BIODEGRADATION

TEST SUBSTANCE

Identity: Perfluorooctanesulfonylamido(ethyl)acetate; may also be referred to as PFOSAA, N-EtFOSAA, FC-128 (acid form) or FC-129 acid. [$C_8F_{17}SO_2N(C_2H_5)CH_2CO_2H$], CAS # 2991-51-7)

Remarks: N-ETFOSAA is a light yellow powder. The 3M production lot number was not noted. Current NMR information indicates a purity of 97.6%.

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-EtFOSAA)
Test Substance	(mineral medium, inoculum, N-EtFOSAA)

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

Test concentration: 2.539 mg/L N-EtFOSAA.
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 with 6 µL of an 10,580 mg/L solution of N-ETFOSAA in methanol yielding 2.539 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°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-ETFOSAA, the additional compounds quantified are specified below. 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
$C_8F_{17}SO_2N(C_2H_5)CH_2CH_2OH$

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

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

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

Perfluorooctane sulfonamide
FOSA
$C_8F_{17}SO_2NH_2$

Reference substance: None. When results from an EtFOSE Alcohol study conducted at the same time are compared to the 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, 9.3% of the 2.455 mg/L solution N-EtFOSAA was biotransformed to yield measurable quantities of M556, FOSA, PFOS and PFOA. Mass balance for N-EtFOSAA test vessels ranged from 96 - 109%.

CONCLUSIONS

N-EtFOSAA was biodegraded by 9.3% over 18 days to yield M556, FOSA, PFOS and PFOA.

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 issuance of the final report. The results and conclusions of the report are not affected. 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.

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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