

LABORATORY-SCALE THERMAL DEGRADATION OF PERFLUORO-OCTANYL SULFONATE AND RELATED SUBSTANCES

TEST SUBSTANCE

Identity: Perfluorooctanesulfonate; may also be referred to as PFOS or $C_8F_{17}SO_3^-K^+$. (1-Octanesulfonic acid, 1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-heptafluoro-, potassium salt, CAS # 2795-39-3). Sample container was a clear glass vial with black plastic screw on cap. Label read " $C_8F_{17}SO_3^-K^+$, 98-0211-3916-1, Lot 217". The purity of this substance is 86.9%. Storage conditions were not described.

Remarks: Thermal degradation of FC-807A and FC-1395 (C_8 perfluorooctyl sulfonamides) was also evaluated as a potential source of PFOS in the environment. FC-807A is a mixture of mono- and di-phosphate esters of fluorochemical alcohols ($HOCH_2CH_2N(R)SO_2C_8F_{17}$). FC-807A had previously been used in production of oil-resistant papers and packaging material. FC-1395 is a urethane polymer to which the fluorochemical alcohol is bound through urethane linkages. FC-1395 had previously been used as a carpet treatment material prior to the production phase-out in December 2000. The sample container for FC-807A was made of clear glass with a metal screw cap. The Label for this substance read "Material FC-807A/8681/BC AS/Time: 11:10/Lot No. 30177/Drum T1/Step/Date: 12-22-2K/Sampled by C. Senior." The sample container for FC-1395 was an amber glass vial with black plastic screw on cap. The label for this substance read "Name: FC-1395/Lot#: 90086/Date: 11/7/00." Storage conditions were not described for FC-807A and FC-1395. Purity was also not noted for these test substances.

METHOD

Method: Method developed by Takahiro Yamada and Philip H. Taylor, Environmental Sciences and Engineering Group, University of Dayton Research Institute, based on the use of batch-charged continuous flow reactors developed at UDRI to study the thermal stability of organic materials (Rubey and Carnes, 1985, Rubey and Grant, 1988).

Method comments: Laboratory-scale study simulating a full-scale hazardous waste incinerator. The System for Thermal Diagnostic Studies (STDS) was used for the incineration study. The instrument consists of several major components: a thermal reaction compartment; a transfer line; an analytical gas chromatograph (GC), a mass selective detector and a computer workstation.

Year completed: Phase I testing began in 2001 and the final report was written and completed in 2003.

GLP: No

Test Overview:

Testing was completed in 3 phases. Phase I tested the initial test protocol and project objectives, Phase II consisted of the method development work, and Phase III revised and applied the test protocol. The overall goal of this study was to determine if incineration is a potential source of perfluoroalkyl sulfonates, e.g., perfluoro-octanyl sulfonates (PFOS), which has been found widespread in wildlife tissue samples

Phase I:

Objectives of Phase I were to determine if C₈ perfluorosulfonamides form combustion products in the form of perfluorooctane sulfonate (PFOS) or a perfluoro precursor of perfluorooctane sulfonate, to determine the extent of conversion of PFOS under conditions representative of hazardous or municipal waste incineration, to identify the major fluorinated combustion products, and to determine if the sulfur present in the PFOS is quantitatively converted to sulfur dioxide and/or thionyl fluoride (SO₂F₂) and sulfuryl fluoride (SO₂F₂) at high temperature, fuel-lean combustion conditions.

The development of the test protocol was based on the use of batch-charged continuous flow reactors developed at UDRI to study the thermal stability of organic materials. Analysis was performed using in-line gas chromatography/mass spectrometry (GC/MS). The analytical focus was identification of stable fluorinated organic intermediates and the quantification of sulfur oxides in an attempt to recover 100% of the initial sulfur in the sample. Sulfur quantification was performed using a mass selective detector (MSD).

Gas-phase thermal stability analysis was performed to determine 1) the temperature needed to gasify sample, 2) if the phase change is the result of evaporation or decomposition, and 3) if a non-volatile residue is deposited by the sample. Then testing was performed to assess whether the gasification products have the ability to be transported under the flow reactor conditions.

The STDS was selected to carry out this incineration study. Methane was chosen as the fuel for samples that were hydrogen deficient. Two temperatures (600 and 900°C) were selected for the study based on preliminary combustion tests.

Phase II:

The purpose of Phase II of the incineration study was to verify gasification of PFOS and transport of PFOS through the UDRI thermal instrumentation system. Recovery efficiencies and detection limits for sulfur compounds (SO_2 , SOF_2 , and SO_2F_2) and perfluorooctane sulfonyl fluoride (POSF, a precursor of PFOS) were determined through analysis using a GC/MS system. Measurements were performed in duplicates.

Hexafluoropropene (HFP) was selected as the surrogate volatile fluorocarbon. Recovery efficiencies and detection limits of volatile $\text{C}_1\text{-C}_4$ fluorocarbons were also established. A quantitative method of sampling the reactor effluent was established. ORBO PUF cartridges were used for sampling PFOS and precursors from the reactor effluent.

Linear Fit Equations and Detection Limits

Sample Name	Linear Fit (Y: peak area, X: concentration (ppm))	R	Detection Limit (ppm)
SO_2	$Y = 5.8813\text{E}3 * X - 3.8541\text{E}5$	0.9971	78.5
SOF_2	$Y = 8.3335\text{E}3 * X - 7.0267\text{E}4$	0.99941	30.3
SO_2F_2	$Y = 1.0331\text{E}4 * X + 1.8273\text{E}6$	0.99708	20.1
POSF	$Y = 1.0423\text{E}5 * X - 8.4043\text{E}5$	1.0	14.1
HFP	$Y = 1.4975\text{E}4 * X - 2.8253\text{E}6$	0.9997	3.9

Transport Efficiency

System Transport				Direct Injection			Efficiency
Peak Area				Peak Area			(%)
Sample	1 st	2nd	AVG (1)	1 st	2 nd	AVG (2)	(1)/(2)×100
SO_2	9130332	8980717	9055525	11952302	11762267	11857285	76.4
SOF_2	25244352	25203780	25224066	24862639	24773683	24818161	101.6
SO_2F_2	86850304	85572809	86211557	84435720	79738316	82087018	105.0
POSF	1280370	1228718	1254544	1064431	1067947	1066189	117.7
HFP	148679354	145606343	147142849	148372504	142271896	145322200	101.3

Phase III:

Phase III consisted of 8 different tests (SO_2 Transfer Efficiency Tests, Laboratory Spike Analysis for PFOS, Heated Blank Combustion Test, Combustion Tests for PFOS and two C_8 perfluorosulfonamides, Heated Blank Combustion Test (repeat), Transfer Efficiency Test for PFOS, Sulfur Recovery Analysis as SO_2 , and Extracted Ion Analysis). In-line and off-line GC/MS analysis was conducted on the combustion gases. The off-line GC/MS analysis was used in replicate runs in order to capture the

more volatile compounds and because there were resolution issues with the in-line sampling approach for the sulfur recovery rate. PUF (polyurethane foam) collection of the reactor effluent and chemical extraction of the reactor and associated transfer lines was completed. PUF cartridges and extracts were delivered to 3M for analysis of PFOS by LC/MS.

SO₂ Transfer Efficiency Tests were performed to confirm results from phase II. The Heated Blank Combustion Tests were used to examine system contamination. Four analyses were conducted for the Heated Blank Combustion test (in-line GC/MS analysis, PUF collected off-gas sample analysis, off-line GC/MS analysis using Tedlar bag, and reactor/transfer line system extraction using methanol).

RESULTS

SO₂ Transfer Efficiency Test:

SO₂ transport efficiency was 83.7% in Phase III, slightly higher than the Phase II results, 76.4%, which gives average value of 80.1%. The transport efficiency for SOF₂ was 100% in Phases II and III.

Laboratory Spike Analyses for PFOS:

Phase III laboratory spikes included fourteen 1 µg and twelve 10 µg spikes of PFOS (as the PFOS anion) into the PUFs (see Appendix 5.), and a single 1 µg PFOS spike (as the PFOS K salt) of the tubing used as the reactor (combustion chamber) and transfer line. For the PUF spikes, the spiking solution was injected just below the surface, and allowed to dry for 30 minutes, and then extracted as in the test procedure. An average of 82% of the PFOS was recovered from the 1 µg PUF spikes with a relative standard deviation of 10%. An average of 92% of the PFOS was recovered from the 10 µg PUF spikes with an RSD of 7%. The single combustion chamber spike involved injecting a 1 µg sample of PFOS into the reactor as a 1 µg/µl solution in HPLC grade methanol, and drying by blowing with high purity nitrogen. After drying, the transfer line was assembled to the reactor and the 1.1 mL volume assembly was extracted by pumping of 5.5 mL of methanol through the assembly in two sequential washings. The total PFOS recovery from this reactor/transfer line spike was 188%. This single spike result suggests that an error likely occurred during preparation, extraction or analysis. Nevertheless, this spike result confirms that PFOS can be extracted from reactor/transfer lines.

PFOS Laboratory Reactor/Transfer Line Spike Analysis:

Sample Extracts	PFOS ⁻ K ⁺ (pg/µl)	PFOS ⁻ K ⁺ (µg)
PFOS 1 st	232	1.6

Extracts		
PFOS 2 nd Extracts	40.5	0.28

Heated Blank Combustion Analysis:

The flow profile and carrier flow volume used for the heated blank analyses at 600°C and 900°C are given in the following two tables.

Flow Rate Profile for Heated Blank Analysis at 600°C

Time Period (sec)	Reactor Flow Rate (ml/min)	Pyroprobe Flow Rate (ml/min)	Total Flow Rate (ml/min)	Total Volume (ml)	Sampled Volume ^d (ml)
0 – 120	10.5	0.80	11.30	22.60	20.60
120 – 130	10.5	0.80 → 4.63 ^a	11.30 → 14.63	2.16	1.99
130 – 140	10.5	4.63	15.13	2.52	2.35
140 – 160	9.03 (He) ^b	4.53 (He) ^c	13.56	4.52	4.19
Total Volume (ml)				31.80	29.13

^a Linear increase (approximate). ^{b,c} Switched to helium for sweep. ^d Sampled volume for PUF and Tedlar bag collection.

Flow Rate Profile for Heated Blank Analysis at 900°C

Time Period (sec)	Reactor Flow Rate (ml/min)	Pyroprobe Flow Rate (ml/min)	Total Flow Rate (ml/min)	Total Volume (ml)	Sampled Volume ^d (ml)
0 – 150	7.60	0.70	8.30	20.75	18.25
150 – 160	7.60	0.70 → 4.63 ^a	8.30 → 12.23	1.71	1.54
160 – 170	7.60	4.63	12.23	2.04	1.87
170 – 190	6.54 (He) ^b	4.53 (He) ^c	11.07	3.69	3.36
Total Volume (ml)				28.19	25.02

^a Linear increase (approximate). ^{b,c} Switched to helium for sweep. ^d Sampled volume for PUF and Tedlar bag collection.

There was no measurable contamination for either temperature (600 and 900°C) for both in-line and off-line GC/MS analysis. A small amount of PFOS (0.08µg) was detected in the reactor/transfer line extract in the first heated blank combustion test. The amount of PFOS extracted in the second heated blank combustion test was below detection limits.

Combustion Tests for PFOS and two C₈ perfluorosulfonamides:

PFOS in Transfer-Line Extracts: Table 5.4.1.1 of the report shows that in the four sequential combustion tests on PFOS, a total of 1.83 mg of PFOS were gasified in the pyroprobe.

$$0.45+0.38+0.5+0.5 = 1.83 \text{ mg}$$

Tables 5.4.2.1 and 5.4.3.1 of the report show that, in the four sequential combustion tests on FC-807A and FC-1395, totals of 2.16 mg and 2.02 mg, respectively, of product dry mass were gasified in the pyroprobe. For example, for FC-807A:

$$0.59+0.59+0.45+0.53 = 2.16 \text{ mg}$$

The gasified samples then passed through heated (260°C) transfer lines to a combustion chamber. In the combustion chamber the volatilized gases mixed with near-stoichiometric amounts of air (FC-807A and FC-1395) or excess air (PFOS) and were exposed to 600 or 900°C for about 2 seconds. From here, the combustion products passed through 260°C transfer lines to ambient temperature polyurethane foam containing cartridges or Tedlar bags.

Following the combustion tests at 900°C, two sequential methanol extracts of the second half of the combustion chamber and the transfer lines downstream of the combustion chamber were analyzed for PFOS. The following table shows the concentration and amounts of PFOS (as the K salt), detected in the chamber/transfer line. This is the amount that accumulated in the chamber/transfer line from the four sequential tests, two at 600°C and two at 900°C, for each product. The total amount of PFOS detected in the extracts was equivalent to about 0.04% of the PFOS gasified in each of the four tests or, as shown in the following equation, 0.009% of the 1.83 mg of PFOS gasified in the four sequential tests.

$$100 \times (0.00011+0.00005)/1.83 = 0.009\%$$

Following the four sequential combustion tests on each of the two perfluorooctylsulfonamide products, PFOS was below detection limits in the chamber/transfer line extracts. Based on the detection limits, the amount of PFOS that could have been present in each of the two sequential extract was less than 0.035 µg. Based on the stoichiometry given in the Table 1 on page 7 of Appendix 4, the 2.16 mg and 2.02 mg of FC-807A or FC-1395 solids gasified in the four sequential tests could have formed a maximum of 1.87 mg or 1.55 mg of PFOS (as the K salt), respectively. For example, for FC-807A:

$$2.16 \text{ mg} \times 0.519/0.600 = 1.87 \text{ mg}$$

where 0.519 and 0.600 are the fractions of fluorine in FC-807A and PFOS (as the K salt). The following shows how this calculation was done for PFOS using the data in Table 1, page 7 of Appendix 4:

$$\text{Fraction F in PFOS}^-\text{K}^+ = (19 \cdot 17) / (12 \cdot 8 + 19 \cdot 17 + 16 \cdot 3 + 32 \cdot 1 + 39.1 \cdot 1) = 0.600$$

Where 19, 12, 16, 32 and 39.1 are the molecular weights of fluorine, carbon, oxygen, and potassium, respectively.

Thus, this analysis shows the amount of PFOS in the chamber/transfer lines was less than 0.004% and less than 0.005% of the maximum amount of PFOS that could have been formed from FC-807A or FC-1395 respectively. For example, for FC-807A:

$$100 \times (< 0.000035 + < 0.000035) / 1.87 = < 0.004\%.$$

Transfer-Line Extraction Results:

PFOS		
Extraction	PFOS⁻K⁺ (pg/μl)	PFOS⁻K⁺ (μg)
1	15.4	0.11
2	8.61	0.059
FC-1395		
Extraction	PFOS⁻K⁺ (pg/μl)	PFOS⁻K⁺ (μg)
1	<5.00	<0.035
2	<5.00	<0.035
FC-807A		
Extraction	PFOS⁻K⁺ (pg/μl)	PFOS⁻K⁺ (μg)
1	<5.00	<0.035
2	<5.00	<0.035

PFOS in PUF Extracts: The calculation below, using data in the following table, shows that the amount of PFOS making it through the combustion system and extracted from the PUFs was equivalent to less than 0.5% of the 0.45 mg of PFOS gasified at 600 °C.

$$100 \times (0.00062 + 0.0016) / 0.45 = 0.49\%$$

The data in this table also show that about 0.07 % of the 0.5 mg of PFOS gasified at 900°C makes it through the system and was extracted from the PUFs.

$$100 \times (0.00011 + 0.00022) / 0.5 = 0.066\%$$

PUF Extraction Results:

Substance	Temp (Degrees C)	Extraction	PFOS⁻K⁺ (pg/μl)	PFOS⁻K⁺ (μg)
PFOS	600	1,2	25.1, 64.0	0.62, 1.6
	900	1,2	4.31, 9.01	0.11, 0.22
FC-1395	600	1,2	<5.00, <5.00	<0.12, <0.12
	900	1,2	<5.00, <5.00	<0.12, <0.12

FC-807A	600	1,2	<5.00, <5.00	<0.12, <0.12
	900	1,2	<5.00, <5.00	<0.12, <0.12

The PUF Extraction Results table (above) also shows that, following combustion of perfluoroalkylsulfonamide compounds, no PFOS was detected in PUF extracts at concentrations above the lower limit of quantification (LLOQ). Based on the detection limits, the amount extracted from each of the two sequential PUFs was less than 0.12 µg. Of the 0.45 and 0.55 mg of FC-807A and FC-1395 gasified at 900°C, combustion could have formed a maximum of 0.39 mg or 0.42 mg of PFOS (as the K salt), respectively. For example for FC-807A:

$$0.45 \times 0.519 / 0.6 = 0.39 \text{ mg}$$

where 0.519 and 0.600 are the fractions of fluorine in FC-807A and PFOS K salt respectively. The following equations thus show that the amount of PFOS in the combined PUF extracts of the 900°C tests was less than 0.07% of the PFOS that could have been formed from both FC-807A:

$$100 \times (<0.12 + <0.12) / 390 = <0.062\%$$

and FC-1395:

$$100 \times (<0.12 + <0.12) / 420 = <0.057\%.$$

Transport Efficiency Tests for PFOS:

Three types of tests were conducted to examine transport efficiency. These tests were done to validate that PFOS could be detected if it were generated during the combustion of fluorochemicals in the test system. The first test examines the transfer efficiency of samples gasified in the pyroprobe and transported through the reactor to the PUF cartridges. The second test investigates the possibility that the volatilized PFOS condensed on the walls of the pyroprobe/reactor transfer line. The third test examines how much PFOS volatilized in the combustion reactor could be transferred to the PUFs and how much would condense in the reactor/transfer line tubing.

In the 1st Transfer efficiency test 0.48 mg of PFOS was volatilized. The following table shows that no detectable amount of the volatilized PFOS was recovered from the PUF cartridge. This result indicates that the sample was either thermally dissociated in the pyroprobe chamber or the gasified sample was completely condensed in the pyroprobe/reactor transfer line tubing.

PUF Extraction Results for 1st Transfer Efficiency Test

Sample	PUF Extracts	PFOS ⁻ K ⁺ (pg/μl)	PFOS ⁻ K ⁺ (μg)
PFOS	1 st	<5.00	<0.12
	2 nd	<5.00	<0.12

The next two tables show that, in the 2nd transfer efficiency test, measurable amounts of the 0.47 mg of PFOS that was volatilized survive pyrolysis conditions of the pyroprobe, and collected in the heated transfer lines. However, no detectable amount of PFOS survives transit to the PUF sampling cartridge.

Methanol Extraction Results for 2nd Transfer Efficiency Test

Sample	Extracts	PFOS ⁻ K ⁺ (pg/μl)	PFOS ⁻ K ⁺ (μg)
PFOS	1 st	897	21
	2 nd	<10.0	<0.24

PUF Extraction Results for 2nd Transfer Efficiency Test

Sample	PUF Extracts	PFOS ⁻ K ⁺ (pg/μl)	PFOS ⁻ K ⁺ (μg)
PFOS	1 st	<10.0	<0.25
	2 nd	<10.0	<0.25

The following two tables show results of the 3rd transfer efficiency test. In this test, PFOS was volatilized in the combustion chamber (reactor) by heating it to 575°C. The first table shows that measurable amounts of volatilized PFOS passed from the reactor to the PUFs. Calculations using data from the first of these tables show that 12.8% of the 0.46 mg of PFOS volatilized in He reached the PUF, as did 5.2% of the 0.48 mg of PFOS volatilized in air.

$$100 \times (0.058 + 0.0011)/0.46 = 12.8\%$$

$$100 \times 0.025/0.48 = 5.2\%$$

The second table below shows methanol extract data obtained in the 3rd transfer efficiency test. These were from extracts of: 1) the 260°C reactor/transfer line tubing and 2) the room temperature valve and associated transfer line tubing just upstream of the PUF cartridges. Calculations using data from this second table show that larger amounts of PFOS (5.6% air, 39.3% He) accumulated in the reactor/transfer lines upstream of the PUF cartridges. The majority of the PFOS accumulated in

the portion of the transfer line heated to 260°C, suggesting that this compound could condense, or was in a particulate form, at this temperature.

$$100 \times (0.024 + 0.00045 + 0.0024 + 0.000079)/0.48 = 5.6\%$$

$$100 \times (0.171 + 0.0019 + 0.0077 + 0.00035)/0.46 = 39.3\%$$

3rd Transfer Efficiency Test PUF Extraction Results

Sample	Carrier Gas	Cartridge	PFOS ⁻ K ⁺ (pg/μl)	PFOS ⁻ K ⁺ (μg)
PFOS	He	1 st	2330	58
		2 nd	44	1.1
	Air	1 st	997	25
		2 nd	<10.0	<0.12

3rd Transfer Efficiency Test Reactor/Valve Transfer Line Extraction Results

Sample	Gasification	Location	Extracts	PFOS ⁻ K ⁺ (pg/μl)	PFOS ⁻ K ⁺ (μg)
PFOS	Air	Reactor ¹	1 st	1908	24
			2 nd	35.4	0.45
		Valve ²	1 st	696	2.4
			2 nd	22.8	0.079
	He	Reactor ¹	1 st	13530	171
			2 nd	150	1.9
		Valve ²	1 st	2218	7.7
			2 nd	102	0.35

Notes:

1. The location labeled "Reactor" included the second half of the combustion chamber (575°C) and the heated (260°C) transfer line leading from the combustion chamber to the valve.
2. The location "Valve," included the valve and the transfer line leading from the valve to the PUF. The valve and this transfer line were at room temperature during the test.

Sulfur Recovery Rate:

Based on the in- and off-line GS/MS analyses, sulfur was found mainly as SO₂. No SOF₂ and SO₂F₂ were detected. Because the SO₂ peaks using the off-line GC/MS system were much sharper than SO₂ peaks observed using in-line GC/MS, it was decided to use off-line GC/MS analytical results to quantitatively analyze the sulfur recovery as SO₂.

SO₂ Calibration Results Using the off-line PLOT Column

Conc. (ppm)	Mol. #	Area 1	Area 2	Area (Avg)
1000	4.09E-08	7191079	6980771	7085925
700	2.86E-08	4414365	4366705	4390535
400	1.63E-08	2304594	2295497	2300046
100	4.09E-09	425431	416699	421065

In the SO₂ transfer efficiency test conducted using the off-line analysis approach-the average recovery rate was 75.6%. This is very similar to the recovery rates obtained from the in-line analysis, i.e. 83.7 and 76.4%, suggesting that the lack in 100% recovery is due to sample losses in the combustion system and not the sampling and analysis procedures.

Standard SO₂ Transfer Efficiency

Volume (ml)	Area	Calculated Mol. #	# of Mol. Used	Transfer Efficiency (%)
22.13	10591947	1.36E-06	1.63E-06	83.4
22.13	8515987	1.11E-06	1.63E-06	67.8
			Average	75.6

Extracted Ion Analysis:

The following ions (51-CF₂H, 69-CF₃, 119-C₂F₅, and 67-SOF) were among those extracted from the total ion chromatograms from tests on PFOS, a lower molecular weight perfluoroalkylsulfonate (designated as PFXS), and the C₈ perfluorosulfonamides. This was done for both the in-line and off-line GC/MS analyses in order to analyze for the presence of perfluorinated and sulfonate-containing intermediates. Importantly, the inability to detect the 67-SOF ion in any of these analyses suggested low levels or an absence of perfluoroalkylsulfonyl compounds in the combustion products. This result strongly suggests that incineration is not likely to be a significant source of compounds that are environmental precursors of PFOS or other perfluoroalkyl sulfonates. Extracted ion chromatograms for 51, 69, and 119 ions, when evaluated in conjunction with hydrogen flame ionization detector (HFID) chromatograms, suggest that tri- and tetrafluoromethane and hexafluoroethane were likely combustion products.

Due to an apparent loss of sensitivity of the in-line MS detector during the PFOS test, the extracted ion analysis generated from PFOS combustion did not indicate the presence of fluorinated combustion products, unlike the results for all other compounds tested. An evaluation of the PFOS HFID data in conjunction with the HFID and extracted ion analysis data for a lower molecular weight perfluoroalkylsulfonate (PFXS) strongly

suggested that PFOS combustion also generated some volatile (low MW) fluorochemical combustion products. The PFXS data showed fluorochemicals were present at the same retention time as HFID peaks. Additionally, the retention times of the HFID response from PFOS and PFXS combustion were nearly identical. Thus, the same fluorochemical combustion products likely formed from these two different compounds.

The following two tables show that the HFID peak area at 900°C is approximately 1% of that at 600°C. This result suggests nearly complete destruction of fluorochemicals combustion products at 900°C.

Integrated HFID Peak Area of PFXS and PFOS at 600°C

Sample	Peak Area	Net Amount of Gasified Sample (mg)
PFXS	1190193	0.52
PFOS	3547614	0.38

Integrated HFID Peak Area of PFOS at 900°C

Sample	Peak Area	Net Amount of Gasified Sample (mg)
PFOS	39041	0.50

CONCLUSIONS

This laboratory study used a system designed to simulate the thermal degradation conditions likely to occur during municipal or hazardous waste incineration. When perfluorooctane sulfonamide containing products (FC-1395 and FC-807A) were exposed to these conditions, SO₂ recoveries were 100±25% and LC/MS analysis showed that no quantifiable amounts of PFOS (<0.07% of that stoichiometrically possible) were released from the system. The SO₂ results suggest that the perfluorosulfonamides were substantially destroyed and the LC/MS results strongly suggest that incineration of perfluorooctane sulfonamides would not release PFOS to the environment. Furthermore, mass spectral extracted ion analysis of the emissions, showed an absence of 67-SOF ions. These ions are indicative of perfluoroalkylsulfonyl compounds, which could potentially transform to PFOS in the environment. The absence of the 67-SOF ions suggests that the carbon-sulfur bond was completely destroyed (and did not reform) in the combustions tests. This result suggests that environmental transformation of combustion products to form PFOS is also unlikely.

When PFOS was exposed to this combustion system, sulfur recoveries varied from 50 to 60% and LC/MS analysis showed that only a small fraction of the PFOS that was volatilized made it through the system. The authors speculate that low sulfur recoveries were likely due to condensation within the apparatus. The fraction of PFOS passing through the system to the PUFs decreased as the temperature of the combustion chamber increased. At 600 °C, about 0.5% of the gasified PFOS made it completely through the system and was collected on the PUFs. When the combustion chamber temperature was increased to 900°C, the amount of PFOS collected on the PUFs was decreased to about 0.07%.

C₁ or C₂ fluoroalkanes (likely products are either CHF₃, CF₄, or C₂F₆), 1,1-difluoroethene (PFOS only) and 1,2-difluoroethene (FC-1395 only) were the only highly fluorinated compounds observed in the system effluent. Fluorobenzene was also detected (FC-1395 and FC-807A only). No higher molecular weight fluorinated polycyclic aromatic hydrocarbons, and no perfluorooctane sulfonyl precursors of PFOS were present in the system effluent at detectable levels.

The report states that the data from this laboratory-scale incineration study indicates that properly operating full-scale incineration systems can adequately dispose of PFOS and the C₈ perfluorosulfonamides. Incineration of these fluorinated compounds is not likely to be a significant source of PFOS into the environment. With the exception of stable C₁ and C₂ fluorocarbons, fluorinated organic intermediates are unlikely to be emitted during the incineration of PFOS or perfluorooctane sulfonamides.

Submitter: 3M Company, Environmental Laboratory, P.O. Box 33331, St. Paul, Minnesota, 55133

DATA QUALITY

Reliability: Klimisch ranking 2. Purity/Composition of FC-807A and FC-1395 were not properly characterized in the study.

REFERENCES

Study conducted at the request of 3M Company by University of Dayton Research Institute.

OTHER

Last changed: 7/03/03