

REQ 41220

R. Howells

GC/MS analyses of these samples were accomplished using a 25 m X 0.32 mm HP-1 GC column to introduce the samples into the Finnigan SSQ-70 mass spectrometer. The sample components were ionized using chemical ionization with methane as the reagent gas. The GC column was operated from 40 to 300 C at a rate of 10 degrees per minute. The results of these analyses combined with their corresponding GC/FID area percents show the following:

Component I.D.	L-10059 N.R. N-EtFOSE Lot	W.R. N-EtFOSE Lot 547
N-Ethyl-carboxamides ($Rf-C(O)N(Et)H$)	0.22 %	1.76 %
N-EtFOS Amide ($C_8F_{17}SO_2N(Et)H$)	0.04 %	0.13 %
$C_2F_5SO_2N(Et)CH_2CH_2OH$	0.01 %	0.12 %
$C_3F_7SO_2N(Et)CH_2CH_2OH$	0.13 %	1.17 %
$C_4F_9SO_2N(Et)CH_2CH_2OH$	0.11 %	1.52 %
$C_8F_{17}SO_2N(Et)_2$	0.03 %	0.04 %
$C_5F_{11}SO_2N(Et)CH_2CH_2OH$	0.03 %	1.34 %
$C_6F_{13}SO_2N(Et)CH_2CH_2OH$	0.51 %	3.52 %
$C_8F_{17}SO_2N(Et)CH_2CH_2Cl$	0.08 %	0.16 %
$C_7F_{15}SO_2N(Et)CH_2CH_2OH$	0.82 %	1.40 %
N-EtFOSE $C_8F_{17}SO_2N(Et)CH_2CH_2OH$	96.09 %	87.16 %
$C_8F_{15}SO_2N(Et)CH_2CH_2OH$	0.78 %	0.50 %
$C_8F_{17}SO_2N(Et)(CH_2CH_2O)_2H$	0.17 %	-----
$C_8H_{17}SO_2N(Et)H$	0.16 %	0.37 %
$C_8F_{17}SO_2N(Et)CH_2CH_2OCO_2CH_2CH_3$ (or similar)	0.21 %	0.22 %
Other High Boilers	0.28 %	0.59 %

Component I.D.	L-1276 N-MeFOSE Lot
N-Methyl-carboxamides ($R_f-C(O)N(Me)H$)	1.46 %
$C_2F_5SO_2N(Me)CH_2CH_2OH$	0.24 %
$C_8F_{17}SO_2N(Me)_2$	trace
$C_3F_7SO_2N(Me)CH_2CH_2OH$	1.15 %
$C_8F_{17}SO_2N(Me)H$	trace
$C_4F_9SO_2N(Me)CH_2CH_2OH$	1.62 %
$C_5F_{11}SO_2N(Me)CH_2CH_2OH$	1.34 %
$C_6F_{13}SO_2N(Me)CH_2CH_2OH$	5.05 %
$C_8F_{17}SO_2N(Me)CH_2CH_2Cl$	0.13 %
$C_7F_{15}SO_2N(Me)CH_2CH_2OH$	1.69 %
N-MeFOSE $C_8F_{17}SO_2N(Me)CH_2CH_2OH$	83.88 %
$C_9F_{19}SO_2N(Me)CH_2CH_2OH$	0.87 %
$C_8F_{17}SO_2N(Me)(CH_2CH_2O)_2H$	0.34 %
$C_8H_{17}SO_2N(Me)H$	0.54 %
Other High Boilers	1.55 %

Further work has been done by GC on these samples which involves derivatization of the alcohols with trifluoroacetic anhydride (TFAA) and again with BSA (to give the trimethylsilyl ethers). This work was designed to investigate the potential problems that could be overlooked by any one method of analysis. Preliminary results show that analyzing the EtFOSE underivatized could hide a significant amount of N-EtFOS Amide under the C-3 alcohol peak in wide range material. However, analyzing the same material that has been derivatized with TFAA shows that any EtFOSE-chloride that is present in the sample is now completely masked by the derivatized C-8 alcohol. The BSA derivative has not been evaluated yet, but similar problems are expected because of the number of different components in the sample. The same sort of problems will most likely exist with MeFOSE and will be even more complicated in the analysis of MeFOSEA.

7/2/93

003107

GC/MS analyses of these samples were accomplished using a 25 m X 0.32 mm HP-1 GC column to introduce the samples into the Finnigan SSQ-70 mass spectrometer. The sample components were ionized using chemical ionization with methane as the reagent gas. The GC column was operated from 40 to 300 C at a rate of 10 degrees per minute. The results of these analyses combined with their corresponding GC/FID area percents show the following:

Component I.D.	W.R. N-EtFOSE Precut 97900-112-1	W.R. N-EtFOSE main cut 97900-112-2
N-Ethyl-carboxamides (R_f -C(O)N(Et)H)	68.34 %	1.27 %
C ₆ F ₁₃ SO ₂ N(Et)H	1.28 %	0.32 %
C ₂ F ₅ SO ₂ N(Et)CH ₂ CH ₂ OH	trace	trace
C ₇ F ₁₅ SO ₂ N(Et)H	trace	trace
N-EtFOS Amide (C ₈ F ₁₇ SO ₂ N(Et)H)	0.41 %	trace
C ₆ F ₁₃ SO ₂ N(Et) ₂	1.41 %	trace
C ₃ F ₇ SO ₂ N(Et)CH ₂ CH ₂ OH	1.53 %	0.51 %
C ₄ F ₉ SO ₂ N(Et)CH ₂ CH ₂ OH	0.62 %	0.42 %
C ₅ F ₁₁ SO ₂ N(Et)CH ₂ CH ₂ OH	1.48 %	2.16 %
C ₆ F ₁₃ SO ₂ N(Et)CH ₂ CH ₂ OH	20.16 %	60.86 %
C ₇ F ₁₅ SO ₂ N(Et)CH ₂ CH ₂ OH	3.52 %	22.55 %
N-EtFOSE C ₈ F ₁₇ SO ₂ N(Et)CH ₂ CH ₂ OH	1.14 %	11.92 %

Component I.D.	N.R. N-MeFOSE B.P. 132 97900-107-2
----------------	--

mw 137 possibly $\text{---SO}_2\text{N(Me)CH}_2\text{CH}_2\text{O---}$	0.58 %
$\text{C}_7\text{F}_{15}\text{SO}_2\text{N(Me)CH}_2\text{CH}_2\text{OH}$	0.41 %
N-MeFOSE $\text{C}_8\text{F}_{17}\text{SO}_2\text{N(Me)CH}_2\text{CH}_2\text{OH}$	98.19 %
$\text{C}_9\text{F}_{19}\text{SO}_2\text{N(Me)CH}_2\text{CH}_2\text{OH}$	0.59 %
$\text{C}_8\text{F}_{17}\text{SO}_2\text{N(Me)(CH}_2\text{CH}_2\text{O)}_2\text{H}$	trace
$\text{C}_8\text{H}_{17}\text{SO}_2\text{N(Me)H}$	trace
Other High Boilers	0.23 %

Component I.D.	W.R. N-EtFOS Amide 97900-111
N-Ethyl-carboxamides (Rf-C(O)N(Et)H)	10.23 %
$\text{C}_3\text{F}_7\text{SO}_2\text{N(Et)H}$	trace
$\text{C}_4\text{F}_9\text{SO}_2\text{N(Et)H}$	0.71 %
$\text{C}_5\text{F}_{11}\text{SO}_2\text{N(Et)H}$	3.58 %
$\text{C}_6\text{F}_{13}\text{SO}_2\text{N(Et)H}$	52.50 %
$\text{C}_7\text{F}_{15}\text{SO}_2\text{N(Et)H}$	20.06 %
N-EtFOS Amide ($\text{C}_8\text{F}_{17}\text{SO}_2\text{N(Et)H}$)	12.93 %
$\text{C}_8\text{F}_{15}\text{SO}_2\text{N(Et)H}$	trace
numerous other impurities of most homologs that include hydrides, chlorine in the backbone, and unidentified high boilers	trace

003109