TDT-FC-95 CC.D

3M

Form 6747-11-A

TECHNICAL REPORT SUMMARY

Date: 6/23/80

TO: TECHNICAL COMMUNICATIONS CENTER – 201-2CN

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

Division: Environmental Engineering and Pollution Control	Dept. Number: 0222
Project: Fate of Fluorochemicals	Project Number: 78-2740
Report Title: Detection of Fluorochemicals in Decatur Wastewater	Report Number: 001
To: D. L. Bacon	
Author(s): Arthur Mendel, C. H. Schrandt and J. E. Gagnon	Employee Number(s): REDACTED
Notebook Reference: 80	No. of Pages Including Coversheet: 4

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.
(Env. Lab)

CURRENT OBJECTIVE:

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.

Wastewater from Decatur, Alabama Plant was analyzed for Fluorochemicals.

Information Liaison Initials: MO

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Exhibit 1233
State of Minnesota v. 3M Co.,
Court File No. 27-CV-10-28862

INTRODUCTION:

The purpose of this study was to sample the Decatur plant wastewater during high volume (HV) and low volume (LV) plant production to identify and quantify any fluorochemicals.

RESULTS:

The HV sample contained 96 parts per billion (ppb) of organic fluorine and less than one ppb of inorganic fluoride. The LV sample was not analyzed for these parameters.

Two dimensional thin-layer chromatography (TLC) indicated the presence of FC-95 and FC-143 in both HV and LV samples. Gas chromatography (GC) was used to quantify FC-143 at 28 ppb in the HV sample and 4 ppb in LV sample.

Fluorine-containing organics identified in the LV sample by GC-mass spectroscopy (GC/MS) were:

(1) $C_4F_9SO_2NHCH_3$

(2) $C_8F_{17}N(CH_3)CH_2CH_2OH$

Fluorochemical fragments indicated by GC/MS were:

$C_6F_{13}SO_2N(CH_3)CH_2CH_2-$

$R_fSO_2-N(OC_3H_7)-C_2H_5$

where R_f is C_6F_{13} or higher perfluoro homolog.

The only significant fluorochemical detected in the HV sample was compound 1 above (N-methylperfluorobutanesulfonamide)[1].

DISCUSSION:

The Decatur, Alabama plant makes fluorochemicals. This is the first pilot study to determine if fluorochemicals could be identified and possibly quantitated in the plant effluent. Accordingly, the plant effluent was sampled at a time of high production schedule and about nine months later when the plant was at low production schedule. Sampling was done by pumping the effluent through a carbon bed (Figure 1). Organic materials,

extracted from the carbon bed, were then analyzed using a combination of analytical techniques[2]. The results of this study (see above) indicate that carbon sorption is satisfactory for concentrating organic fluorochemicals from waters. Further in-depth studies would have to address recoveries of fluorochemicals from carbon and show no interaction and/or degradation of fluorochemicals on carbon adsorption and desorption. A reliable, less moisture-sensitive analytical method for FC-95 and FC-807 would be desirable.

EXPERIMENTAL:

See Notebooks 49400, 51050, 51568 for details of this investigation[3].

Rigid polyvinyl-chloride (PVC) columns (Figure 1) were wet slurry packed with about 30 g of Hydrodarco^{R} 4000 (HD 4000) activated carbon and shipped to the Decatur, AL, plant (1/30/79). It was requested that the seven columns be kept refrigerated until use to minimize bacterial growth[4].

HD 4000 was purified before packing by first rinsing with deionized water (D.I.) and decanting the fines and then rising with methanol followed by D.I. water. The carbon was dried in a forced draft oven (110°C) and stored in a desiccator until needed.

Sampling at high-volume production (HV) was begun on February, 1979, and low volume (LV) sampling was started on October, 1979, with 1,144 and 623 gallons of effluent sampled respectively. Several backup columns were sampled, but only the HV and LV samples will be discussed in this study.

The carbon, removed from the sampling columns, was extracted (Soxhlet) for three hours with analytical reagent grade pyridine. Extraction thimbles were not used; silanized glass wool held the carbon in the extractor. Thirty grams of clean HD-4000 served as the "blank" in another extraction experiment. The pyridine extracts were filtered through Whatman GF/F paper and the filtrate was brought to 250 ml with pyridine (analytical solution). Some of this solution was analyzed then for organic and inorganic fluorine (see results).

Another portion of this analytical solution was concentrated tenfold (rotary evaporator) and the concentrate was spotted on silica gel TLC plates. They were developed in two dimensions with two different developing liquids. FC-143 and FC-95 were tentatively identified in both the HV and LV samples.

The above concentrate was also analyzed by GC/MS for fluorochemicals (see results).

A portion of the pyridine extract was diluted with deionized water, acidified then with hydrochloric acid and extracted three times with analytical grade toluene. Some of this extract was methylated with alcohol-free diazomethane. The methylated solution was analyzed by GC using an electron capture detector (see results).

NOTES:

(1) One reason for the isolation of compound 1 in both plant samples may be that it is one of the more soluble fluorochemicals compared to the rest of the fluorochemicals generated. (Phone discussion of A Mendel with R. A. Guenthner, Commercial Chemicals.)

(2) It was assumed that all fluorochemicals adsorbed onto carbon were quantitatively recovered and unaltered. It is known, however, that many compounds, once sorbed onto carbon, are not completely removed by extraction procedures.

(3) C. H. Schrandt has since transferred to Automotive Trades Division.

(4) For further studies, carbon should be slurried with water to allow removal of the floating fine particles which tend to plug the column. The column should be wet (water) slurry packed and then refrigerated until use to prevent bacterial growth. Such columns in preference to dry-packed columns do not plug easily during effluent sampling and less back pressure was noted.

AM 8/5/80

FIGURE I

DIAGRAM OF COLUMN FOR SAMPLING