TECHNICAL REPORT SUMMARY

TO: TECHNICAL COMMUNICATIONS CENTER – 201-2CN

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

Division
Central Research - Analytical

Project

Report Title
Det'n. and Characterization of Trace Fluorochemicals

1977 Summary

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
☐ Yes
☐ No

CURRENT OBJECTIVE:

Continue to develop methods and analyze incoming samples.

Central Research
Chemical/Analysis
Trace/Analysis
Fluorochemical

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

Methods are available for the analysis of fluorochemicals PC-26/143 and PC-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 PM-3422 in urine and serum should soon be available.
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\textsubscript{8}F\textsubscript{17}SO\textsubscript{2}N(C\textsubscript{2}H\textsubscript{5})C\textsubscript{2}H\textsubscript{4}OH
FC-26: C\textsubscript{7}F\textsubscript{15}COOH
FC-143: C\textsubscript{7}F\textsubscript{15}COONH\textsubscript{4}
FC-95: C\textsubscript{8}F\textsubscript{17}SO\textsubscript{3}K

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\textsubscript{4}F\textsubscript{7}COOH 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\textsuperscript{-} 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\textsubscript{5} followed by formation of the phenyl ester and GC.
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 PC15 reaction.

The following is a list of the analytical requests completed the past year on this project: A64037, C46955, 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  
D. F. Hagen