HYDROLYSIS (ALKALINE)

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-heptadecafluoro-N-(2-hydroxyethyl)-, CAS # 1691-99-2)

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

METHOD

Method/guideline followed: Procedure developed by 3M
Type (test type): Alkaline potassium hydroxide solution exposure
GLP (Y/N): No
Year: 1977 or before
Remarks field:

- **Test medium (air, water, soil, other - specify):** 20% alcoholic potassium hyrdoxide.
- **Duration:** 24 hours
- **Positive Controls:** None
- **Negative Controls:** None

RESULTS

Concentration of Substance: 362 mg/L
Temperature °C: 50°C
Degradation %: 92% after 24 hours

Remarks field: IR and NMR studies confirmed that the N-EtFOSE Alcohol was broken down to perfluorooctane sulfonate under these conditions.

CONCLUSIONS

Under the conditions of this test, N-EtFOSE alcohol partially degrades to form perfluorooctane sulfonate.

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

DATA QUALITY

Reliability: Klimisch ranking 2. Purity of the test substance was not characterized.

REFERENCES

3M Technical Report "Analytical Methodology on FM 3422." A. Mendel, Fate of Fluorochemicals, Page 11, Project 9970612643, November 15, 1977

OTHER

Last changed: 5/18/00

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TECHNICAL REPORT SUMMARY

Date: 11/15/77

TO: TECHNICAL COMMUNICATIONS CENTER – 201-2CN

(Important – If report is printed on both sides of paper, send two copies to TCC.)

Division: Environmental Laboratory (EE & PC)

Dept. Number: 0222

Project: Fate of Fluorochemicals

Project Number: 9970612643

Report Title: Analytical Methodology on FM 3422

Report Number:

To: D. L. Bacon

Author(s): A. Mendel

Employee Number(s): 43939

Notebook Reference:

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SECURITY ▶ ☐ Open (Company Confidential) ☐ Closed (Special Authorization)

3M CHEMICAL REGISTRY ▶ New Chemicals Reported ☐ Yes ☒ No

KEYWORDS: (Select terms from 3M Thesaurus. Suggest other applicable terms.)

EE & PC-Div.
Fluorochemical
Analytical

CURRENT OBJECTIVE:

Progress Report

REPORT ABSTRACT: (200-250 words) This abstract information is distributed by the Technical Communications Center to alert 3M'ers to Company R&D. It is Company confidential material.

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Information Liaison Initials

ALKALINE HYDROLYSIS OF FM 3422

The stability or instability of FM 3422 toward base was apparently studied several years ago under aqueous conditions. Because this material steam distills and because of its lack of water solubility, the stability of FM 3422 toward base in alcohol was studied in the present work as follows:

Six samples each of a 25 ml solution of 362 ppm of FM 3422 in 20% alcoholic potassium hydroxide was kept in a 50° oil bath for varying lengths of time. A sample was taken after 0.5-, 1-, 7- and 24- hrs., and neutralized with concentrated nitric acid to the phenolphthalein indicator end point; this allows precipitation of potassium nitrate and minimizes water which tends to interfere with the EC/GC analyses. GC analysis of the alcohol solution gave the results in TABLE V.

TABLE V

HYDROLYSIS STUDIES OF FM 3422

Time (in hrs.)	0	0.5	1	7	24
3422 (in ppm)	362	380	362	256	28

A sixth sample was allowed to remain in the oil bath for 50 hrs/50° C. It was neutralized as above and filtered to remove solids. The filtrate was concentrated to dryness and the residue was analyzed by TLC, and IR and NMR (Req. C46749). A comparison of the R_f value of the residue with that of authentic FC-95 showed they were the same. IR and NMR studies confirmed that the FM 3422 was broken down to FC-95 under these conditions.

GAS CHROMATOGRAPHY OF PERFLUOROOCTANE SULFONAMIDE AND FC-128 (41947-48)

The retention time of FM 3422 was 2.8 minutes isothermal at 160° using the conditions described earlier. To determine whether the above sulfonamide and FC-128 posed as an interference to FM 3422, dilute solutions (in methanol) were made for these two materials. The sulfonamide could be chromatographed isothermally at 200° (about 3.3 minutes retention) while FC-128 could not be chromatographed. Therefore, there is no interference problem.

THIN-LAYER CHROMATOGRAPHY STUDIES OF FM 3422

Radiolabeled FM 3422 (5) was examined by TLC for purity. About ten micrograms of sample was spotted on a plate which was then developed in ethyl acetate. The plate was exposed to an X-ray film for about one week and the film was developed. It revealed that the FM 3422-^{14}C had three impurities, two components of greater R_f and one component remaining at the origin. Accordingly, the entire radioactive material, nearly 1.7 g, was column chromatographed on silica gel by chloroform elution. About forty 15 ml fractions were collected and monitored by TLC-autoradiography. Pure material was combined for future studies. Impure FM 3422-^{14}C. was combined and rechromatographed as above until essentially all of the material was rendered pure (6).