Form 6747-11-A

TECHNICAL REPORT SUMMARY

Dept. 11/30/77

TO: TECHNICAL COMMUNICATIONS CENTER – 201-2CN

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

JAN 09 1978

Division: Central Research - Analytical	Dept. Number: 0502
Project: Det'n. and Characterization of Trace Fluorochemicals	Project Number: 9172110004
Report Title: 1977 Summary	Report Number: 002
To: B. W. Nippoldt	
Author(s): Jon Belisle and D. F. Hagen	Employee Number(s): 88240 - 42608
Notebook Reference: 44290	No. of Pages Including Coversheet: 5

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.)

Central Research
Chemical/Analysis
Trace/Analysis
Fluorochemical

CURRENT OBJECTIVE:

Continue to develop methods and analyze incoming samples.

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.

Methods are available for the analysis of fluorochemicals FC-26/143 and FC-95 in serum and water (environmental studies). The methods have been and are in the process of being applied to urine, liver, and other biological samples. In addition, a method for compound FM-3422 in urine and serum should soon be available.

Information Liaison Initials: RCV 1/4/78

3M CONFIDENTIAL

Exhibit 1148
State of Minnesota v. 3M Co., Court File No. 27-CV-10-28862

3MA00016495

1148.0001

We continue to develop methods and analyze samples relative to the project "Determination and Characterization of Trace Fluorochemicals in the Environment".

Definitions

FM 3422: $C_8F_{17}SO_2N(C_2H_5)C_2H_4OH$

FC-26: $C_7F_{15}COOH$

FC-143: $C_7F_{15}COONH_4$

FC-95: $C_8F_{17}SO_3K$

Report

As of 11/77, we have the capability of determining the following:

(1) Total fluorine and inorganic fluoride in a whole blood, plasma, serum, and liver sample down to 0.01 ppm. The method has been applied to a known urine sample but due to the use of fluoridated drinking water, the background inorganic fluoride is approximately 1 ppm.

(2) FC-26/143 in serum down to 0.1 ppm

(3) FC-26/143 in urine down to 0.1 ppm

(4) FC-26/143 in rat liver down to 0.5 ppm

(5) FC-95 in serum down to 1 ppm

A number of related analyses have been carried out. For example, C_3F_7COOH in serum (GC column 10' 1/8 ss n-octane/Porasil-C 100/200), FC-43/FC-70 in rat liver from toxicological study and concentration of FC-95 from urine. We have begun the analysis of FM 3422 from blood and urine by direct GC on Carbowax 20M.

Method 1) is based on Parr oxygen bomb combustion and triethylsilanol reaction with F^- followed by GC. It has been written, cleared by 3M, and accepted for publication by Analytical Biochemistry.

Methods 2-4) are based on hexane/ether extracts followed by diazomethane and GC.

Method 5) is based on ether extraction and the preparation of the sulfonyl chloride from the sulfonic acid using PCl_5 followed by formation of the phenyl ester and GC.

3M CONFIDENTIAL

A number of experiments have been done with ^{14}C labeled FC-95 to better understand the extraction and concentration of FC-95 from plasma and urine. Presently a small micro column (0.5 cm x 3 cm) of silica gel is used to clean up the ether extract of serum/plasma prior to PCl_5 reaction.

The following is a list of the analytical requests completed the past year on this project: A64037, C46956, A64969, A65353 and A66624. Requests A66860 and A67105 are in progress and should be completed by the end of the year.

Attached are 2 letters from the toxicology lab to emphasize the diverse nature of the samples that will be submitted and the need for additional analytical methods.

Jon Belisle
/jz

D. F. Hagen

3M CONFIDENTIAL