

SOIL ADSORPTION (METHOD DEVELOPMENT / PILOT STUDIES)

TEST SUBSTANCE

Identity: N-ethylperfluorooctane sulfonamidoethanol; may also be referred to as N-EtFOSE Alcohol or FM-3422. (1-Octanesulfonamide, N-ethyl-1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-heptafluoro-N-(2-hydroxyethyl)-, CAS # 1691-99-2)

Remarks: Material is an off-white, waxy solid of uncharacterized purity.

METHOD

Method: The attached studies explore the use of organic solvents (1977 study) or Thin Layer Chromatography Plates (1978 study) for determining the soil mobility of N-EtFOSE alcohol. These methods were developed by 3M while looking for alternatives for studying the adsorption/desorption properties of the test substance.

GLP (Y/N): No

Year (study performed): 1977 and 1978

Statistical methods: None.

Temperature: Not noted in the reports.

Remarks field: Very little detail is available on these study methods.

RESULTS

The 1977 study using 20% and 5% alcohol as a portion of the solvent indicated between 64% and 98% of the N-EtFOSE alcohol was sorbed to the sandy loam soil. The 1979 study using TLC greenhouse soil plates indicated that N-EtFOSE alcohol did not move from the spot it was placed.

CONCLUSIONS

Both studies concluded that N-EtFOSE alcohol is likely to sorb to soil.

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

DATA QUALITY

Reliability: Klimisch ranking 3. These studies lack sufficient explanation of the methodologies. Purity of the test substance is not characterized. The analytical methodology is also either missing or lacks validation.

REFERENCES

Method and results summary, "Adsorption of FM-3422 on Soil." Stephen K. Welsh, March 7, 1977, April 12, 1977, May 9, 1977.

Excerpt from 3M Technical Notebood No. 48277 30, "Soil Thin-Layer Chromatography – FC 95, FC 143, FM 3422", Page 30, A. Mendel, Project Number 997061200, October 13, 1978

Review of Technical Notebook No. 48277-30. Soil Thin Layer Chromatography. Professor Stephen A. Boyd, Michigan State University, May 19, 1993.

OTHER

Last changed: 5/18/00

006211

Attached are comments on the 3M Technical Report "Soil Thin Layer Chromatography - FC95, FC143, FM3422. A. Mendel, Project No. 997061200 3M Technical Notebook No. 48277, Page 30, Oct. 13, 1978 (one page from a technical notebook)" made by Professor Stephen A. Boyd, Michigan State University, dated May 19, 1993.

006212

Review of Technical Notebook No. 48277-30
Soil Thin Layer Chromatography

I am not too sure how useful these plates are. Apparently both FC 143 and FM 3422 remained at the origin of the thin layer plate where they were originally spotted. This implies no movement with the water used to develop the plate. Basically this tells me the method is insensitive, and doesn't yield much useful information. That is because FM 143 has a much lower soil sorption coefficient ($K_{oc} = 17$) than FM 3422 ($K_{oc} = 15,000$) and yet they behaved similarly in the soil thin layer chromatography plate.

Mobility studies are probably best done in soil columns using miscible displacement experiments. These are more difficult to do experimentally but would yield more useful data that could be compared to other well studied compounds. The experimental approach is well established now and there are several laboratories doing this type of work, including my own. These experiments augment batch equilibration (isotherm) experiments very nicely.

SUBJECT NO. 9970612600

3M TECHNICAL NOTEBOOK NO.

Date: 10-13-78

Subject:

SOIL THIN-LAYER CHROMATOGRAPHY - FC95, FC143, FM3422

Object: TO COAT GLASS PLATES WITH SOIL FOR STUDIES

See also A. Mendel - Tech Report - 1/17/79Reference: 42554-72

VARIOUS SOILS WERE SIEVED EARLIER BY M.T. ELNABARAWY AND IT WAS FOUND EMPIRICALLY THAT THE 250 MICRON SIZE SOIL PARTICLES WHEN SLURRIED WITH WATER AND COATED ON GLASS PLATES GAVE GOOD OVERALL PLATES. ACCORDINGLY THE FOLLOWING WAS DONE.

AN INTIMATE MIXTURE OF 150 g. OF 250 MICRON-SIEVED GREEN-HOUSE SOIL [OBTAINED FROM W. BERS, AGRICHEM AND SOIL SIEVED BY M.T.E. AND L.H. PRITCHARD] AND 80 ML. WATER WAS WELL-SHAKEN AND SPREAD ONTO TEN 10x20 CM. GLASS PLATES [NEXT TIME TRY USING 85-90 ML. WATER TO MAKE MIXTURE SLIGHTLY LESS VISCOUS AND SPREADING MAY BE SLIGHTLY QUICKER]. THE PLATES WERE ALLOWED TO AIR DRY. THE SPREADER USED TO COAT THE PLATES WAS DESIGNED BY DR. J.W. BEUBLE, G. GUTHRIE (MACHINE SHOP-201) AND A. MENDEL 10/13/78

ONE OF THE 10x20 CM PLATES WAS SPOTTED 2 CM FROM THE BOTTOM WITH RADIO LABELED FC 95 (LEFT SIDE), FC 143 (MIDDLE) AND FM 3422 (RIGHT SIDE, AS ONE VIEWS PLATE); SPOTS WERE APPLIED BY CONNIE SCHRAMDT. THE PLATE WAS DEVELOPED 100 MM. IN A FILTER PAPER [WHATMAN NO. 1]-LINED TANK PREHEATED [OVER 1 DAY] WITH WATER. THE DEVELOPMENT REQUIRED ABOUT 1 HR 40 MIN. THE PLATE WAS AIR DRIED. IT WAS NOTED THAT WHEN THE SOIL TLC PLATE WAS PLACED INTO THE TANK CONTAINING ABOUT 1 CM DEEP WATER, THE SOIL IN THE WATER GRADUALLY FELL FROM THE PLATE BUT THIS DID NOT SEEM TO AFFECT THE DEVELOPMENT PROCESS. 10/18/78

THE PLATE WAS PLACED ON X-RAY FILM (AGRICHEM FACILITIES) AND EXPOSED FOR 10 DAYS. (TEN). THE DEVELOPED PLATE (KINDLY DEVELOPED BY LARRY STEIFCK, AGRICHEM) SHOWED THAT FC143 AND FM3422 DID NOT LEAVE ORIGIN. THE SPOT DUE TO FC-95 WAS TOO FAINT FOR VISUALIZATION. THE LOW RADIOACTIVITY OF THE COMPOUNDS REQUIRE HIGHER CONCENTRATION OF SPOTTING (i.e. MORE SAMPLE). 11/6/78

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Signature

A. Mendel

006209

Date:

Nov 13, 1978

Read and Understood

Date:

11/11/78

Soil
 Sand 57%
 silt 36%
 clay 7%
 Organic Matter 2.5%
 CEC 15.3 meq/100g

5610

ADSORPTION OF FM 3722 (in FC-206) ON SOIL, 12/30/75

Soil - sandy loam, 40673, p. 17; air dried

1. Make up solutions of FC-206 in DI water at 600 ppm, 6,000 ppm, and 60,000 ppm.
2. In 500 ml flasks put 100g soil and 100 ml of the following solutions:
 Flask 18A - DI water
 Flask 18B - 600 ppm FC-206
 Flask 18C - 6,000 ppm FC-206
 Flask 18D - 60,000 ppm FC-206
3. Shake flask for 24 hours at 100 oscillation/minute.
4. Let soil settle and decant of the liquid. Centrifuge the liquid.

RESULTS (12/30/75)

FC-206 Conc.	DI Water	600 ppm	6,000 ppm	60,000 ppm
FM-3722 conc., ppm	---	4.2	42	420
Equil. FM-3722 conc. C, ppm (measured)	---	0.14	2.2	210
FM-3722 adsorbed, x/m, $\mu\text{g/g}$ (calculated)	---	4.1	40	210
% Adsorbed	---	98	95	50

ADSORPTION OF FM 3422 ON SOIL, 3/7/77

Soil - sandy loam, 40673, p. 17; air dried
 Solvent - 20% alcohol in DI water

1. Put a few mg FM 3422 (Lot 676) in the solvent and shake for at least 24 hours. Let FM 3422 settle and decant off the supernatant (the stock solution).
2. Make 10% and 1% dilutions of the stock solution with the same solvent.
3. Put 20g soil in each of 4-50 ml centrifuge tubes, then fill the tubes with DI water, shake thoroughly, and centrifuge five minutes at 6,000 rpm (4,000 G).
4. Pour off the water and add 20 ml of the sample solutions and 20 ml DI water in the blank. Shake the tubes for 24 hours (100-120 oscillation/min.).
5. After shaking, centrifuge as above and pour off the liquid.

RESULTS (3/7/77)

		100%	10%	1%
FM 3422 conc. ppm (measured)	DI Water	23	1.8	0.19
Equil. Conc. C, ppm (measured)	---	1.5	0.16	0.07
Adsorbed, x/m, $\mu\text{g/g}$ (calculated)	---	21	1.6	0.12
% Adsorbed	---	94	91	64

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ADSORPTION OF FM 3422 ON SOIL, 4/12/77

Soil - sandy loam, 40673, p. 17; air dried
Solvent - 5% alcohol in DI water

1. Put a few mg FM 3422 in the solvent and shake for at least 24 hours. Let FM 3422 settle and decant off the supernatant (the stock solution). Centrifuge at 10,000 rpm for 10 minutes.
2. Make 10% and 1% dilutions of the stock solution with the same solvent.
3. Put 20g soil in each of 4-50 ml. centrifuge tubes, then fill the tubes with DI water, shake thoroughly, and centrifuge at 10,000 rpm for 10 min.
4. Pour off the water and add 20 ml of the sample solutions and 20 ml DI water in the blank. Shake the tubes for 24 hours (100-120 oscillations/min.).
5. After shaking, centrifuge as above and pour off the liquid.

RESULTS (4/12/77)

FM 3422 conc. ppm (measured)	DI Water	44	3.9	0.5
Equil. conc. C, ppm (measured)	---	0.8	0.16	0.08
Adsorbed x/m, µg/g (calculated)	---	43	3.7	0.42
% Adsorbed	---	98	96	84

ADSORPTION OF FM 3422 ON SOIL, 5/9/77

Soil - sandy loam, 40673, p. 17; air dried
Solvent - 5% alcohol in DI water

1. Dissolve 50.0 mg FM 3422 (Lot 676) in 50.0 ml alcohol. Put this solution into a one liter volumetric and make up to mark with DI water.
2. Let the above "emulsion" stand for five days. (A white floc appeared in the bottom) Centrifuge some of the supernatant at 10,000 rpm for 10 min.
3. Make 75%, 50%, 25%, and 10% dilutions of the stock solution with the same solvent.
4. Put 20g soil in each of 6-50 ml centrifuge tubes, then fill the tubes with the 5% alcohol solvent, shake thoroughly, and centrifuge five minutes at 5,000 rpm.
5. Pour off the solvent and add 20 ml of the sample solutions and 20 ml DI water in the blank. Shake the tubes for 24 hours (100-120 oscillations/min.).
6. After shaking, centrifuge (5,000 rpm, 5 min.) and pour off the liquid.

RESULTS (5/9/77)

FM 3422 conc., ppm (measured)	DI Water	3.1	2.25	1.5	0.64	0.38
Equil. conc., C, ppm (measured)	---	0.06	0.05	*	0.02	0.02
Adsorbed, x/m, µg/g (calculated)	---	3.0	2.2	*	0.62	0.36
% Adsorbed	---	98	98	*	97	95

* Dropped the sample

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