BIODEGRADATION

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-heptadecafluoro-, potassium salt, CAS # 2795-39-3)

Remarks: PFOS is a white powder. The original 3M production lot number was not noted. The PFOS was HPLC purified. An Interim Certificate of Analysis, reports the purity to be 97.9%. All results in this report are based on a purity of 86.4%, however. The lower purity value was associated with a standard the laboratory was using during this study.

METHOD:

Method: Based on EPA Guidelines OPPTS 835.3200, OPPTS 835.3210, OPPTS 835.5045.
Test Type: Aerobic
GLP: No
Year completed: 2001
Contact time: 35 days
Inoculum: Activated sludge collected 09/18/00 from the aeration basin at the Metro Wastewater Treatment Plant, St Paul, MN. The suspended solids were allowed to settle for approximately 2 days at 4°C and the settled sludge, approximately 20% of the volume, was used to prepare cultures for the biodegradation study. The mixed liquor suspended solids, MLSS, was not noted.

Test medium: Test flasks were prepared using a mineral salts medium defined in EPA Guideline OPPTS 835.3200. Fifty mL of settled sludge was added per liter of mineral salts medium.
Study design:

Blank Sludge Controls (mineral medium, inoculum)
Abiotic Controls (mineral medium, PFOS)
Test Substance (mineral medium, inoculum, PFOS)

Test vessels were set in duplicate. Additional quality control samples (blanks) were prepared and analyzed as appropriate.

Test concentration: 2.582 mg/L
Incubation conditions:

Temperature: 25°C +/- 3°C
Agitation: 200 rpm
Test vessels: Sterile 125 mL Nalgene polycarbonate culture flasks containing 25 mL of media

Dosing procedure: Test vessels were spiked 6 µL of 10,760 mg/L solution of PFOS in methanol yielding a 2.582 mg/L PFOS solution.
Sampling Frequency: Days 0, 2, 5, 7, 14 and 35.
Analytical method: The day zero test vessels were prepared and immediately placed in a freezer that was maintained at –20°C +/- 7°C. Upon removal from the incubator, test vessels on other days were either immediately frozen, or prepared by solid phase extraction (SPE). Following thawing, if needed, test vessel contents were adjusted to 1% acetic acid and then passed through a conditioned SEP-VAC C18 6cc SPE cartridge

containing a plug of quartz wool to deter plugging. Methanol was then added to the emptied culture flask, shaken vigorously and then passed through the SPE cartridge to extract adsorbed analytes. A second methanol wash was collected separately for analysis to ensure quantitative extraction.

Quantitative analysis was conducted on an HP1100 high performance liquid chromatograph with mass spectrometer detector (HPLC/MSD) system. The MSD was operated in electrospray ionization in negative-ion mode using selected-ion monitoring (SIM) for quantitation.

In addition to the parent, PFOS, the compounds below were quantified. In the case of the compounds that are potassium or ammonia salts, only the concentration of the fluorochemical anion was quantified and reported.

Compound Name
Acronym
Chemical Formula

2-(N-Ethyl Perfluorooctane sulfonamido) acetic acid
N-EtFOSAA
$C_8F_{17}SO_2N(C_2H_5)(CH_2COOH)$

2-(Perfluorooctane sulfonamido)
acetic acid
M556
$C_8F_{17}SO_2NH(CH_2COOH)$

N-Ethyl Perfluorooctane sulfonamide
N-EtFOSA
$C_8F_{17}SO_2NH(C_2H_5)$

Perfluorooctane sulfonamide
FOSA
$C_8F_{17}SO_2NH_2$

Perfluorooctane sulfinate, potassium salt
PFOSulfinate
$C_8F_{17}SO_2^- K^+$

Perfluorooctanoate, ammonium salt
PFOA
$C_7F_{15}COO^- NH_4^+$

2-(N-ethylperfluorooctanesulfonamido) ethyl alcohol
EtFOSE-OH
$C_8F_{17}SO_2N(CH_2CH_3)(CH_2CH_2OH)$

RESULTS

The analytical results[1] demonstrate that when exposed to municipal wastewater treatment sludge for 35 days, the 2.582 mg/L PFOS samples generated no quantifiable degradation products. PFOS was recovered at 2.553 mg/L +/- 0.102 mg/L in the pooled study samples, at 2.653 mg/L +/- 0.083 mg/L in the pooled abiotic samples. The measured concentration of PFOS was always 100 +/- 7% of the expected concentration.

CONCLUSIONS

This six-sample point screening study established that PFOS is not biodegraded by the microbial populations of the municipal waste treatment inoculum used under the conditions tested.

DATA QUALITY

Klimisch ranking = 2. The study was conducted as a non-GLP study but with the understanding that good data quality objectives be met.

Toxicity was not addressed as part of this study. The microbial respiration inhibition by PFOS has been tested previously[2], and PFOS was found to not be a significant inhibitor or microbial respiration processes at the concentration tested here.

No target analytes were detected in any of the test vessels or blank sludge controls.

No sample matrix spikes were included in this study. However, PFOS was recovered at expected concentrations, and previous duplicated results obtained during a related project showed excellent PFOS recoveries from sludge using the same extraction method.

A series of positive controls were not run with this study. However, the sludge used in this study was also used for the preparation of samples in other biodegradation studies, and in some of those studies, preliminary results demonstrated positive biological activity for degradation of test samples.

REFERENCES

[1]Biodegradation Study Report, "*The 35-Day Aerobic Biodegradation Study of PFOS"*, Contract Analytical Project ID: CA104, 3M Project ID: LIMS E01-0444, March 29, 2001.

[2]Activated Sludge Respiration Inhibition Test for PFOS. Test conducted under GLP requirements for 3M by Wildlife International, Ltd. 3M Environmental Lab request number U2723.

Study number 1 was conducted at the request of the 3M Company by Pace Analytical Services, Inc., Minneapolis, MN

Study number 2 was conducted at the request of the 3M Company by Wildlife International, Ltd., Easton, MD

OTHER

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