

3M SPECIALTY ADHESIVES & CHEMICALS ANALYTICAL LABORATORY

Request #'s 53030

To: Leo Gehlhoff - (309727) -3M Chemicals - 236-2A-01
From: Tom Kestner - (3-5633) SA&C Analytical Lab - 236-2B-11
Subject: *Fluorochemical Isomer Distribution by ^{19}F -NMR Spectroscopy*
Date: December 1, 1997

SAMPLE DESCRIPTIONS:

- FC-95, lot # 217 (T-6295); Nominal product = $\text{C}_8\text{F}_{17}\text{-SO}_3(-)\text{K}(+)$

INTRODUCTION:

This sample was subjected to a ^{19}F -NMR spectral analysis method to determine the identities and relative concentrations of the fluorochemical isomers and as many other identifiable impurity components as possible.

EXPERIMENTAL:

A portion of the sample solid was totally dissolved in DMSO-d_6 and then a 376 MHz ^{19}F -NMR spectrum (F53030.401) was acquired using a Varian UNITYplus 400 FT-NMR spectrometer. Aida Robbins prepared the sample for analysis and she also acquired and plotted the NMR spectrum.

RESULTS:

The ^{19}F -NMR spectrum was used to determine the identities and relative concentrations of the nominal fluorochemical isomers and three other impurity components in this sample. The qualitative and quantitative compositional results which were derived from the single trial ^{19}F -NMR spectral analysis are summarized in TABLE-1 on the following page.

A copy of the NMR spectrum and the spectral assignments data page are attached for your reference. If you have any questions about these NMR results, please let me know. I apologize for the delay in completing this work.

Tom Kestner

c: Jim Johnson - EE&PC - 2-3E-09

File Reference: LG53030.DOC/43

TABLE-1
Sample: T-6295 (FC-95, Lot 217)
¹⁹F-NMR Compositional Results

Structural Assignments	¹⁹ F-NMR Relative Mole% Concentrations
CF ₃ (CF ₂) _x -SO ₃ (-) K(+) (Normal chain, where x is mainly 7)	70.0%
CF ₃ (CF ₂) _x -CF(CF ₃)-(CF ₂) _y - SO ₃ (-) K(+) (Internal monomethyl branch, where x+y is mainly 5, and x ≠ 0, y ≠ 0)	17.0%
(CF ₃) ₂ CF-(CF ₂) _x - SO ₃ (-) K(+) (Isopropyl branch, where x is mainly 5)	10.3%
C _x F _{2x+1} -CF(CF ₃)- SO ₃ (-) K(+) (Alpha branch, where x is mainly 6)	1.6%
R _f -CF ₂ -SF ₄ -F	0.35%
(CF ₃) ₃ C-(CF ₂) _x - SO ₃ (-) K(+) (t-butyl branch, where x is mainly 4)	0.23%
CF ₃ -(CF ₂) _x -C(CF ₃) ₂ -(CF ₂) _y - SO ₃ (-) K(+) (Internal gem-dimethyl branch, where x+y is mainly 4, and x ≠ 0)	0.15%
Possible CF ₃ -SO ₃ (-) K(+)	0.25%
Possible CF ₃ -CO ₂ (-) K(+)	0.05%