BIODEGRADATION

TEST SUBSTANCE

Identity: N-Ethylperfluorooctanesulfonamide; may also be referred to as N-PFOSA, EtPOSA, F-6309, or FX-12. (1-Octanesulfonamide, 1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-heptadecafluoro-N-ethyl-, CAS # 4151-50-2)

Remarks: N-EtFOSA was a white solid. The 3M production lot number was 529, the 3M standard identification # SD-012. Available NMR information indicates the purity to be 99.3%. All results in this study were calculated assuming 100% purity.

METHOD:

Method: Based on EPA Guidelines OPPTS 835.3200
Test Type: Aerobic
GLP: No
Year Completed: 2001
Contact time: 18 days

Inoculum: Activated sludge collected 7/31/00 from the aeration basin at the Metro Wastewater Treatment Plant, St Paul, MN. The MLSS was determined to be 2,280 mg/L when first collected. The MLSS was stored at 4º C for approximately 5 weeks prior to being used for this study. The sludge was allowed to settle and the solids used for inoculum. The settled sludge constituted approximately 20% of the volume (~200 mL) of the MLSS used.
Test medium: Test flasks were prepared using a mineral salts medium defined in EPA Guideline OPPTS 835.3200. Methanol (1 mL per liter) was added per liter of mineral medium. Fifty mL of settled sludge was added per liter of mineral salts medium. Mineral medium plus sludge was prepared 9/7/00, while fresh mineral medium without sludge (abiotic controls) was prepared 8/10/00.
Study design:

Blank Sludge Controls	(mineral medium, inoculum)
Abiotic Controls	(mineral medium, N-EtFOSA)
Test Substance	(mineral medium, inoculum, N-EtFOSA)

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

Test concentration: 2.825 mg/L N-EtFOSA.
Incubation conditions:
Temperature: 25^{o}C +/- 3^{o}C
Agitation: ~200 rpm
Test vessels: Sterile 125 mL Nalgene polycarbonate culture flasks containing 25 mL of media

Dosing procedure: Test vessels were spiked with 6 µL of an 11,770 mg/L solution of N-EtFOSA in methanol yielding 2.825 mg/L.
Sampling Frequency: Days 0 and 18.
Analytical method: The day zero test vessels were prepared and immediately placed in a freezer that was maintained at –20^{o}C until analyzed. After 18 days, the test vessels were removed from the incubator and frozen until final sample preparation by solid phase

extraction (SPE). Following thawing, test vessel contents were adjusted to 1% acetic acid and then passed through a conditioned SEP-VAC C18 6cc SPE cartridge. 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 N-EtFOSA, the compounds quantified 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) ethyl alcohol
N-EtFOSE Alcohol
$C_8F_{17}SO_2N(C_2H_5)CH_2CH_2OH$

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
$C_8F_{17}SO_2NH(CH_2COOH)$

Perfluorooctane sulfonamide
FOSA
$C_8F_{17}SO_2NH_2$

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

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

Perfluorooctane sulfonate potassium salt
PFOS
$C_8F_{17}SO_3^- K^+$

Reference substance: None. When results from an EtFOSE Alcohol study conducted at the same time are compared to a previous EtFOSE Alcohol 35-day study, the viability of the microbial inoculum is confirmed.

RESULTS

After 18 days, the analytical results[1] demonstrate that when exposed to municipal wastewater treatment sludge, 2.825 mg/L N-EtFOSA degraded. Only 13.7% of the starting concentration was remaining. The major metabolites detected were M556 and FOSA. However, only 31% of the original N-EtFOSA added to the abiotic controls remained after 18 days and no biotransformation products were detected in these flasks. Mass balance for N-EtFOSA test vessels was poor and ranged from 30 - 35% recovery. These results may indicate loss of N-EtFOSA to the air through volatilization, since the vapor pressure may be significant, and the test vessels were loosely capped to allow for air exchange during the study.

CONCLUSIONS

Loss of 86.3% of 2.825 mg/L of N-EtFOSA was demonstrated. The major metabolites identified were M556 and FOSA. However, the results of this study are somewhat confounded by poor mass balance and by degradation in the abiotic control in which no metabolites were detected, suggesting an apparent loss of N-EtFOSA through volatilization during incubation. Mass balance between parent material and measured products was only 30-35%. The results from this study indicated slower biotransformation of N-EtFOSA than expected when compared to the results from the previous 35-day study of N-EtFOSE alcohol.

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.

Determination of analyte recovery from spiked sample matrices was not deemed necessary as they have been determined twice previously at several different concentrations, and in both instances recoveries were near 100% with some exceptions for M556. Methanol and sample (mineral medium) blanks contained no detected target analytes.

Analyses of the blank sludge controls (mineral media plus inoculum) at days 0 and 18 demonstrated that the inoculum source did not contain endogenous concentrations of test substance or its metabolites.

A calculation error was discovered subsequent to the issuance of the final report. The PFOS molar conversion calculation should have used 538 ng/nmole rather than 522 ng/nmole. The corrected value for PFOS on page 13 of the report should be 92.9 nM. This does not affect any of the reported results or conclusions.

REFERENCES

[1]Biodegradation Study Report, "*The 18-Day Aerobic Biodegradation Study of Perfluoroctanesulfonyl-Based Chemistries"*, Contract Analytical Project ID: CA097, February 23, 2001. Conducted at the request of the 3M Company by Pace Analytical Services, Inc., Minneapolis, MN.

OTHER

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