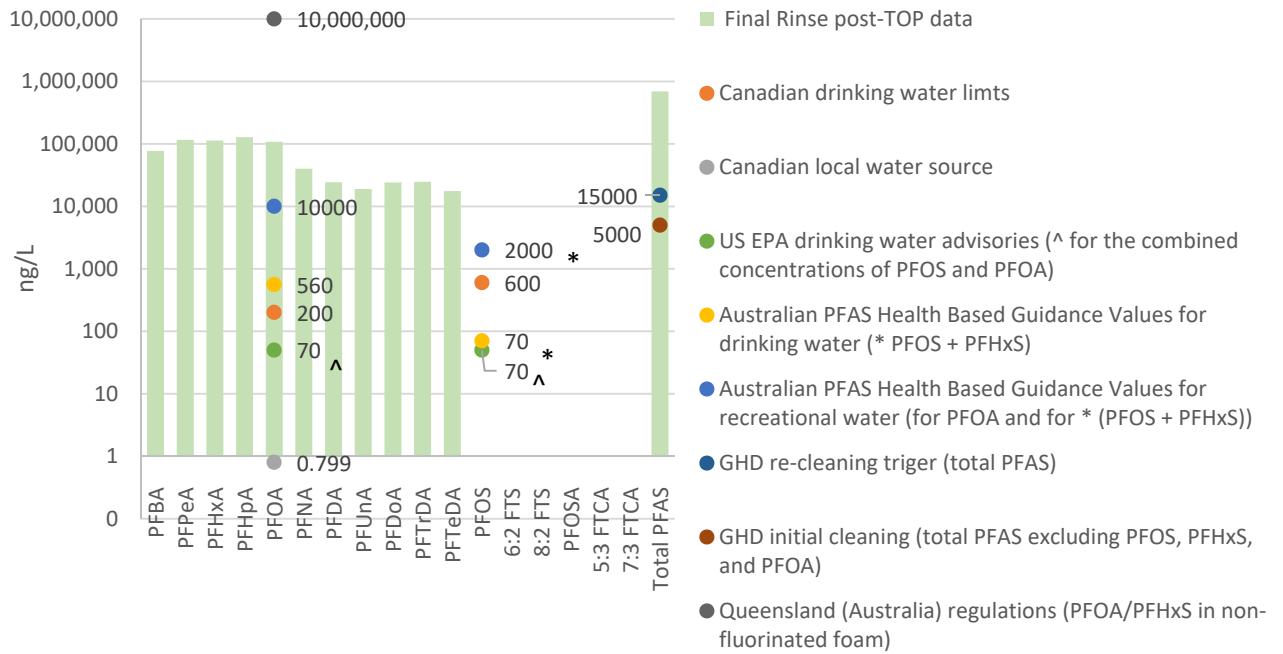


Figure 7 Post-TOP data of the final sample (2nd Rinse) in comparison with PFAS threshold concentrations

7 Conclusions

Assessment for Environmental impacts

The GHS classifications of a mixture for short-term (acute) and long-term (chronic) aquatic environmental hazard are used as indicators for the ecotoxicity of the final sample (2nd Rinse) effluent. The GHS methodology used in this trial provided systematic assessment of the collected samples, taking into account toxic aspects of AFFF foams, which include acute, chronic, and potential for bioaccumulation and degradation. To obtain data necessary for the GHS classification, the post-TOP assay was appropriate since the post-TOP analysis enabled to detect PFAS substances including precursor compounds that are not measurable by regular analytical methods.

The AFFF and effluent samples collected from the trial decontamination were analyzed using the pre-TOP and post-TOP assays. 16 fluorinated compounds were detected in all samples, which include the components categorized into 'Chronic 1 (i.e. Very toxic to aquatic life with long lasting effects)' and 'Acute 1 categories (i.e. very toxic to aquatic life)'. Using the GHS methodology, the AFFF sample was analyzed as Acute 3 ("Harmful to aquatic life") and Chronic 3 ("Harmful to aquatic life with long lasting effects") whereas the final sample (2nd Rinse) was found to be neither an acute nor chronic aquatic hazard.

In addition, highly ecotoxic fluorinated compounds were identified by the GHS method. The concentrations of highly toxic fluorinated compounds (e.g. PFTeDA and PFTrDA) in the final sample (2nd Rinse) remained greater than their L(E)C50 values.

Assessment for human health

While assessment for human health is outside the scope of this study, the existing PFAS drinking water and recreational water thresholds were considered. In the final sample (2nd Rinse), the concentration measured for PFOA was much greater than the Australian drinking/recreational water and the Canadian drinking water thresholds.

The existing guides and standards for human health consider concentrations of only few PFAS components (e.g. PFOS and PFOA) while foam mixtures/rinse water contain multiple different fluorinated compounds with potential human health impacts. Thus, it is quite questionable if the existing guides and standards are sufficient to be adopted to assess the efficiency of the AFFF decontamination procedures pertaining to human health.

The efficiency of decontamination procedure

While the cleaning procedures reduced the total PFAS significantly, the total PFAS in the final sample (2nd Rinse) was over 690,000 ng/L (for post-TOP). Assuming that residual PFAS levels in the tank and pipelines are similar to the measured total PFAS concentration in the final sample (2nd Rinse), it is concluded that the effectiveness of the trial decontamination procedures was limited.

There are criteria suggested to gauge the efficiency of the decontamination cleaning procedure with values of total PFAS concentrations or PFOA concentrations. The lab data resulted from the final sample (2nd Rinse) were also above these suggested values.

The efficiency of decontamination procedures could be generally assessed by monitoring reduction in the concentration of total PFAS or a certain component. However, the efficiency of decontamination cannot be fully evaluated without the assessment for environment and human health impacts. In addition, as demonstrated in the ecotoxicity analysis, rinse water/effluents contain various fluorinated components so that it is important to identify highly toxic fluorinated compounds and their contributions to the overall environmental and health impacts, to ensure effective decontamination.

In addition, recovery rates of PFAS concentration throughout the cleaning process could also be monitored to ensure the cleaning efficiency (i.e. this requires to monitor the volume of water used at each cleaning cycle).

More importantly, it is recommended to evaluate potential rebound of PFAS in the tank when replaced with new fluorine free foams since the goal of the decontamination of ARFF vehicles is to lower the AFFF toxicity level in the vehicle to protect the environment and firefighters operating the equipment.

8 References

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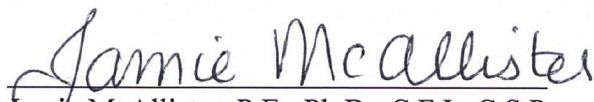
Supplement A: Ecotoxicity analysis



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**ECOTOXICITY ANALYSIS OF RINSE SAMPLES FROM
FIREFIGHTING APPARATUS AFFF DECONTAMINATION TRIAL**

by


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1. BACKGROUND

The Department of National Defence (DND) performed a trial decontamination procedure. The goal of this procedure was to remove aqueous film forming foam (AFFF) and associated per- and polyfluoroalkyl substances (PFAS) from an aircraft rescue and firefighting vehicle (ARFF). The ARFF vehicle had an 800-liter foam tank containing AFFF. According to DND, the AFFF was Ansulite 3% foam concentrate (Product code 431441). The tank and associated distribution piping were assumed to be contaminated with PFAS from the AFFF foam. As part of the decontamination protocol, DND utilized Arcadis V171 cleaning agent. According to Arcadis, the V171 product is effective in breaking down PFAS contaminants which can adhere to holding tank and distribution piping surfaces. If PFAS substances are not effectively removed from tank and piping surfaces, they can contaminate non-fluorinated replacement foams.

In advance of the trial decontamination procedure, NRC requested that FireTox complete the following: 1) review the DND trial protocol for the decontamination of ARFF vehicles to ensure it adequately addressed the safety of the environment and personnel, and 2) review the sampling and lab analysis plan for testing of the foam and rinse effluent collected during the decontamination procedure. After completing Tasks 1 and 2, FireTox issued a letter report to NRC on October 21, 2020. FireTox's findings were subsequently incorporated into NRC's letter report to DND issued on October 28, 2020. After completion of the trial decontamination procedure, NRC requested that FireTox review the laboratory test results and evaluate the ecotoxicity of the chemicals found within the foam and rinse samples.

The decontamination trial procedure was conducted over four days from November 23, 2020 to November 26, 2020. The protocol utilized by DND is provided in Appendix A. During Day 1, the Ansulite AFFF foam was drained from the tank and the tank and piping were rinsed with water. A rinse sample was collected on Day 1 at 1300 hours, i.e., *Water Rinse (pre-V171)*. On Day 2, the foam tank was filled with a mixture of V171 cleaning agent and water (650 L cleaning agent to 150 L water, respectively). The solution was agitated for 2-1/2 hours. A sample was collected on Day 2 at 1530 hours, i.e., *V171 First Flush*.

The tank was partially drained and refilled with water and allowed to sit for 12-hours overnight. A sample was collected on Day 3 at 0900 hours, i.e., *V171 Soak 20 hours*. After successive draining and filling with V171, the tank was rinsed with water and another sample was collected at 1305 hours, i.e., *1st Rinse (Water)*. Then, the tank was partially filled with water which remained in the tank overnight. On Day 4, a sample was collected at 0800 hours, i.e., *2nd Rinse (Water)*. The tank was drained and power washed. A final rinse sample was collected at 0940 hours, i.e., *Final Rinse*. Two control samples were collected, one on Day 1 and one on Day 4. An Ansulite 3% AFFF foam sample, i.e., *Pure AFFF* and a local water source sample, i.e., *Local Water Sample* were also obtained. Samples were sent to ALS Environmental and SGS AXYS, Ltd. ("SGS") for analyses.

2. ANALYSIS

The Analysis Section of this report presents the ALS Environmental and SGS laboratory testing results as well as the Task 3 ecotoxicity evaluation. A list of materials and references reviewed as part of this analysis is provided in Appendix B.

2.1 Limonene Ecotoxicity

One of the purposes of the AFFF decontamination procedure was to remove environmentally harmful PFAS contaminants from the ARFF apparatus. With this goal in mind, it was important to ensure that the cleaning agent used in the decontamination process was not environmentally harmful. For this reason, FireTox was asked to identify any chemicals in the V171 cleaning agent that had the potential to be environmentally harmful. According to the V171 Safety Data Sheet (SDS) dated August 18, 2020, d-limonene (CAS 5989-27-5) is the primary component of V171 at less than 65% concentration. Section 12 of the SDS indicates that the Lethal Concentration 50 (LC₅₀) value for fish and Effective Concentration 50 (EC₅₀) values for water fleas and freshwater algae are less than 1 mg/L for d-limonene. These values are a measure of the aquatic ecotoxic potency of d-limonene. A component with an L(E)C₅₀ less than or equal to 1 mg/L for either fish, water fleas, or algae, is classified as Acute 1 under the Global Harmonized System (GHS). Acute 1 classification means that the substance can be “Very Toxic to aquatic life” at certain concentrations.

V171 contains ethanol which has an L(E)C₅₀ of less than 10 mg/L. A component with an L(E)C₅₀ less than or equal to 10 mg/L for either fish, water fleas, or algae, is classified as Acute 2 under GHS. Acute 2 classification means that the substance can be “Toxic to aquatic life” at certain concentrations. Unlike d-limonene, however, the ethanol concentration in the V171 mixture is less than 10%. Given the small percentage in the mixture and further dilution during the rinse cycle, the concentration of ethanol in the V171 rinse would not be environmentally harmful. The V171 cleaning agent contains several other substances which would not be classified as acutely toxic based upon their LC₅₀ values. These substances include diethylene glycol monobutyl ether, methanol, acetic acid, citric acid, potassium chloride, sodium chloride, hexylene glycol, and propylene glycol. As such, these substances and ethanol were not evaluated; the focus of the laboratory analysis and ecotoxic evaluation was on d-limonene.

ALS Environmental evaluated the concentration of d-limonene in the Pure AFFF, 1st Rinse (Water), and Final Rinse samples. The Pure AFFF sample was utilized to establish the concentration of d-limonene detectable in a sample not containing d-limonene, e.g., a blank sample. The 1st Rinse and Final Rinse samples were conservatively assumed to contain the highest concentration of d-limonene that could remain inside the tank and piping surfaces after rinsing. As such, it was assumed that the final rinse sample represented the highest possible concentration of d-limonene available to contaminate a non-fluorinated replacement foam. Table 1 presents the

results of the d-limonene testing for the Pure AFFF, 1st Rinse (Water) and Final Rinse (Water). The method utilized by ALS Environmental had a detection limit of 500 ng/L.

Table 1: ALS Environmental Limonene Analysis Results		
Sample	Limonene (ng/L)	Limonene %
Pure AFFF	< 10,000	0.000001
1 st Rinse (Water)	303,000	0.0000303
Final Rinse	< 500	0.00000005

The results show that the blank sample contained less than 10,000 ng/L (or < 10 ppb) of d-limonene and that the final rinse sample contained less than the blank. The results indicate that no d-limonene remained inside the tank after the final rinse. Moreover, the concentration of d-limonene present in the final rinse was several orders of magnitude less than the L(E)C₅₀ value and one order of magnitude less than the No Observed Effect Concentration (NOEC)¹. The NOEC represents the highest concentration showing no statistically significant adverse impact on aquatic life. Based upon the above findings, it can be concluded that the highest possible concentration of d-limonene remaining inside the tank and piping does not present an aquatic ecotoxic risk.

2.2 PFAS Ecotoxicity

It is known that PFAS substances can adhere to the tank and distribution piping surfaces in ARFF vehicles. The purpose of the V171 is to assist in breaking down and removing the PFAS from these surfaces. If the PFAS is not effectively removed from these surfaces, then non-fluorinated replacement foams can become contaminated with PFAS substances remaining on these surfaces. The focus of this analysis will be determining if PFAS concentrations remaining on surfaces are sufficiently low such that aquatic ecotoxic risk is equally low. This will, in turn, determine the effectiveness of the decontamination protocol as it related to ecotoxic risk².

To evaluate the ecotoxic risk, it will be conservatively assumed that the concentration of PFAS that remains on tank and piping surfaces in the ARFF vehicle is no greater than the concentration found within a given rinse sample. As such, it can be conservatively assumed that the final rinse sample represents the highest possible concentration of PFAS remaining on the tank and piping surfaces. Therefore, the final rinse sample would represent the highest possible concentration of PFAS available to contaminate a non-fluorinated replacement foam. As a worst-case scenario, the ecotoxicity of the Pure AFFF sample (i.e., Ansulite 3% AFFF) will also be evaluated; the Pure AFFF sample contains the highest concentration of undiluted PFAS contaminates.

¹ As reported in the Environmental Protection Agency ECOTOX and ECOSAR databases.

² This analysis does not address human health hazards associated with PFAS, as it was outside of the requested scope. See Discussion Section for further details.

SGS evaluated the Pure AFFF and rinse samples for perfluorinated organics using Method MLA-110 (Revision 2). According to the SGS website, its method measures “40 PFAS to cover all EPA 537.1, EPA 533, and several additional precursors.”³ Table 2 provides a summary the 40 PFAS analyzed by SGS.

Table 2: 40 PFAS analyzed as part of SGS AXYS Method 110			
PFBA	PFTeDA	6:2 FTS	ADONA
PFPeA	PFBS	8:2 FTS	9Cl-PF3ONS
PFHxA	PFPeS	PFOSA	11Cl-PF3OUdS
PFHpA	PFHxS	N-MeFOSA	3:3 FTCA
PFOA	PFHpS	N-EtFOSA	5:3 FTCA
PFNA	PFOS	MeFOSAA	7:3 FTCA
PFDA	PFNS	EtFOSAA	PFEESA
PFUnA	PFDS	N-MeFOSE	PFMPA
PFDoA	PFDoS	N-EtFOSE	PFMBA
PFTTrDA	4:2 FTS	HFPO-DA	NFDHA

Samples were also analyzed for Total Oxidizable Precursor (TOP) using SGS Method MLA-111 (Revision 3). TOP Assay is a post-oxidative method that can assist in detecting PFAS substances that are not measurable by routine analytical methodology (LC-MS/MS). TOP assay converts precursors into detectable substances through oxidation, i.e., polyfluorinated alkyl substances to perfluorinated alkyl substances. As such, post-TOP concentrations are typically higher than pre-TOP concentrations for most PFAS substances.

Table 3 provides a summary of laboratory results for the pre-TOP assay. Table 4 provides a summary of laboratory results for the post-TOP assay.

³ <https://www.sgsaxys.com/2020/06/02/us-dod-and-epa-update-on-pfas-methods/>

Table 3: Pre-TOP Assay Results (SGS Method 110)							
Substance	AFFF (ng/L) ⁴	Water Rinse (pre-V171) (ng/L)	First Flush (V171) (ng/L)	20-hour Soak (V171) (ng/L)	1 st Rinse (Water) (ng/L)	2 nd Rinse (Water) (ng/L)	Final Rinse (Water) (ng/L)
PFBA	1,472,900	538	5,200	4,880	207	730	484
PFPeA	546,930	317	2,100	2,070	84.4	275	233
PFHxA	1,926,100	869	7,170	6,620	256	903	639
PFHpA	472,770	495	1,630	1,480	94	278	187
PFOA	1,081,500	1,200	3,310	3,650	131	671	360
PFNA	147,290	546	965	958	29.1	138	64.5
PFDA	594,310	400	2,050	1,960	84.1	428	169
PFUnA	62,727	107	ND	ND	17.8	56.6	66.6
PFDoA	470,710	152	3,740	3,980	131	453	1,860
PFTTrDA	30,285	15.5	644	752	33	52.7	646
PFTeDA	125,660	52.1	14,200	13,800	707	570	8,260
PFOS	ND	3.12	ND	ND	ND	ND	ND
6:2 FTS	4,717,400	1,500	26,500	17,100	700	3,190	1,260
8:2 FTS	109,180,000	20,500	866,000	910,000	19,000	39,300	26,800
5:3 FTCA	ND	407	ND	ND	ND	103	ND
7:3 FTCA	257,500	326	ND	ND	ND	173	ND
Total PFAS	121,085,770	27,428	933,509	967,250	21,474	47,321	41,029

ND = Not Detected

⁴ Laboratory reporting units were ng/g. The SDS for the Ansolite 3% AFFF (Product ID: 431441) does not report a density. A similar Ansolite 3% AFFF composition (Product ID: 446894) had a reported density of 1.03 g/ml. For the purposes of unit conversion from ng/g to ng/L, a density of 1.03 g/ml was utilized.

Table 4: Post-TOP Assay Results (SGS Method 111)							
Substance	AFFF (ng/L) ⁵	Water Rinse (pre-V171) (ng/L)	First Flush (V171) (ng/L)	20-hour Soak (V171) (ng/L)	1 st Rinse (Water) (ng/L)	2 nd Rinse (Water) (ng/L)	Final Rinse (Water) (ng/L)
PFBA	2,080,600,000	184,000	1,680,000	1,580,000	166,000	77,200	436,000
PFPeA	3,007,600,000	261,000	1,780,000	1,610,000	257,000	116,000	774,000
PFHxA	2,678,000,000	266,000	1,760,000	1,570,000	252,000	113,000	1,150,000
PFHpA	2,461,700,000	203,000	1,980,000	1,720,000	263,000	128,000	1,850,000
PFOA	1,751,000,000	164,000	1,860,000	1,550,000	254,000	108,000	2,360,000
PFNA	795,160,000	54,200	1,090,000	960,000	115,000	40,100	709,000
PFDA	456,290,000	39,300	630,000	580,000	78,600	24,300	487,000
PFUnA	236,900,000	17,500	557,000	480,000	67,200	19,000	612,000
PFDoA	152,440,000	15,100	697,000	554,000	77,600	24,200	832,000
PFTTrDA	80,340,000	6,510	726,000	531,000	82,900	24,800	1,050,000
PFTeDA	42,127,000	3,910	593,000	446,000	54,200	17,600	792,000
Total PFAS	13,742,157,000	1,214,520	13,353,000	11,581,000	1,667,500	692,200	11,052,000

⁵ Laboratory reporting units were ng/g. The SDS for the Ansulite 3% AFFF (Product ID: 431441) does not report a density. A similar Ansulite 3% AFFF composition (Product ID: 446894) had a reported density of 1.03 g/ml. For the purposes of unit conversion from ng/g to ng/L, a density of 1.03 g/ml was utilized.

The GHS summation method was utilized to evaluate the ecotoxicity of the PFAS substances at the concentrations found in the foam and rinse samples. The GHS summation method utilizes aquatic toxicity data along with bioaccumulative potential and degradability of the substance to classify acute and chronic ecotoxic risk⁶. The GHS utilizes three acute hazard categories and four chronic hazard categories (UN, 2019), as summarized in Table 6.

Table 6: GHS hazard category, code, and statement (UN, 2019).		
Category	Hazard Code	Hazard Statement
Acute 1	H400	“Very toxic to aquatic life”
Acute 2	H401	“Toxic to aquatic life”
Acute 3	H402	“Harmful to aquatic life”
Chronic 1	H410	“Very toxic to aquatic life with long lasting effects”
Chronic 2	H411	“Toxic to aquatic life with long lasting effects”
Chronic 3	H412	“Harmful to aquatic life with long lasting effects”
Chronic 4	H413	“May cause long lasting harmful effects to aquatic life”

The GHS acute hazard category of a chemical or mixture is determined using its LC₅₀, EC₅₀ and/or ErC₅₀ value(s). The GHS chronic hazard category of a chemical or mixture is determined using its NOEC, LOEC, or ChV value. In the absence of chronic toxicity data, the chronic hazard category of a chemical or mixture is determined using acute toxicity data as well as degradability and bioaccumulation data for the substance. Appendix C includes extracted content from the 8th edition of the GHS detailing the classification and summation methodology (UN, 2019).

Table 7 provides a summary of the lowest L(E)C₅₀ value for either fish, daphnid (e.g., water fleas), or algae reported in the ECOSAR and ECOTOX databases⁷ as well as the acute hazard category. The values in Table 7 represent the lowest acute effect on either mortality, growth, population, or reproduction. The most conservative, lowest L(E)C₅₀ value from the ECOSAR or ECOTOX databases was selected to categorize the acute hazard. The method set forth in Table 4.1.1(a) in Appendix C was used to categorize the acute hazard. Table 8 provides a summary of the lowest NOEC or ChV values for either fish, daphnid, or algae reported in the ECOSAR and ECOTOX databases as well as the chronic hazard category. The most conservative, lowest NOEC or ChV value from the ECOSAR or ECOTOX databases was selected to categorize the chronic hazard. The European Chemicals Agency (ECHA) database was utilized to evaluate degradability of all PFAS substances (Rapidly Degradable (RD) or Non-Rapidly Degradable (NRD)).

The degradability of a substance is a measure of its persistence in the environment. Biochemical oxygen demand (BOD) and chemical oxygen demand (COD) are used to quantify

⁶ See FireTox’s November 2020 Report “Ecotoxicity of Fluorine Free Foams Used for Fire Extinguishment”, for further details on the GHS Summation Method.

⁷ The search criteria in the ECOTOX database were set to fresh water and Growth, Mortality, Populations, and Reproduction Groups.

biodegradability. The BOD is the amount of dissolved oxygen required by an aerobic organism to disintegrate an organic pollutant. The test is commonly performed for 5 days (i.e., BOD5). The COD is the amount of oxygen needed to oxidize organic and soluble matter. The BOD and COD are derived from standardized tests. In some cases, a manufacturer may report a theoretical oxygen demand (ThOD) which is the amount of oxygen needed to oxidize a substance. The ThOD is a calculated value based on the chemical structure of the substance. The ThOD can be used in place of the COD.

Where data was not available in the ECHA database, it was conservatively assumed that the substance was NRD. The method set forth in Table 4.1.1(b)(i) in Appendix C was used to categorize the chronic hazard. NOEC and ChV data were not available for 5:3 FTCA and 7:3 FTCA, so acute data was utilized in combination with the method outlined in Table 4.1.1(b)(iii) in Appendix C.

Table 7: Lowest Acute Effect Levels and Most Conservative GHS Acute Category				
Substance	CAS No.	ECOSAR Lowest L(E)C ₅₀ (mg/L)	ECOTOX Lowest L(E)C ₅₀ (mg/L)	GHS Acute Category
PFBA	375-22-4	597	> 1006	NAT
PFPeA	2706-90-3	250	32	Acute 3
PFH _x A	307-24-4	79.3	NR	Acute 3
PFHpA	375-85-9	24.5	> 1019	Acute 3
PFOA	335-67-1	7.44	0.12	Acute 1
PFNA	375-95-1	2.22	80.93	Acute 2
PFDA	335-76-2	0.656	10.6	Acute 1
PFUnA	2058-94-8	0.192	NR	Acute 1
PFDoA	307-55-1	0.056	112.38	Acute 1
PFT _r DA	72629-94-8	0.016	NR	Acute 1
PFT _e DA	376-06-7	0.0043	95.69	Acute 1
PFOS	1763-23-1	16.9	NR	Acute 3
6:2 FTS	27619-97-2	521	> 108	NAT
8:2 FTS	39108-34-4	47.7	NR	Acute 3
5:3 FTCA	914637-49-3	NR	22.5	Acute 3
7:3 FTCA	812-70-4	NR	2.1	Acute 2

NR = Not Reported, NAT = No Acute Toxicity

Table 8: Lowest Chronic Effect Levels and Most Conservative GHS Chronic Category				
Substance	CAS No.	ECOSAR Lowest ChV (mg/L)	ECOTOX Lowest NOEC (mg/L)	GHS Chronic Category
PFBA	375-22-4	76.8	137	NCT
PFPeA	2706-90-3	30	NR	NCT
PFHxA	307-24-4	11.3	6.28	NCT
PFHpA	375-85-9	4.15	23.3	NCT
PFOA	335-67-1	1.34	0.1	Chronic 1
PFNA	375-95-1	0.405	0.008	Chronic 1
PFDA	335-76-2	0.121	0.1	Chronic 1
PFUnA	2058-94-8	0.036	0.1	Chronic 1
PFDoA	307-55-1	0.011	0.24	Chronic 1
PFTTrDA	72629-94-8	0.0031	0.1	Chronic 1
PFTeDA	376-06-7	0.00089	NR	Chronic 1
PFOS	1763-23-1	3.03	0.005	Chronic 1
6:2 FTS	27619-97-2	62.8	> 125	NCT
8:2 FTS	39108-34-4	7.77	NR	NCT
5:3 FTCA	914637-49-3	NR	NR	Chronic 3*
7:3 FTCA	812-70-4	NR	NR	Chronic 2*

NR = Not Reported, NCT = No Chronic Toxicity, * Using Table 4.1.1(b)(iii)

The post-TOP PFAS concentrations in the Pure AFFF and 2nd Rinse⁸ samples were selected for the ecotoxicity analysis. Use of the post-TOP values was most conservative, as these values were several orders of magnitude greater than the pre-TOP values. The post-TOP values shown in Table 4 were converted to % concentration to perform the GHS summation method calculations as outlined in Tables 4.1.3 through 4.1.5 in Appendix C. Tables 9 and 10 provide the % concentration in the Ansulite 3% AFFF foam and 2nd Rinse samples as well as the acute and chronic categories and values used for the calculations. M-factors for Acute 1 category substances were determined as outlined in Table 4.1.5 in Appendix C.

⁸ DND requested that the 2nd Rinse sample be used for Ecotoxicity analysis. DND indicated that the Final Rinse sample was mislabeled and is more likely to be the final rinse sample taken after the V171 application, rather than the final rinse sample taken at the end of the test cycle. It is noted that Final Rinse sample PFAS concentrations are greater than the 2nd Rinse Sample PFAS concentrations. Regardless, use of either the Final Rinse or 2nd Rinse samples does not change the outcome of the Ecotoxicity Analysis.

Table 9: Acute Ecotoxicity Category of Mixture					
Post-TOP	AFFF (%)	2nd Rinse (%)	Acute Category	Lowest L(E)C₅₀	M-factor*
PFOA	0.175	0.000011	Acute 1	0.1209	1
PFDA	0.046	0.000002	Acute 1	0.656	1
PFUnA	0.024	0.000002	Acute 1	0.192	1
PFDoA	0.015	0.000002	Acute 1	0.056	10
PFTTrDA	0.008	0.000002	Acute 1	0.016	10
PFTeDA	0.004	0.000002	Acute 1	0.0043	100
PFNA	0.080	0.000004	Acute 2		
PFPeA	0.301	0.000012	Acute 3		
PFHxA	0.268	0.000011	Acute 3		
PFHpA	0.246	0.000013	Acute 3		
Acute 1 if Acute 1 % x M ≥ 25%					
		AFFF %	Final Rinse %		
	Acute 1 Sum	0.90	0.0002		
Acute 2 if (M x 10 x Acute 1 %) + Acute 2% ≥ 25%					
		AFFF %	Final Rinse %		
	Acute 2 Sum	9.06	0.0024		
Acute 3 if (M x 100 x Acute 1 %) + (10 x Acute 2 %) + Acute 3 % ≥ 25%					
		AFFF %	Final Rinse %		
	Acute 3 Sum	91.46	0.0241		

*See Table 4.1.5 in Appendix C

Table 10: Chronic Ecotoxicity Category of Mixture					
Post-TOP	AFFF (%)	Final Rinse (%)	Chronic Category	Lowest NOEC or ChV	M-factor*
PFOA	0.175	0.000236	Chronic 1	0.1	1
PFNA	0.080	0.000071	Chronic 1	0.008	10
PFDA	0.046	0.000049	Chronic 1	0.1	1
PFUnA	0.024	0.000061	Chronic 1	0.036	1
PFDoA	0.015	0.000083	Chronic 1	0.011	1
PFTTrDA	0.008	0.000105	Chronic 1	0.0031	10
PFTeDA	0.004	0.000079	Chronic 1	0.00089	100
Chronic 1 if Chronic 1 % x M \geq 25%					
		AFFF %	Final Rinse %		
	Chronic 1 Sum	1.56	0.0003		
Chronic 2 if (M x 10 x Chronic 1 %) + Chronic 2 % \geq 25%					
		AFFF %	Final Rinse %		
	Chronic 2 Sum	15.56	0.0026		
Chronic 3 if (M x 100 x Chronic 1 %) + (10 x Chronic 2 %) + Chronic 3 % \geq 25%					
		AFFF %	Final Rinse %		
	Chronic 3 Sum	155.64	0.0258		

*See Table 4.1.5 in Appendix C

The criteria Acute or Chronic 1, 2, or 3 is a calculated concentration equal to or greater than 25%. Using the GHS summation method, the Ansulite 3% AFFF foam mixture would be classified as Acute 3/Chronic 3. Acute 3 is defined as “Harmful to aquatic life”. Chronic 3 is defined as “Harmful to aquatic life with long lasting effects”. Using the GHS Summation method, the 2nd Rinse sample was found to be neither an acute nor chronic aquatic hazard; after summing the concentrations of acutely or chronically classified PFAS contaminants in the final rinse sample, the concentration was still orders of magnitude less than 25%.

3. DISCUSSION

The % concentration of PFAS contaminants in the post-TOP final rinse sample were below levels that would result in classification as an acute or chronic aquatic hazard. It is important to note, however, that the ecotoxicity analysis does not address the potential human health hazards related to exposure to PFAS substances. While this study found that the concentration of PFAS in the final rinse samples does not present a hazard to aquatic organisms, the majority of the PFAS

substances found in the post-TOP analysis are NRD. As such, these chemicals persist in the environment and make their way into water and food sources consumed by humans. Additionally, remaining PFAS components present in the tank and distribution piping can contaminate non-fluorinated replacement foams; this introduces the potential for firefighters to be exposed to these contaminants during training and firefighting operations.

The thresholds of concern for human health are much lower than those used to evaluate ecological hazards. This same point is made in a report submitted by GHD Pty Ltd to the Australian Department of Defense. The GHD report provides recommendations for acceptable residual PFAS levels in ARFF vehicles post-decontamination. In their report, GHD stated "it is proposed that the cleaning criteria are developed to be protective of the health of workers operating firefighting vehicles on the Defence estate, rather than environmental receptors." Australia provides guidelines for drinking water and recreational water quality, as shown in Table 11.

Table 11: Australian PFAS Health Based Guidance Values.		
	Total PFOS + PFHxS (ng/L)	PFOA (ng/L)
Drinking Water Quality	70	560
Recreational Water Quality	2,000	10,000

The GHD report suggests the use of recreational water limits shown in Table 11 for ARFF vehicle cleaning criteria. GHD equates swimming (in which exposure is primarily dermal and not through ingestion) with the type of exposure that a firefighter may receive when operating equipment with PFAS contaminated water or liquid. The recreation water limits are a combined concentration of PFOS and PFHxS not to exceed 2,000 ng/L and a concentration of PFOA not to exceed 10,000 ng/L. GHD suggests that the remaining total PFAS concentration (all other components excluding PFOS, PFHxS and PFOA) should not exceed 5000 ng/L. It appears that the 5000 ng/L total PFAS limit for initial cleaning refers to the concentration present in the replacement foam concentrate; this is based upon the statement in the GHD report that “the final samples will be collected after the vehicles have been returned to service...”⁹ GHD further suggests that a total PFAS concentration (presumably including PFOS, PFHxS, and PFOA) of 15,000 ng/L be adopted as a criterion for when apparatus needs to be re-cleaned. In other words, a total PFAS concentration of 15,000 ng/L found in samples collected after a vehicle has been returned to service would suggest that the initial decontamination process was ineffective.

In the present study, the Pre- TOP PFOA concentration was 670 ng/L in the 2nd rinse, and the pre-TOP total PFAS concentration (all other components excluding PFOS, PFHxS and PFOA) was 46,650 ng/L in the 2nd rinse. The Post- TOP PFOA concentration was 108,000 ng/L in the 2nd rinse, and the post-TOP total PFAS concentration (all other components excluding PFOS,

⁹ In the present study, the final rinse sample was used as a highly conservative way to represent the maximum possible concentration remaining inside the tank and piping surfaces. The concentration within the final rinse sample is likely an overestimate of the concentration remaining in the tank and piping. Future testing of the non-fluorinated replacement foam would be required to determine the actual concentration of PFAS remaining in the tank and piping.

PFHxS and PFOA) was 584,200 ng/L in the 2nd rinse. No PFOS or PFHxS were detected in either pre-TOP or post-TOP testing. The pre-TOP PFOA value is below the threshold suggested by GHD (10,000 ng/L), and the total PFAS concentration is above the thresholds suggested by GHD (5,000 ng/L). The post-TOP values are significantly higher than the thresholds suggested by GHD (10,000 ng/L for PFOA and 5,000 ng/L for total PFAS, respectively).

While Canada does not have recreational water quality guidelines for PFAS, it does have maximum acceptable thresholds for PFOA and PFOS in drinking water, as shown in Table 12.

Table 12: Canadian PFOA/PFOS Drinking Water Limits.				
Substance	Canadian Drinking Water (ng/L)	Local Water Source (ng/L)	Pre-TOP Final Rinse (ng/L)	Post-TOP Final Rinse (ng/L)
PFOA	200	0.799	360	2,360,000
PFOS	600	ND	ND	ND

In the present study, both the pre-TOP and post-TOP final rinse values for total PFOA were above the Canadian maximum acceptable threshold for PFOA in drinking water. The guidelines and limits shown in Tables 11 and 12 further reiterates the significant difference between human health hazard thresholds and ecological hazard thresholds.

3.1 Post-decontamination

It is important to consider the safety of the environment and personnel both during and after the decontamination process. In doing this, it is important to identify PFAS concentrations in effluent that would be indicative of a successful decontamination process, and it is important to identify acceptable PFAS concentrations within non-fluorinated replacement foams. It is recommended that DND set two goal standards to measure the success of the decontamination process. The first goal standard should identify the percent (%) reduction of PFAS in the decontamination effluent that will be indicative of a successful process. The second goal standard should identify the maximum acceptable concentration of PFAS within non-fluorinated replacement foams, which will also identify the success of the decontamination process.

The GHD study suggests a total PFAS limit (all other components excluding PFOS, PFHxS, and PFOA) of 5,000 ng/L for initial cleaning, and a total PFAS limit of 15,000 ng/L for a re-cleaning trigger. In a recent publication, Wood, Ramboll, COWI conducted a stakeholder workshop and presented various processes used within the industry for PFAS decontamination. The conclusions within the Wood, Ramboll, COWI report raises questions as to the feasibility of achieving the limits set by GHD. The following is a summary of findings from the Wood, Ramboll, COWI report regarding concentration thresholds:

- One stakeholder recommended adoption of Queensland (Australia) regulations which allows up to 10,000 ppb (10,000,000 ng/L) of PFOA/PFHxS in non-fluorinated foam and

50,000 ppb (50,000,000 ng/L) of PFOA related precursors and higher homologues in non-fluorinated foam.

- **The DND trial decontamination procedure resulted in a post-TOP 2nd rinse sample PFOA concentration of 108,000 ng/L.**
- One stakeholder who is currently using fluorine-free foams indicated that they achieved a PFAS threshold of 10,000 ppb (10,000,000 ng/L)
 - **The DND trial decontamination procedure resulted in a post-TOP 2nd rinse sample Total PFAS concentration of 692,200 ng/L.**
- One stakeholder utilized a cleaning process which involved emptying the tank and flushing it two times with warm water. This process led to “very low” levels of PFAS, both tested immediately after cleaning and a few years after cleaning. The stakeholder cautioned, however, that a threshold below 100 ppb (100,000 ng/L) would be unrealistic.
- Two stakeholders indicated that PFAS levels below 1 ppb (1000 ng/L) would be the lowest achievable concentration threshold. This threshold was achieved with a 32-stage decontamination process using hot water and detergents. The process cost approximately 12,300 euros per appliance. In 1/3rd of cases using this process, PFAS levels were as low as 0.007 ppb (7 ng/L).
- Several stakeholders indicated that achieving a zero percent threshold is not possible and that achieving levels below 100 ppb (100,000 ng/L) is unrealistic.

It is outside of the scope of this review to provide recommendations on concentration limits to be adopted in DND protocol. As such, this information is presented for consideration by DND when determining appropriate goal standards and success measures to implement.

4. CONCLUSIONS

This analysis found that the Ansulite 3% AFFF foam mixture (undiluted) is harmful to aquatic life with long lasting effects (i.e., Acute 3/Chronic 3). Conversely, the % concentration of PFAS contaminates in the post-TOP 2nd rinse sample were below levels that would result in classification as an acute or chronic aquatic hazard. This finding suggests that the decontamination process is effective in reducing PFAS contaminates within the tank and distribution piping to levels below those considered ecotoxic to aquatic life. PFAS human health hazard thresholds are significantly lower. For this reason, it is recommended that human health hazard thresholds be utilized when evaluating the efficacy of the decontamination process. It is further recommended that the non-fluorinated foam used to replace the AFFF foam be tested for PFAS concentration several months after it is introduced into the ARFF tank. It is also recommended that DND set two goal standards for the decontamination process. The first goal standard should identify the percent (%) reduction of PFAS in the decontamination effluent that will be indicative of a successful process. The second goal standard should identify the maximum acceptable concentration of PFAS within the non-fluorinated replacement foams, which will also identify the success of the decontamination process.

Appendix A:
DND ARFFV Foam Decontamination Procedure- Greenwood

Equipment/Material:

1. 4 x Catchall trays (2'x2')
2. 1 x high pressure washer, hose and wand;
3. Packet of large absorbent pads;
4. Hazmat suites;
5. Box of Nitrile gloves;
6. 1 x hose to empty the foam tank;
7. 5 x 1100ltrs totes;
8. 6 x large buckets (5ltr);
9. 100' of garden hose;
10. Strong flashlight;
11. Mechanical dolly;
12. Safety glasses;
13. Portable pallet mover
14. V171 chemical 1100ltrs
15. 1/3 HP Submersible Sump pump with a 2" discharge
16. Large Funnel

Day One

1.	Confirm containment of testing area. Could be built into the building infra or may have to use spill pads/snakes in the event of a spill.	
2.	Obtain the vehicle piping specs.	
3.	Drain foam into totes.	
4.	Set up pressure washer and rinse out the foam tank under normal pressure.	
5.	Capture effluent from the rinse and dispose into a tote. Continue until collecting effluent until water is clear. Took sample #1.	Sample ID "Water Rinse 500ml/100ml". Date and time 23 Nov 1300hrs
6.	Set up pressure washer with high pressure setting and rinse the foam tank. Note significant agitation will occur. Drain out and collect effluent. Dispose in the tote.	
7.	Using a garden hose, insert it into the drain orifice under normal pressure. Collect spilling effluent and dispose.	
8.	Position the V171.	
Day Two		
1.	Using a 1/3 HP Submersible Sump pump with a 2" discharge filled the foam tank with 650 L of V171 and 150 L of water.	

2.	Opened the Foam Pump valve to flood the metering valve with V171.	
2.	From 1100-1430hrs allowed the V171/water solution to soak for 2.5 hrs.	
3.	At 1430 hrs drained the foam tank to 50%.	
4.	Took Sample #2.	Sample ID: V171 First Flush 24 Nov 1530 hrs.
5.	Refilled tank to 75%.	
6.	Drained for 20 minutes.	
7.	Refilled the foam tank to 100%.	
8.	Commenced an overnight soak.	
Day Three		
1.	Started to drain after an overnight soak.	Sample ID: V171 Soak 20 hours. 25 Nov at 0900 hrs
2.		
3.	Drained to the 25% mark on the foam tank,	
4.	Refilled 200 L	
5.	0930 hrs commenced the 100% drain.	
6.	Rinsed the foam tank with vehicle water 1320 hrs. Used low pressure garden hose and rinsing the sides of the container. Rinse water immediately drained into a tote.	
7.	Commencing First Rinse Drain. 1020 hrs.	Lesson Learned: We refilled the tank with water from the trucks water tank. This proved to be a mistake because the water being discharged was the colour of tea and this resulted in significant foam agitation with little actual liquid water in the tank. To counter this we filled the tank again with V171 and drained it again. We refilled the tank one additional time just as a matter of ensuring we are starting at Step #5. Will next fully drain the tank of V171.
8.	Took Limonene sample at 1400 hrs.	Sample ID: 1st Rinse (Water) . Nov 25 @ 1400 hrs.
9	Closed the vehicle drain valve at 1415 hrs to allow the tank to completely fill.	
10.		LL: Be sure to drain the Main Vehicle drain

11.	1430hrs opened the Master Valve to drain out the V171 and captured this effluent into a bucket. Drained it until all V171 finished draining (approx' 80 L)	
12.	Commenced draining the foam tank of water at 1500 hrs.	
13.	Sample taken. First water sample after the first water flush (mid-stream).	Sample Title: 1 st Water Flush at 1505 hrs.
14.	1700 hrs the foam tank completely emptied of water. Commenced to refill with water. Once full this will sit over night.	
Day Four		
1.	Drained the foam tank of water.	
2.	Sample taken.	Sample ID: 2 nd Rinse Water, Nov 26 0800 hrs
3.	Power washed foam tank. No visible foam. Took a sample.	Sample ID: Final Rinse , 26 Nov 1010 hrs.
4.	Samples from foam concentrate for Limonene (NRC).	Sample ID: Pure AFFF . AFFF Concentrate Ansul 3%. C8. 26 Nov 1100 hrs.
5.	Sample from local water source.	Sample ID: Local Water Source. 26 Nov 1330 hrs

Appendix B: List of Materials and References

- ALS Environmental- Analytical Report- Limonene Results- December 10, 2020
- ALS Environmental- Spreadsheet- 1st Rinse, Final Rinse Limonene Results- December 10, 2020
- [Aqueous Film-Forming Foam and the Prohibition of Certain Toxic Substances Regulations](#), Environment and Natural Resources, January 2018.
- Arcadis Draft Report: ARFF Cleaning Pilot-Scale Testing Results, February 18, 2021
- Arcadis Email to DND, Subject RE: Truck flushing trial- TOP results
- Arcadis Preliminary Pre-TOP Assay ARFF Cleaning Data- powerpoint
- Arcadis PFAS Post-TOP Assay Results of Analyses- provided on March 26, 2021.
- Australian Government, Department of Health, [Health Based Guidance Values for PFAS](#).
- Canadian Environmental Law Association, [The Threat of PFAS: The Forever Chemicals](#), 2019.
- DND ARFFV Decontamination Trial Protocol CFB Greenwood 2020
- DND ARFFV Final Decontamination Trial Protocol CFB Greenwood 2020
- DND Response to Initial Comments on Decontamination Protocol
- [Firefighting Foam Management Policy](#), Queensland Government, May 2018.
- GHD Recommendations on Cleaning Criteria- Australian Department of Defence and Broadpectrum Firefighting Vehicles.
- [Guidelines for Canadian Drinking Water Quality: Guideline Technical Document – Perfluorooctane Sulfonate \(PFOS\)](#), Health Canada, December 2018.
- NRC Review Letter- ARFFV Decontamination Procedure, October 28, 2020.
- PFAS Testing Methodologies (2020), Pace Analytical, www.pacelabs.com.
- [PFAS: The Determination of Total Oxidizable Precursors \(TOPS\)](#), Bureau Veritas, October 2018.
- SDS for Ansulite 3% AFFF Concentrate (Formulation DC-3)- Product Code 431441, May 21, 2018.
- SDS for Arcadis Cleaning Agent V171, August 18, 2020.
- SGS AXYS Perfluorinated Organics (Pre) Analysis Report, Control 1, Water Rinse (pre-V171), 1st Rinse (Water), 2nd Rinse (Water), Final Rinse, January 21, 2021.
- SGS AXYS Perfluorinated Organics (Pre) Analysis Report, Pure AFFF, January 25, 2021.
- SGS AXYS Perfluorinated Organics (Pre) Analysis Report, First Flush (V171) and 20-hour Soak (V171), January 29, 2021.
- SGS AXYS Perfluorinated Organics (Pre) Spreadsheet, First Flush (V171) and 20-hour Soak (V171), January 29, 2021.
- SGS AXYS Perfluorinated Organics (Pre) Analysis Report, Control 2 and Local Water Source, February 2, 2021.

- SGS AXYS Perfluorinated Organics (Post) Analysis Report, Pure AFFF, February 5, 2021.
- SGS AXYS Perfluorinated Organics (Post) Analysis Report, V171 (First Flush), V171 (20 hr Soak), 1st Rinse, and Final Rinse, February 24, 2021.
- United Nations (2019). Globally Harmonized System of Classification and Labeling of Chemicals (GHS). Eighth revised edition. Retrieved from http://www.unece.org/fileadmin/DAM/trans/danger/publi/ghs/ghs_rev08/ST-SG-AC10-30-Rev8e.pdf
- Wood, Ramboll, COWI. [The Use of PFAS and Fluorine-Free Alternatives in Fire-fighting Foams](#), European Commission DG Environment/European Chemicals Agency (ECHA), June 2020.

Appendix C: Extracted GHS Classification Tables and Summation Method (UN, 2019)

Table 4.1.1: Categories for substances hazardous to the aquatic environment (Note 1)

(a) Short-term (acute) aquatic hazard

<u>Category Acute 1:</u> (Note 2)	
96 hr LC ₅₀ (for fish)	≤ 1 mg/l and/or
48 hr EC ₅₀ (for crustacea)	≤ 1 mg/l and/or
72 or 96hr ErC ₅₀ (for algae or other aquatic plants)	≤ 1 mg/l (Note 3)
Category Acute 1 may be subdivided for some regulatory systems to include a lower band at L(E)C ₅₀ ≤ 0.1 mg/l	
<u>Category Acute 2:</u>	
96 hr LC ₅₀ (for fish)	>1 but ≤ 10 mg/l and/or
48 hr EC ₅₀ (for crustacea)	>1 but ≤ 10 mg/l and/or
72 or 96hr ErC ₅₀ (for algae or other aquatic plants)	>1 but ≤ 10 mg/l (Note 3)
<u>Category Acute 3:</u>	
96 hr LC ₅₀ (for fish)	>10 but ≤ 100 mg/l and/or
48 hr EC ₅₀ (for crustacea)	>10 but ≤ 100 mg/l and/or
72 or 96hr ErC ₅₀ (for algae or other aquatic plants)	>10 but ≤ 100 mg/l (Note 3)
Some regulatory systems may extend this range beyond an L(E)C ₅₀ of 100 mg/l through the introduction of another category.	

Table 4.1.1: Categories for substances hazardous to the aquatic environment (Note 1) (cont'd)

(b) Long-term (chronic) aquatic hazard (see also figure 4.1.1)

(i) Non-rapidly degradable substances (Note 4) for which there are adequate chronic toxicity data available

Category Chronic 1: (Note 2)	
Chronic NOEC or EC _x (for fish)	≤ 0.1 mg/l and/or
Chronic NOEC or EC _x (for crustacea)	≤ 0.1 mg/l and/or
Chronic NOEC or EC _x (for algae or other aquatic plants)	≤ 0.1 mg/l
Category Chronic 2:	
Chronic NOEC or EC _x (for fish)	≤ 1 mg/l and/or
Chronic NOEC or EC _x (for crustacea)	≤ 1 mg/l and/or
Chronic NOEC or EC _x (for algae or other aquatic plants)	≤ 1 mg/l

(ii) Rapidly degradable substances for which there are adequate chronic toxicity data available

Category Chronic 1: (Note 2)	
Chronic NOEC or EC _x (for fish)	≤ 0.01 mg/l and/or
Chronic NOEC or EC _x (for crustacea)	≤ 0.01 mg/l and/or
Chronic NOEC or EC _x (for algae or other aquatic plants)	≤ 0.01 mg/l
Category Chronic 2:	
Chronic NOEC or EC _x (for fish)	≤ 0.1 mg/l and/or
Chronic NOEC or EC _x (for crustacea)	≤ 0.1 mg/l and/or
Chronic NOEC or EC _x (for algae or other aquatic plants)	≤ 0.1 mg/l
Category Chronic 3:	
Chronic NOEC or EC _x (for fish)	≤ 1 mg/l and/or
Chronic NOEC or EC _x (for crustacea)	≤ 1 mg/l and/or
Chronic NOEC or EC _x (for algae or other aquatic plants)	≤ 1 mg/l

(iii) Substances for which adequate chronic toxicity data are not available

Category Chronic 1: (Note 2)	
96 hr LC ₅₀ (for fish)	≤ 1 mg/l and/or
48 hr EC ₅₀ (for crustacea)	≤ 1 mg/l and/or
72 or 96hr ErC ₅₀ (for algae or other aquatic plants)	≤ 1 mg/l (Note 3)
and the substance is not rapidly degradable and/or the experimentally determined BCF is ≥ 500 (or, if absent, the log K _{ow} ≥ 4). (Notes 4 and 5)	
Category Chronic 2:	
96 hr LC ₅₀ (for fish)	> 1 but ≤ 10 mg/l and/or
48 hr EC ₅₀ (for crustacea)	> 1 but ≤ 10 mg/l and/or
72 or 96hr ErC ₅₀ (for algae or other aquatic plants)	> 1 but ≤ 10 mg/l (Note 3)
and the substance is not rapidly degradable and/or the experimentally determined BCF is ≥ 500 (or, if absent, the log K _{ow} ≥ 4). (Notes 4 and 5)	
Category Chronic 3:	
96 hr LC ₅₀ (for fish)	> 10 but ≤ 100 mg/l and/or
48 hr EC ₅₀ (for crustacea)	> 10 but ≤ 100 mg/l and/or
72 or 96hr ErC ₅₀ (for algae or other aquatic plants)	> 10 but ≤ 100 mg/l (Note 3)
and the substance is not rapidly degradable and/or the experimentally determined BCF is ≥ 500 (or, if absent, the log K _{ow} ≥ 4). (Notes 4 and 5).	

NOTE 1: The organisms fish, crustacea and algae are tested as surrogate species covering a range of trophic levels and taxa, and the test methods are highly standardized. Data on other organisms may also be considered, however, provided they represent equivalent species and test endpoints.

NOTE 2: When classifying substances as Acute 1 and/or Chronic 1 it is necessary at the same time to indicate an appropriate M factor (see 4.1.3.5.5.5) to apply the summation method.

NOTE 3: Where the algal toxicity ErC_{50} [$= EC_{50}$ (growth rate)] falls more than 100 times below the next most sensitive species and results in a classification based solely on this effect, consideration should be given to whether this toxicity is representative of the toxicity to aquatic plants. Where it can be shown that this is not the case, professional judgment should be used in deciding if classification should be applied. Classification should be based on the ErC_{50} . In circumstances where the basis of the EC_{50} is not specified and no ErC_{50} is recorded, classification should be based on the lowest EC_{50} available.

NOTE 4: Lack of rapid degradability is based on either a lack of ready biodegradability or other evidence of lack of rapid degradation. When no useful data on degradability are available, either experimentally determined or estimated data, the substance should be regarded as not rapidly degradable.

NOTE 5: Potential to bioaccumulate, based on an experimentally derived $BCF \geq 500$ or, if absent, a $\log K_{ow} \geq 4$, provided $\log K_{ow}$ is an appropriate descriptor for the bioaccumulation potential of the substance. Measured $\log K_{ow}$ values take precedence over estimated values and measured BCF values take precedence over $\log K_{ow}$ values.

Table 4.1.3: Classification of a mixture for short-term (acute) hazards based on summation of the concentrations of classified ingredients

Sum of the concentrations (in %) of ingredients classified as:	Mixture is classified as:
Acute 1 × M ^a ≥ 25%	Acute 1
(M × 10 × Acute 1) + Acute 2 ≥ 25%	Acute 2
(M × 100 × Acute 1) + (10 × Acute 2) + Acute 3 ≥ 25%	Acute 3

^a For explanation of the M factor, see 4.1.3.5.5.5.

Table 4.1.4: Classification of a mixture for long-term (chronic) hazards based on summation of the concentrations of classified ingredients

Sum of the concentrations (in %) of ingredients classified as:	Mixture is classified as:
Chronic 1 × M ^a ≥ 25%	Chronic 1
(M × 10 × Chronic 1) + Chronic 2 ≥ 25%	Chronic 2
(M × 100 × Chronic 1) + (10 × Chronic 2) + Chronic 3 ≥ 25%	Chronic 3
Chronic 1 + Chronic 2 + Chronic 3 + Chronic 4 ≥ 25%	Chronic 4

^a For explanation of the M factor, see 4.1.3.5.5.5.

Table 4.1.5: Multiplying factors for highly toxic ingredients of mixtures

Acute toxicity L(E)C ₅₀ value	M factor	Chronic toxicity NOEC value	M factor	
			NRD ^a ingredients	RD ^b ingredients
0.1 < L(E)C ₅₀ ≤ 1	1	0.01 < NOEC ≤ 0.1	1	-
0.01 < L(E)C ₅₀ ≤ 0.1	10	0.001 < NOEC ≤ 0.01	10	1
0.001 < L(E)C ₅₀ ≤ 0.01	100	0.0001 < NOEC ≤ 0.001	100	10
0.0001 < L(E)C ₅₀ ≤ 0.001	1000	0.00001 < NOEC ≤ 0.0001	1000	100
0.00001 < L(E)C ₅₀ ≤ 0.0001	10000	0.000001 < NOEC ≤ 0.00001	10000	1000
(continue in factor 10 intervals)		(continue in factor 10 intervals)		

^a Non-rapidly degradable

^b Rapidly degradable

Supplement B: NRC Review letter



28 October 2020

A1- 016841

CFFM 5, Canadian Forces Fire Marshal
Canadian Armed Forces

Attention: Major Rick Dunning

Re: Review Letter-ARFFV Decontamination Procedures

Dear Major Dunning:

We are enclosing a copy of review letter A1-016841 giving you results of the work carried out by the NRC Construction as requested in the Agreement dated 08 Oct. 2020.

We trust everything is to your complete satisfaction. Should you have any questions regarding this report, you may contact Yoon Ko at 613-998-2585 | Yoon.Ko@nrc-cnrc.gc.ca. We appreciate the opportunity to be of service to you.

Sincerely,

Ahmed Kashef, Ph.D., P. Eng.
Program Leader
Fire Safety
NRC Construction

cc: Yoon Ko

ARFFV Decontamination Procedure Review

Yoon Ko, Ph.D.¹, Rokib Hassan, Ph.D.¹ and Jamie McAllister, P.E., Ph.D., C.S.P.²

¹Fire Safety Unit, Construction Research Centre, National Research Council of Canada

²FireTox, MD, US

The Department of National Defence (DND) intends to perform a trial decontamination procedure to remove aqueous film forming foam (AFFF) and associated per- and polyfluoroalkyl substances (PFAS) from Aircraft Rescue and Fire Fighting (ARFF) vehicles and structural response vehicles. In support of this effort, NRC has been requested to perform the following three tasks:

- Task 1: Review the DND trial protocol for the decontamination of ARFF vehicles to ensure that it adequately addresses the safety of the personnel and environment.
- Task 2: Review the sampling details and lab analysis plan associated with the testing of the blank sample and effluents collected during the decontamination process.
- Task 3: Review the lab test results, assess the efficacy of the decontamination process and evaluate the ecotoxicity of the decontamination effluents (up to 4 samples).

This summary report presents reviews of the trial protocol for the decontamination of ARFF vehicles (Task 1) and reviews and recommendations for the sampling and lab analysis plan (Task 2). A comprehensive report including Task 3 findings will follow upon the completion of the trial decontamination and analysis of lab test results.

1 Decontamination Protocol Review

The DND trial decontamination protocol is reviewed to ensure the safety of the personnel and environment during the on-site work in CFB Greenwood, NS, planned for the month of November 2020.

1.1 Materials

Ansulite AFC-3DC 3% AFFF

According to the SDS for the AFFF concentrate, the foam product contains Polyfluorinated alkyl betaine, which is harmful to humans and the environment due to its low degradability and high persistence. Also, the foam can cause “serious eye damage” (GHS Health Hazard Code: H318, Category 1) and “mild skin irritation.” The AFFF SDS indicates that users should “**Avoid contact with skin, eyes, and clothing.**”

Cleaning agent Arcadis V171

The Arcadis V171 is combustible liquid and contains d-Limonene which is toxic to aquatic life. According to the SDS for the Arcadis V171 Cleaning Fluid, the chemical can cause “serious eye irritation” (GHS Health Hazard Code: H319, Category 2). The V171 SDS indicates that users should “**Avoid contact with skin, eyes, and clothing.**” and “**Avoid ingestion and inhalation.**”

To ensure safety and proper use and handling of the above listed materials, DND should have the SDS's for both chemicals readily accessible during use.

Effluent containers

DND should ensure that the size and type of the totes they intend to use for effluent containment are approved by the transporter and authorized hazardous waste management facility.

1.2 Safety equipment

Personal Protective Equipment (PPE)

Any personnel in contact with or within splash range of V171, AFFF, or the decontamination effluents should wear safety glasses with side-shields, tight sealing safety goggles, or full-face protection. Anyone handling or within splash range of V171, AFFF, or decontamination effluents should also wear long sleeved duty wear or coveralls, and nitrile rubber gloves with a minimum thickness of 0.4 mm. Anyone handling or within splash range of V171, AFFF, or decontamination effluents should wear non-absorptive, closed toed shoes should also be worn.

All contaminated PPE should be disposed according to applicable laws and regulations, and decontamination stations should be provided for non-disposal items, such as shoes. Contaminated PPE should be removed, and hands should be thoroughly washed and dried prior to eating or drinking.

Personnel that will have direct contact with V171 and AFFF, such as those personnel responsible for pouring the V171 into the foam tank or inspecting the foam tanks, should also wear respiratory protection to eliminate inhalation risks. Inhalation of vapors from V171 can cause respiratory tract irritation. DND has specified P100 cartridges (purple) or organic vapor cartridges (brown) both of which are acceptable.

The trial protocol indicates that personal protective equipment (PPE) will include coveralls, safety glasses, and work gloves for all participants. DND further stated that a full-face respirator with a P100 cartridge or an organic vapour cartridge would be required for anyone inspecting the tank. Additionally, DND stated that they would use butyl-style rubber gloves and would follow SDS recommended safety procedures.

Spill kits

DND has indicated that spill kits and berms will be utilized to ensure that any material spilled outside a capture tote is contained inside the berm.

1.3 Decontamination Procedure

As summarized in Table 1, the DND trial decontamination protocol consists of **preparation, foam drain, 5 cycles of rinse and clean-up**. In the DND protocol, only four samples were noted; however, it is recommended that a total of five samples be collected during the decontamination procedure as indicated in Table 1.

Table 1. Overview of the on-site decontamination procedure

PROCEDURE	Action	Sampling	Schedule
PREPARATION	<ul style="list-style-type: none">Position vehicle over berm.Position totes for capture of foam from main tank.Position bucket for capture of foam from drain lines.		Day 1
DRAIN	<ul style="list-style-type: none">Drain tank and drain lines.Empty bucket contents into totes.Collect a foam sample for a laboratory analysis testing.	(blank sample; SAMPLE B0)	

RINSE 1	<ul style="list-style-type: none"> Position vehicle over berm. Pump Arcadis V171 into foam tank at 50% V171:/50% water by volume. Agitate solution to ensure thorough mixing. Turn on foam system to flood internal system but not discharge effluent. 	SAMPLE R1	Day 2
SOAK	<ul style="list-style-type: none"> Let solution sit for prescribed time. Drain tank and piping using winterization protocol and capture effluent into totes. Collect an effluent sample for a lab analysis. 		
RINSE 2	<ul style="list-style-type: none"> Fill tank with water. Drain tank and piping using winterization protocol and capture effluent into totes. 		
RINSE 3	<ul style="list-style-type: none"> Fill tank with water. Drain tank and piping using winterization protocol and capture effluent into totes. Collect an effluent sample for a lab analysis. 	SAMPLE R3	
RINSE 4	<ul style="list-style-type: none"> Fill tank with water. Drain tank and piping using winterization protocol and capture effluent into totes. Take sample of effluent for laboratory testing. 	SAMPLE R4	Day 3
RINSE 5	<ul style="list-style-type: none"> Fill tank with water. Drain tank and piping using winterization protocol and capture effluent into totes. Take sample of effluent for laboratory testing. 	SAMPLE R5	
CLEAN UP	<ul style="list-style-type: none"> Clean and return equipment 		Day 4

Preparation

It is recommended that **the DND trial protocol include a spill response plan which identifies the mitigation process, mitigation team members, member roles and responsibilities, equipment needs, and emergency contact information.** The trial protocol identifies spill prevention strategies; however, it does not outline a spill response plan.

Drain, Containment and Handling

DND has indicated that effluent from the decontamination process will be captured in the totes, which should be approved by the transporter and authorized hazardous waste management facility. Label container[s] with legible, visible safety marks as prescribed by federal and provincial regulations. When required to be stored on the site, the totes should be closed, sealed and placed in a location that will prevent them from spilling or otherwise contaminating the environment.

Rinse procedure and Soak

It is also recommended that DND identify soak periods in the protocol. A soak period during the decontamination process appears to be common based upon a review of other decontamination processes. A soak period could be used to identify residual PFAS on equipment surfaces. **It is recommended that DND perform a minimum 24-hour soak period during RINSE 3 and prior to collection of SAMPLE R3 to evaluate the effectiveness of the decontamination process. It is also recommended that DND perform a minimum 24-hour soak period during RINSE 5 prior to collection of SAMPLE R5.**

Clean-up and effluent disposal

Improper disposal of decontamination effluent can have a negative impact on the environment. Products containing PFAS, even in diluted form, should never be dumped into landfills, sewer systems, or other land or waterways. Additionally, the V171 cleaning fluid to be used in this process is classified as hazardous waste. Guidance on disposal procedures is outside of the scope of this review, therefore, it is recommended that **DND retain the services of a company that is licensed in the handling and disposal of PFAS-contaminated products and other hazardous materials.**

The company should provide DND with a protocol for the handling and disposal of the AFFF, the decontamination effluent, and contaminated personal protective equipment (PPE). The protocol should identify the authorized hazardous waste management facility where the materials will be taken for disposal as well as the method of disposal. Additionally, the protocol should outline the specific type of totes or containers to be utilized and the requirements for hazardous material container labeling.

DND should identify a chemical hygiene officer who will serve as the liaison with the hazardous material disposal contractor to ensure proper packing and labeling of waste.

2 Sampling and Lab Analysis Plan

2.1 Overview and Objectives

Samples of the foam and effluent will be acquired throughout the decontamination procedure. The samples will be analysed for PFAS so that the efficacy of the decontamination method can be analysed for different rinse cycles based on the changes in the level of PFAS in each sample. The samples will be analysed not only for PFAS but also for d-Limonene, and with the lab analysis data of PFAS and d-Limonene, the ecotoxicity of the effluents will be analysed.

Samples are intended to be analyzed for PFAS using methodologies based on EPA Method 537.1 or EPA Method 533 as well as for Total Oxidisable Precursor (TOP) Assay. Therefore, DND should arrange for the sampling and sample analysis with a lab accredited for the analysis of PFAS in environmental matrices. Also, the sampling should be conducted in accordance with the EPA methods using the sample containers and sampling techniques provided/instructed by the testing lab.

2.2 Sampling protocols

Sampling container and equipment

Sampling should be conducted in accordance with the EPA methods using the sample containers and equipment required by the testing lab. The plastic containers that meet the requirements of the testing lab and the EPA methods should be used, and any equipment made of materials (e.g. glass) allowing PFAS to adhere to the surface should not be used for the sampling. Pre-cleaned sample containers and any necessary coolers, sample labels, as well as a chain of custody form should be obtained from the testing lab. The samples shall be placed in a cooler maintained at the required temperature (e.g. $4 \pm 2^{\circ}\text{C}$) by the EPA methods or the testing lab.

Sample identification and logging

A label shall be attached to each sample container with a sample identification. A sample log shall document the sampling details and visual description of the material.

PPE

The sampler should wear PPE (i.e. Level D is anticipated to be appropriate for most of sampling) and nitrile gloves while collecting, preparing and processing samples as well as handling sample containers.

2.3 Sampling

The DND trial decontamination protocol includes sampling of the foam and effluent throughout the decontamination procedure in CFB Greenwood, NS, as indicated in Table 1. In each sampling event, samples shall be collected using the sample containers provided by the testing lab for PFAS and Limonene analysis.

Blank foam sampling (SAMPLE B0)

As planned in the trial protocol, a blank foam sample (SAMPLE B0: Ansilite 3%) should be collected from the vehicle as charged in the vehicle container/tank. This will set the benchmark for the initial concentration of PFAS compounds with respect to the effluents and help assess the efficacy of the decontamination process. Collecting the blank sample directly from the vehicle tank will also ensure to capture any aging impact on the PFAS composition of the foam concentration stored inside the tank under the given condition.

Sampling after RINSE 1 (SAMPLE R1)

It is recommended to collect a sample after RINSE 1 to assess the effectiveness of the cleaning solution (50% V171:/50% water by volume) and to evaluate the concentration of PFAS.

Sampling after RINSE 3 (SAMPLE R3)

As planned in the trial protocol, a sample should be obtained after RINSE 3.

Sampling after RINSE 4 (SAMPLE R4)

As planned in the trial protocol, a sample should be obtained after RINSE 4.

Sampling after RINSE 5 (SAMPLE R5)

As planned in the trial protocol, a sample should be obtained after RINSE 5, from which the actual effectiveness of the decontamination will be assessed based on the level of PFAS concentration to be analysed.

2.4 Lab analysis

The collected samples shall be sent to an accredited testing lab for the following analyses:

- **PFAS Full suite analysis using the EPA methods**
- **PFAS Total Oxidisable Precursor (TOP) Assay**
- **D-Limonene**

Table 2 shows the recommended lab analysis plan for each sample.

Table 2 Lab analysis plan for each sample

Analysis	Sample ID				
	B0	R1	R3	R4	R5
PFAS- Full suite analysis	V	V	V	V	V
PFAS Total Oxidisable Precursor (TOP) Assay	V	V	V	V	V
Limonene	V		V		V

Requirements for PFAS analysis and reporting

The collected samples shall be analyzed in accordance with the following EPA methods:

- Method 533: "Determination of Per- and Polyfluoroalkyl Substances in Drinking Water by Isotope Dilution Anion Exchange Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry"
- Method 537.1: "Determination of Selected Per- and Polyfluorinated Alkyl Substances in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)"

The TOP Assay can assist in detecting PFAS substances that are not analyzable by routine analytical methodology (LC-MS/MS) by converting precursors into detectable substances through oxidation.

The lab analysis report should provide the following:

- Measurements for PFAS analytes (up to 32 analytes including precursors)
- combined concentration of PFOA and PFOS in ng/L, and
- total PFAS (the combined sum of all PFAS concentrations detected)

The PFAS analyte list should include nine suggested PFAS compounds (PFOS, PFOA, PFNA, PFHxS, PFHpA, PFBS, PFBA, PFPeA and PFHxA) as well as other potential PFAS compounds. The lab analysis should report PFAS compounds in the acid form. Also, the testing lab should ensure that the PFAS Chemical Abstracts Service (CAS) number and compound name shown on written reports and/or electronic data deliverables also reflect the acid form of the PFAS compounds.

The suggested reporting limit for the test methods is at least 2-4 nanograms per liter (ng/L), which is equivalent to parts-per-trillion (ppt). This should be reported along with the method detection limit.

Limonene analysis

The collected samples for d-Limonene analysis shall be sent to the testing lab following the instructions provided by the testing lab. Following the testing lab's standard procedure, extract d-limonene from water samples, and carry out the analyses using Gas Chromatography/Mass Spectrometry (GC/MC) to quantify the d-limonene concentrations. Please consult with the NRC Fire Safety Unit if required for assistance for a detailed d-limonene analysis procedure.

3 Post-Decontamination Safety

3.1 Success measures

PFAS concentrations in effluents would be indicative of a successful decontamination process, and even after the decontamination, PFAS residues could remain in the vehicle and dissolve into fluorine free foams replaced. Thus, it is important to identify acceptable PFAS concentrations within non-fluorinated replacement foams. One of the decontamination success measures noted in the DND trial protocol is "Effluent discharge, after the cleaning process, is less than baseline reading¹, and approved by EC as the goal standard." The specific "goal standard" or PFAS concentration threshold within the effluent, however, is not defined in the protocol.

¹ Baseline reading in this context means criterion values since DND has indicated that the baseline reading is the initial PFAS concentration within the original foam concentrate.

It is recommended that DND set two goals to measure the success of the decontamination process. The first goal should identify the percent (%) reduction of PFAS in the decontamination effluent that will be indicative of a successful decontamination process. The second goal should identify the maximum acceptable concentration of PFAS remaining in the vehicle or dissolving in non-fluorinated replacement foams, which will also identify the success of the decontamination process. Although, it is outside of the scope of the work to provide recommendations on concentration limits to be adopted in DND protocol, NRC will provide a review on criteria and their threshold values in the comprehensive report.

3.2 Post-decontamination residue examination

Decontamination can be challenging because fluorinated compounds can settle and adhere to tank and piping surfaces. If the fluorinated foam is not sufficiently removed from these surfaces, fluorinated compounds can leach into and contaminate non-fluorinated replacement foams. This reintroduces a risk to the environment and personnel. Re-cleaning may be necessary to successfully mitigate this risk. As such, the trial protocol should consider this potential and outline a process for examination of equipment post-decontamination.