AIR/WATER PARTITION COEFFICIENT

TEST SUBSTANCE

Identity: Perfluorooctanesulfonate; may also be referred to as PFOS or FC-95. (1-Octanesulfonic acid, 1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-heptadecafluoro-, potassium salt, CAS # 2795-39-3)

Remarks: The test substance is a white powder. No information was recorded on the purity.

METHOD

Method: There is no standardized methodology used to determine this value for regulatory purposes. The experiment was designed by Dr. Richard Purdy of 3M's Environmental Laboratory and Don Mackay of D. Mackay Environmental Research Limited.

GLP (Y/N): No

Year completed: 1999

Remarks: The following method was devised and used:

1. Weigh approximately 0.01 gram of the test substance directly into a tared 250-mL Pyrex® beaker. Record weight.
2. Transfer 200 mL of NANOpure® water into the beaker using a Class A glass volumetric pipet.
3. Prepare solvent blank sample. Using a gas-tight syringe, transfer a 250 µL aliquot of NANOpure® water into a 25-mL Class A glass volumetric flask partially filled with 50% methanol / 50% ammonium acetate buffer reagent. Bring to volume with 50% methanol / 50% ammonium acetate buffer reagent. Ampulate in an amber glass autosampler vial.
4. Mix and sonicate the test substance in water sample (50 µg test substance/mL target nominal concentration) for approximately 10 minutes to ensure dissolution of the test material.
5. Prepare the control sample. Transfer a 250 µL aliquot of the test substance in water sample into a 25-mL Class A glass volumetric flask partially filled with 50% methanol / 50% ammonium acetate buffer reagent. Bring to volume with 50% methanol / 50% ammonium acetate buffer reagent. Ampulate in an amber glass autosampler vial.

6. Place the test substance in water sample beaker on a hotplate and bring solution to a boil. After approximately 10 mLs (5%) of water has evaporated, remove beaker from hotplate and cool to room temperature in an ice-water bath. Transfer contents of sample into a graduated cylinder and record actual volume.
7. Process a 250 µL aliquot of sample as described above for solvent blank and control samples.
8. Return sample to original beaker and bring sample to boil.
9. Repeat steps 6-8 until sample has evaporated to 100 mLs. Submit all ampulated samples for LCMS analysis.

RESULTS

K_{aw}: 0*
Temperature °C: Not recorded.

Remarks: *Don Mackay provided the following interpretation of the analytical results:

> "As I interpret the lab results they established an initial concentration of 50 mg/L in 200 mL water then distilled off 10 mL aliquots and analyzed the residue. They then calculated the percentage of the original test substance present which remained in the beaker unevaporated. These 'percentage recoveries'; ranged from 136 to 105 with no real trend. I conclude that the test substance did not evaporate to any measurable extent. This is a very sensitive method of measuring low air-water partition coefficients. It can be shown that if water and the solute evaporate equally (e.g., the contents do not change in composition as would occur with an azeotrope) then K_{aw} or H is identical for water and the solute. For water, H is approximately 2400 Pa (approximately 20°C) divided by 55000 mol/m^3 or 0.044 Pa m^3/mol or a K_{aw} of about 2 x 10^{-5}. The test substance must thus have a K_{aw} considerably less than this, i.e. less than 2 x 10^{-6}. It is thus essentially non-volatile from aqueous solution. This is probably because of its ionic nature. The simple expedient is to assign it a K_{aw} of zero, i.e. is a type 2 involatile chemical in our nomenclature."

DATA QUALITY

Reliability: Klimisch ranking 2. Data has limited reliability. Sample purity was not noted. Study temperature was not recorded.

CONCLUSIONS

Testing indicates this substance is essentially non-volatile from aqueous solution.

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

REFERENCES

Study conducted at the request of 3M Company by Wildlife International, Ltd. of Easton, Maryland.

Study review by Don Mackay of D. Mackay Environmental Research Limited,

OTHER

Last changed: 5/1/00

INTERNAL CORRESPONDENCE/3M

TO: Dale Bacon – Env Lab 2-3E-09
Craig Burton – FC Tech. Center 236-3C-89
Mike Nash- OGC 220-12E-02
Bill Weppner – Chem EHS&R 236-1B-10

CC: Rich Purdy – Env Lab 2-3E-09
Joseph Fiksel – Battelle Columbus
Denise Appleton – Env. Lab 2-3E-09
Janine Seiffert – Env. Lab 2-3E-09

FROM: SUSAN A. BEACH

SUBJECT: Testing Results: Air-Water Partition Coefficient (K_{AW}) for PFOS

DATE: March 19, 1999

Wildlife International conducted a study to estimate the air-water partition coefficient (K_{AW}) for PFOS. There is no standardized methodology used to determine this value for regulatory purposes; thus it was conducted as a non-GLP study. The experiment was designed by Rich Purdy and Don Mackay. Wildlife International provided Rich and Don with analytical results; Don provided the interpretation below:

"As I interpret the lab results they established an initial concentration of 50 mg/L in 200 mL water then distilled off 10 mL aliquots and analyzed the residue. They then calculated the percentage of the original PFOS present which remained in the beaker unevaporated. These "percent recoveries" ranged from 136 to 105 with no real trend. I conclude that the PFOS did not evaporate to any measurable extent. This is a very sensitive method of measuring low air-water partition coefficients. It can be shown that if water and the solute evaporate equally (i.e. the contents do not change in composition as would occur with an azeotrope) then K_{AW} or H is identical for water and the solute. For water, H is approximately 2400 Pa (approximately 20°C) divided by 55000 mol/m^3 or 0.044 Pa m^3/mol or a K_{AW} of about 2 x 10^{-5}. PFOS must thus have a K_{AW} considerably less than this, i.e. less than 2 x 10^{-6}. It is thus essentially non-volatile from aqueous solution. This is probably because of its ionic nature. The simple expedient is to assign it a K_{AW} of zero, i.e. it is a type 2 involatile chemical in our nomenclature".

Please see the attached analytical results for more information.

Questions should be directed to Rich Purdy.

Regards,

Susan A. Beach

Attachment (1)

Wildlife International Ltd.

8598 Commerce Drive ● Easton, Maryland ● 21601
Phone: (410) 822-8600 ● FAX: (410) 822-0632

FAX TRANSMISSION

If this transmission is not received correctly or if pages are missing, please call Wildlife International Ltd. at (410) 822-8600

FAXED

Date: Thurs, January 14, 1999 Fax Number: (651) 778-6176 3 page(s) follow this cover sheet.

PLEASE DELIVER TO: Rich Purdy
3M Corporation
3MEnvironmental Technology and Safety Services

From: Raymond L. Van Hoven
Scientist

NOTES:

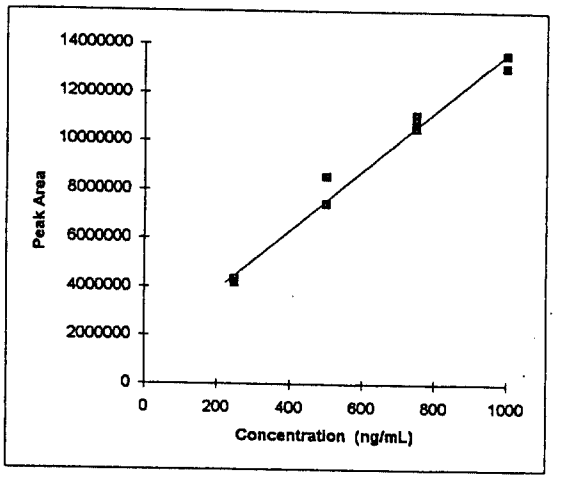
Rich:

Enclosed you will find a completed data summary the PFOS air-water partition coefficient experiment including the data summary, method outline, and instrumental condition summary. Please do not hesitate to contact me to discuss the findings, or to provide any additional experimental details. I look forward to speaking with you in the near future.

000099

WILDLIFE INTERNATIONAL, LTD.
PFOS
Air-Water Partition Coefficient
Sequence I.D.: 12JAN99
Project No.: 454C-101
Date of Analysis :01/12/99

Sample Name	Vial	(ng/mL)	Peak Area	Fit
4675A-001A4	1	250	4155837	222.9372
4675A-001A3	2	500	7388823	488.0412
4675A-001A2	3	750	10491144	742.4307
4675A-001A1	4	1000	13024361	950.1538
4675A-001A4	9	250	4246855	230.4007
4675-001A2	13	750	11036778	787.1725
3736-001A4	18	250	4308786	235.4790
3736-001A3	19	500	8493724	578.6428
3736-001A2	20	750	10834410	770.5784
3736-001A1	21	1000	13561071	994.1639
		12195.2	1437081.661	
		537.33	355408.849	
		0.985	473031.497	
		515.1	8.000	
		1.1526E+14	1790070380970	
		#N/A	#N/A	



Y-intercept	1437082
SLOPE	12195
R2	0.9847

1-13-99

Sample Identification	Nominal Concentration (ug/mL)	Vial No.	Peak Area	Initial Volume (mL)	Final Volume (mL)	Dilution Factor	Y-Evaluate (ng/mL)	Analytical Result (ug/mL)	Percent Recovery (%)
454C-101-REB-1	0.0	5	178690	0.250	25.0	100.000	<250	<25.0	NA
454C-101-AWP-1	50.00	6	9753678	0.250	25.0	100.000	681.9587	68.1959	136.4
454C-101-AWP-2	52.63	7	8606177	0.250	25.0	100.000	587.8639	58.7864	111.7
454C-101-AWP-3	57.14	8	8811737	0.250	25.0	100.000	604.7197	60.4720	105.8
454C-101-AWP-4	60.61	10	9642044	0.250	25.0	100.000	672.8047	67.2805	111.0
454C-101-AWP-5	64.94	11	10220321	0.250	25.0	100.000	720.2233	72.0223	110.9
454C-101-AWP-6	69.44	12	11137255	0.250	25.0	100.000	795.4116	79.5412	114.5
454C-101-AWP-7	75.63	14	11133423	0.250	25.0	100.000	795.0974	79.5097	105.1
454C-101-AWP-8	80.65	15	11851738	0.250	25.0	100.000	853.9990	85.3999	105.9
454C-101-AWP-9	87.72	16	13073257	0.250	25.0	100.000	954.1632	95.4163	108.8
454C-101-stability	50.00	22	7869398	0.250	25.0	100.000	527.4482	52.7448	105.5

Y-evaluate (ng/mL) = ((Peak area-y-intercept)/slope)
Analytical result (ng/mL) = (Y-evaluate * dilution factor)
Dilution factor =(Final volume/initial volume)
Percent recovery = [analytical result (ng/mL)/nominal concentration (ng/mL)] x 100
All spreadsheet calculations performed using EXCEL 5.0 V in the full precision mode.

CERTIFIED COPY
OF THE ORIGINAL

INITIALS 1-12-99 DATE

Original located w/ data pack for project 454C-101

Wildlife International Ltd.

METHOD OUTLINE FOR THE DETERMINATION OF PFOS AIR-WATER PARTITION COEFFICIENT

Project Number:454C-101

1. Weigh appproximately 0.01 gram of PFOS (WIL# 4675A) directly into a tared 250-mL Pyrex® beaker. Record weight.

2. Transfer 200 mL of Nanopure® water into the beaker using a Class A glass volumetric pipet.

3. Prepare solvent blank sample. Using a gas-tight syringe, transfer a 250 µL aliquot of Nanopure® water into a 25-mL Class A glass volumetric flask partially filled with 50% methanol/50% ammonium acetate buffer reagent. Bring to volume with 50% methanol/50% ammonium acetate buffer reagent. Ampulate in an amber glass autosampler vial.

4. Mix and sonicate the PFOS in water sample (50µg PFOS/mL target nominal concentration) for approximately 10 minutes to ensure dissolution of the test material.

5. Prepare the control sample. Transfer a 250 µL aliquot of the PFOS in water sample into a 25-mL Class A glass volumetric flask partially filled with 50% methanol/50% ammonium acetate buffer reagent. Bring to volume with 50% methanol/50% ammonium acetate buffer reagent. Ampulate in an amber glass autosampler vial.

6. Place the PFOS in water sample beaker on a hotplate and bring solution to a boil. After approximately 10 mLs (5%) of water has evaporated, remove beaker from hotplate and cool to room temperature in an ice-water bath. Transfer contents of sample into a graduated cyllinder and record actual volume.

7. Process a 250 µLaliquot of sample as described above for solvent blank and control samples.

8. Return sample to original beaker and bring sample to boil.

9. Repeat steps 6-8 until sample has evaporated to 100 mLs. Submit all ampulated samples for LCMS analysis.

LC/MS Operational Parameters for the Determination of PFOS Air-Water Partition Coefficient

MASS SPECTROMETER:	Perkin-Elmer Sciex API 100 LC Mass Spectrometer
ION SOURCE:	Perkin-Elmer Turbo Ionspray
ION POLARITY:	Negative
ION SOURCE VOLTAGE:	5200 V
ION SOURCE TEMPERATURE:	450 ^{0}C
ION MONITORED:	498.6 amu
LIQUID CHROMATOGRAPH:	Hewlett-Packard 1090 Series II
ANALYTICAL COLUMN:	Keystone Betacil C-18 (2 mm ID, 100 mm, 3 µm particle size)
MOBILE PHASE:	65% methanol/35% ammonium acetate buffered water
FLOW RATE:	240µL/min
INJECTION VOLUME:	10 µL
RUN TIME:	8 minutes

DMER

D. Mackay Environmental Research Limited

1664 Champlain Drive
Peterborough, Ontario
Canada K9L 1N6
Tel (705) 740-2911
GST Reg. #R-101420263

3M991

To: Dr. Rich Purdy, 3M
Dr. Joyce ~~Smith~~ Cooper, Battelle

Dear Rich and Joyce

I received the "Fluorochemical Fugacity Modeling Requirements" statement. It is generally in agreement with our expectations and we will do our best to satisfy your needs. The principal problem will, I suspect, be gathering the input data. We can keep in close contact with you regarding the model development.

We obtained from you the PFOS air-water partitioning data. As I interpret the lab results they established an initial concentration of 50 mg/L in 200 mL water then distilled off 10 mL aliquots and analysed the residue. They then calculated the percentage of the original PFOS present which remained in the beaker unevaporated. These "percent recoveries" ranged from 136 to 105 with no real trend. I conclude that the PFOS did not evaporate to any measurable extent. This is a very sensitive method of measuring low air-water partition coefficients. It can be shown that if water and the solute evaporate equally (i.e. the contents do not change in composition as would occur with an azeotrope) then K_{AW} or H is identical for water and the solute. For water H is approximately 2400 Pa (approximately 20°C) divided by 55000 mol/m^3 or 0.044 Pa m^3/mol or a K_{AW} of about 2 x 10^{-5}. PFOS must thus have a K_{AW} considerably less than this, i.e. less than 2 x 10^{-6}. It is thus essentially non-volatile from aqueous solution. This is probably because of its ionic nature. The simple expedient is to assign it a K_{AW} of zero, i.e. it is a type 2 involatile chemical in our nomenclature.

We did sample EQC model runs using a K_{AW} of zero, and partition coefficients L/kg for soil-water of 2.4, sediment-water of 4.8 and fish-water (dimensionless) of 1000. The dimensionless partition coefficients are higher by a factor corresponding to the density. The degradation half lives were all set at 10 years. I attach the Level I, II and III results.

The Level I results show that PFOS has a strong tendency to remain in water, i.e. typically 80% with 20% in soil.

The Level II results show an identical distribution. The major loss is by advection in water with

only very slow degradation. The overall persistence in a region is controlled by the water residence time.

The Level III results show that:-

If discharged to air it will rapidly deposit to soil (90%) and water (10%).

If discharged to soil it tends to remain there with the major loss being run-off to water because of its low sorption tendency.

If discharged to water it tends to remain in solution and is subject to removal by advective flow (90%).

There is little sorption to sediments.

In summary, it behaves much as one would expect of strongly ionic compound.

I believe it is important to measure real partition coefficients for inclusion in the model and perhaps refine the half lives. In the present regulatory climate a substance which is so persistent is going to come under very close scrutiny. I wonder if it is possible to build a degradation weakness into the molecule?

In conclusion, we are ready and willing to do modeling at your request. Please regard this as just a small first step.

Yours truly

Don Mackay

DM/nm