United States
CONSUMER PRODUCT SAFETY COMMISSION
Washington, D.C. 20207

MEMORANDUM

DATE: May 10, 2005

TO : Dale R. Ray, Project Manager, Upholstered Furniture, Directorate for Economic Analysis

THROUGH: Andrew G. Stadnik, Associate Executive Director, Directorate for Laboratory Sciences (LS)
Edward W. Krawiec, Division Director, Electrical and Flammability Engineering (LSE)
for Joel R. Recht, Division Division Director, Chemistry (LSC)

FROM : David Cobb, LSC
Shing-Bong Chen, LSC

SUBJECT : Analysis of FR Chemicals Added to Foams, Fabric, Batting, Loose Fill, and Barriers*

SUMMARY:

Seven foam types were analyzed for flame retardant chemicals (FRC) to determine the identity and concentration consistency of FRC between sample pieces and within each piece. Spatial variation within the foam samples was not significant. FRC content for most foams varied 10-20% relative to the mean, though melamine content for the foam T was more variable. In some instances the measured concentrations did not agree with the manufacturer's stated chemical treatment level. Many loose fill, fabric, batting, and barrier samples were also analyzed for FRC content. Most had no significant FRC content.

BACKGROUND:

LSC was requested to determine the flame retardant chemical (FRC) load and variability of seven types of resilient foams, some of which are being considered by CPSC staff for use as a standard foam for evaluating the performance of other upholstered furniture components. The FRCs used to treat the foam include melamine, tris (1,3 –dichloro-2-propyl) phosphate (TDCP), and FM-550®. The material safety data sheet (MSDS) for FM-550® indicates it contains a mixture of halogenated aryl esters and aromatic phosphates, such as triphenyl phosphate.

Initially three slabs of each foam type T, U, P, and Y were submitted for analysis. Thirteen sample aliquots were obtained from each foam slab for each of the two chemical analyses.

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Aliquots were obtained from each of the 8 corners, the middle of each of the 4 side edges, and a center piece obtained from the middle of the foam slab. The 8 corner aliquots were identified C1-C8. The 4 side aliquots were obtained 2" interior from the middle of each of the 4 side edges and were identified S1-S4. The center aliquot was identified CE.

Subsequent sample submittal requests included additional samples of foams T, U, P, and Y, plus samples of foams Z, R, and S. Additionally, various fabric, loose fill, batting, and barrier samples have been submitted to LSC to chemically determine if FR chemicals were present, and if so identify them.

FOAM SAMPLES

Sample ID

Information on the seven types of foam samples along with manufacturer's stated chemical content and the average chemical load found by LSC are contained in table 1. Manufacturer's claimed FR chemical content was obtained from catalog specifications or ordering correspondence. [1,2,3]

Table 1. Foam Identification

CPSC Staff Designation	Color	Melamine % (w/w)		TDCP % (w/w)	
		Manufacturer Claim	CPSC Analysis	Manufacturer Claim	CPSC Analysis
U	White	0	Avg=1.2 Range=1.1-1.5	0	<0.05
Y	Yellow	12	Avg=11.1 Range=10.3-12.4	3	Avg=3.5 Range=3.1-4.6
P	Pink	30	Avg=28.4 Range=23.2-34.1	3	Avg=2.9 Range=2.6-3.4
T	Grey	2	Avg=2.2 Range=1.2-4.2	6	Avg=8.2 Range=6.6-9.2
S	Silver	0	<0.005	7.8	Avg=6.6 Range=6.3-6.9
J		0	<0.005	0	<0.05
K		0	<0.005	0	<0.05
L		0	<0.005	0	<0.05
		Melamine %		**FM-550 %**	
Z*	White	3.63	Avg=2.8 Range=2.2-3.3	6.96	Avg=6.0 Range=5.5-6.2
		Polybrominated Diphenyl Ether %		**FM-550 %**	
R*	White	NA	Avg 3.0% Range 2.9-3.2	4.1	Avg=3.3 Range=3.1-3.5

***** FM-550® and PBDE results are based on HPLC analysis. See below in FM-550® Extraction/Analysis section for details and FM-550® chemical compositions. FM-550® measurement of "R" foam gave significantly higher values indicating likely additional phosphorus compounds in "R" foam.

Note: Detection Limit for Melamine <0.005%, and detection limit for TDCP <0.05%

1. E-mail 3/24/05 from David Kelly, WTB Foam
2. E-mail 2/15/05 from Beat Niederoest, Foamex
3. E-mail 2/2/05 from Doug Sullivan, Hickory Springs

Melamine Extraction/Analysis:

Aliquots weighing from 20-200 milligrams were obtained from the various foam samples. The aliquots were placed in separate vials to which 10 ml or 20 ml of deionized water was added. Aliquots obtained from the P foam samples were extracted with 20 ml of deionized water solution due to the higher melamine content. The sample vials were covered, shaken, and then placed in water bath at 60°C for at least 24 hours to extract the melamine FR chemical. Some foam aliquots were extracted a 2nd time with deionized water to verify all melamine extracted during 1st extraction. Melamine levels for 2nd extraction were less than 5% of 1st extraction and are likely due to residual extract liquid remaining from 1st extraction. Some foam P aliquots were also extracted using Soxhlet extraction to verify all melamine had been extracted. Foam aliquots extracted using Soxhlet extractor had melamine results similar to foam aliquots extracted in the water bath. The extracts were analyzed using high pressure liquid chromatography (HPLC) to determine melamine content. The conditions that were used for the HPLC analysis were as follows:

Column: Waters Sperisorb 5µm NH_2, 4.6mm x 250 mm
Eluant: 95% acetonitrile, 5% water
Flow: 1.0 ml/min
Detector: Photodiode Array (UV-Vis)
Wavelength of maximum absorbance: 207 nm
Sample volume injected: 5µl

The peak for melamine occurred at about 10.2 minutes. Calculation of melamine was done by measuring the peak areas of standards and samples at this retention time, and doing a linear regression of peak area versus amount of melamine injected.

TDCP Extraction/Analysis:

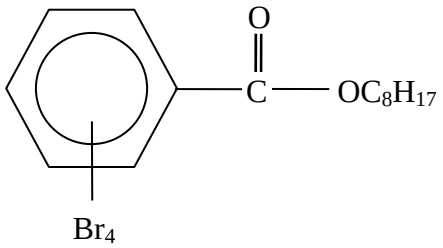
Aliquots weighing from 20-200 milligrams were obtained from each of the foam samples. The aliquots were placed in separate vials to which known volumes of acetonitrile were added. The sample vials were covered, shaken, and then allowed to sit at room temperature for at least 24 hours to extract the TDCP FR chemical. The extract was spiked with an internal standard of decanoic acid and analyzed using gas chromatography mass spectrometry (GC-MS) to determine TDCP content. The retention time for decanoic acid was1.7 minutes, and the retention time for TDCP was 5.0 minutes. A linear regression was developed on a set of reference samples containing TDCP and decanoic acid as an internal standard in acetonitrile solution. Peak area ratios were used to determine the concentration of TDCP in solution. The conditions that were used for the GC analysis were as follows:

Column-DB-5MS, 0.25 mm ID, 30 m, 0.25 um
Oven Temperature – 210 ^{o}C (1 min)/50 ^{o}C/min/260^{o}C
Injector temperature 280 ^{o}C
Carrier gas – Helium, 1.0 ml/min
Injection – 1ul liquid, 50:1 split injection

FM-550® Digestion/Analysis:

FM-550® consists of a mixture of halogenated aryl esters and aromatic phosphates. The company product specification sheet states FM-550® contains 4.3% phosphorus and 27.1% bromine. For foams containing FM-550®, 20-100 mg foam sample aliquots were digested in 2 ml of nitric acid, diluted to 10 ml and analyzed for phosphorus using an inductively coupled plasma atomic emission spectrometer. GC-MS and HPLC methods of analysis were developed after receiving a sample of FM-550® from the manufacturer. Solutions of FM-550® diluted in acetonitrile were analyzed by GC-MS and HPLC. Two components were detected by HPLC analysis. GC-MS chromatographs showed three major components. The mass spectra indicated they are triphenyl phosphate (4.7 minute retention time), tri(propylphenyl) phosphate (5.3 minute retention time) and octyl tetrabromo benzoate (7.4 minute retention time), with the structure given below:

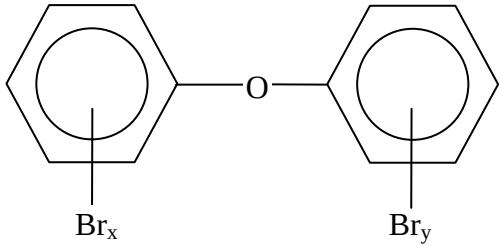
Figure 1:Structure and Formula for Brominated Component of FM-550®



The FM-550® content of foams Z and R was quantitatively determined by extracting 20-100 mg foam aliquots with acetonitrile. The extracts were analyzed using both GC-MS and HPLC. Decanoic acid was used as internal standard for the GC-MS analysis. Calibration curves were developed based on peak ratios for each of the 3 peaks detected by GC-MS analysis and the peak areas for the 2 peaks detected by HPLC analysis. The 2 peaks that were detected by HPLC analysis of FM-550® occurred at 3.9 minutes and 7.3 minutes. Neither of these peaks is attributable to triphenyl phosphate. The FM-550® results listed in Tables 8 and 9 are based on the 1st peak detected during HPLC analysis, which was the tallest peak.

The FM-550® results based on HPLC analysis for Foam Z are within 14% of the reported values for FM-550®, but Foam R was found to contain an additional chemical component. The chromatograph and mass spectra of this component matched the mass spectra of an FR chemical DE-71®. The product sheet for DE-71® indicates it consists of polybrominated diphyenyl ethers (PBDE's). Diluted solutions of DE-71® in acetonitrile were analyzed using GC-MS. The chromatogram clearly showed three major components. The mass spectra indicate they are tetrabromo diphenylether (5.5 minute retention time) and pentabromo diphenyl ether (6.8 and 7.4 minute retention times). The chromatographs and mass spectra for the foam R extracts also contained the same components. Tetra- (x+y=4) and penta-brominated (x+y=5) are represented in Figure 2. The presence of DE-71® in the foam R extracts was confirmed and quantitated using HPLC. Three major peaks were detected in the HPLC analysis of DE-71®. The peaks had retention times of 4.0, 5.2 and 6.6 minutes under the analysis conditions. The DE-71® results listed in table 8 are based on the peak with a retention time of 4.0 minutes by HPLC analysis. The foam R also was found to have more phosphorus content than predicted based on FM-550® analysis, indicating another phosphorus source may be present.

Figure 2: Structure and Formula for PBDE's



The conditions that were used for the GC analysis were as follows:

Column-DB-5MS, 0.25 mm ID, 30 m, 0.25 um
Oven Temperature – 210 ^{o}C (1 min)/40 ^{o}C/min/280^{o}C
Injector temperature 280 ^{o}C
Carrier gas – Helium, 1.0 ml/min
Injection – 1ul liquid, 50:1 split injection

The conditions that were used for the HPLC analysis were as follows:

Column: Waters Symmetry 3.5µm C18, 2.1mm x 100 mm
Eluant: 100% Acetonitile for FM-550®
90% Acetonitrile/10%Water for DE-71®
Flow: 0.6 ml/min
Detector: Photodiode Array (UV-Vis)
Wavelength of maximum absorbance: 227 nm
Sample volume injected: 5µl

Figures 3a-3d contain HPLC chromatographs of DE-71®, FM-550® and foam R extracts. Figures 4a-4h contain GC-MS chromatographs and mass spectra of FM-550® and DE-71®.

FABRIC, LOOSE FILL, AND BARRIER SAMPLES:

During the course of LSE flammability testing, various fabric, loose fill, batting, and barrier samples have been submitted to LSC to chemically identify and determine if FR chemicals were present. These samples were generally analyzed using Fourier transform infrared spectrometry (FTIR) to identify major components, digested in nitric acid or hydrochloric acid and analyzed using ICP to determine the presence of potential FR chemicals such as antimony (Sb), silicon (Si), phosphorus (P), and boron (B). Most of the samples were also analyzed using thermo gravimetric analysis (TGA) to get thermal degradation profile data such as decomposition temperature, and the amount of residual ash remaining after combustion. The loose fill and batting samples were also extracted with acetonitrile and analyzed by GC-MS to determine presence of any organic FR chemicals. No organic FR chemicals were detected. The loose fill and batting samples were also extracted with hot deionized water and analyzed for melamine using HPLC. Melamine was not detected in any of the loose fill or batting samples. Table 2 provides sample identification and analysis results.

Table 2. Fabric, LooseFill, Batting and Barrier ID and Analysis Results

Fabric ID	Sb %	Si%	B %	P %	FTIR ID	Thermal Analysis Results		%Ash
						1st Degra-dation Temp °C	2nd Degra-dation Temp °C	
24 - O	0.002	0.001	0.024	0.003	cotton	341		0.0
24 - S	0.010	0.001	0.023	0.000	cotton	349		0.0
24 - X	ND	0.002	0.022	0.003	cotton	344		0.0
24 - Y	ND	ND	0.029	0.002	cotton	344		0.0
24 - Z	ND	0.002	0.029	0.003	cotton	334		0.0
24 - Log # 1266	0.002	ND	0.029	0.004	cotton	342		0.0
24 - Log # 1267	ND	0.001	0.037	0.004	cotton	339		0.1
24 - Log # 1277	ND	ND	0.037	0.004	cotton	348		0.7
24 - Log # 1278	ND	0.003	0.023	0.003	cotton	341		1.1
24 - Log # 1279	ND	ND	0.031	0.004	cotton	346		0.8
24 - Log # 1289	ND	0.000	0.035	0.003	cotton	345		1.5
24 - Log # 1290	ND	0.001	0.037	0.004	cotton	344		0.0
24 - Log # 1291	ND	0.002	0.031	0.003	cotton	337		1.2
24 - Log # 1292	0.001	ND	0.029	0.003	cotton	336		1.0
5 - Log # 1302	ND	ND	0.035	0.004	Polyester/ Cellophane	339	434	1.2
23 - Log # 1315	ND	0.003	0.029	0.016	cotton	329		0.0
23 - Log # 1316	ND	0.001	0.032	0.014	cotton	333		0.0
23 - Log # 1317	ND	0.003	0.028	0.011	cotton	325		0.5
36 - Log # 1318	ND	0.011	0.038	0.021	Polyethyl acyrlate/ Polypropylene	361		9.0
37 - Log # 1319	ND	ND	0.037	0.005	Polypropylene	354		6.7
1 - Log # 1320	ND	ND	0.068	0.007	cellulose acetate	350		0.0
1 - Log # 1321	ND	ND	0.037	0.007	cellulose acetate	351		4.3
2 - Log # 1322	ND	ND	0.037	0.006	cotton	357		4.5
5 - Log # 1324	ND	ND	0.028	0.003	Polyester	340	437	0.0
24 - Roll 3	ND	0.001	0.026	0.001	cotton	348		2.2
24 - Roll 4	ND	0.004	0.028	0.001	cotton	343		0.0
24 - Roll A	0.010	ND	0.038	0.003	cotton	343		1.0
24 - Roll B	0.001	0.002	0.028	0.003	cotton	335		0.7

Table 2. Continued								
Fabric ID	**Sb %**	**Si%**	**B %**	**P %**	**FTIR ID**	**1st Degra-dation Temp °C**	**2nd Degra-dation Temp °C**	**%Ash**
24 – Log # 1378	0.027	0.005	0.005	0.000	cotton	349		0.0
17 – AA	0.020	0.004	0.002	0.000	polyamide			
Loose Fill ID								
A	0.022	0.003	0.002	0.001	polyester			
B	0.035	0.004	0.033	0.001	polyester			
C	0.017	0.001	0.137	0.003	polyester			
W	0.024	0.027	0.003	0.000				
V	0.000	0.025	0.001	0.000				
&	1.2	0.001	0.001	1.5	polyester			
Batting or Barrier	**Sb %**	**Si%**	**B %**	**P %**	**FTIR ID**			
W	0.026	0.005	0.037	0.001	polyester			
G	0.028	0.427	0.012	0.001	polyester			
P_1	0.013	0.015	0.003	0.003	polyester			
P_2	0.023	0.009	0.003	0.007	polyester			
$	---	*95% SiO_2	---	---	Silicon Dioxide			

* Determined by weight loss after heating to 550C

DISCUSSION OF RESULTS:

Table 3 is a summary table of the average results, range and standard deviations found for the three slabs of each of the four foam sample types (T, U, P, and Y) that were initially tested for chemical variability within and between blocks. Tables 4 and 5 contain the results of foams R, S, and Z. The following observations were noted:

a. The FR chemical load for foam Y samples was close to the manufacturer's stated treatment levels. The FR chemical content appeared consistent between the 3 foam slabs, and across the various sample locations.

b. Foam T had an average measured TDCP content of 8.2% versus the 6% content stated by manufacturer. The melamine content was not consistent between the foam slabs. One foam slab contained 3.6% melamine, 2 other foam slabs contained about 1.5% melamine, and other foam pieces averaged 2.0-2.5% melamine. The FR chemical content was more consistent across the various sample locations within a foam slab.

c. Foam U samples are the CPSC staff draft standard untreated foam used for the upholstered furniture testing. The foam is supposed to be untreated with FR chemical. TDCP was not detected, but melamine was detected throughout the foam slabs at concentrations of around 1%.

d. The average FR chemical load for foam P samples was close to the manufacturer's stated treatment levels, but there was considerable variability especially with melamine. Melamine levels detected ranged from 1.5 to 3.4%.

e. Foam Z had fairly consistent FM-550® levels based on phosphorus analysis and HPLC analysis. The average level was within 14% of manufacturer's estimated dosage, which is a process specification based calculation versus post production analytical measurement. Melamine showed more variability (2.2 -3.3%), and the average measured melamine was over 20% lower than the manufacturer's estimated dosage.

f. The FM-550® results for foam R averaged only 3.3%, about 20% lower than the manufacturer's stated claim. PBDE (DE-71®) was also detected in the R foam. The average concentration of DE-71® was 3.0%. The manufacturer initially claimed that no PBDEs had been added, then claimed that the product was being reformulated and they were unsure what formulation was provided.

g. FR chemicals were not detected in any of the fabric, loose fill, or batting samples submitted for analysis, except for the "&" loose fill sample. Phosphorus and antimony were detected in the "&" loose fill sample. The phosphorus compound could not be identified by GC-MS analysis. The phosphorus compound must be something other than FM-550® or TDCP, both of these compounds can easily be determined by GC-MS analysis.

Table 3. Summary Data for Foam Analysis			
Foam P			
Foam/Location	**Statistical Parameter**	**% Melamine**	**% TDCP**
Piece 1	Average	27.9	3.0
	Range	26.6 - 29.3	2.6 – 3.4
	Standard Deviation	0.7	0.2
Piece 2	Average	28.6	2.9
	Range	23.2 – 34.1	2.6 – 3.2
	Standard Deviation	2.3	0.2
Piece 3	Average	28.6	2.8
	Range	25.6 – 31.7	2.7 – 3.0
	Standard Deviation	1.5	0.1
Corners	Average	28.2	2.9
Sides	Average	28.7	3.0
Center	Average	28.0	2.8
All	***Average (Stdev)***	***28.4(1.6)***	***2.9 (0.2)***
Foam Y			
Foam/Location	**Statistical Parameter**	**% Melamine**	**% TDCP**
Piece 1	Average	11.2	3.4
	Range	10.6 – 12.4	3.2 – 3.8
	Standard Deviation	0.6	0.2
Piece 2	Average	11.0	3.5
	Range	10.3 – 11.7	3.1 – 4.6
	Standard Deviation	0.4	0.4
Piece 3	Average	11.2	3.6
	Range	10.7 – 12.2	3.1 – 4.0
	Standard Deviation	0.5	0.2
Corners	Average	11.1	3.5
Sides	Average	11.2	3.5
Center	Average	11.2	3.4
All	***Average(Stdev)***	***11.1 (0.5)***	***3.5 (0.3)***

Table 3. Cont			
Foam T			
Foam/Location	Statistical Parameter	% Melamine	% TDCP
Piece 1	Average	1.6	8.6
	Range	1.2 – 2.0	8.0 -9.2
	Standard Deviation	0.2	0.3
Piece 2	Average	1.4	8.2
	Range	1.2 – 1.8	7.4 – 9.2
	Standard Deviation	0.2	0.5
Piece 3	Average	3.6	7.9
	Range	3.4 -4.2	6.6 – 8.5
	Standard Deviation	0.2	0.5
Corners	Average	2.2	8.3
Sides	Average	2.1	8.1
Center	Average	2.3	7.7
All	***Average (Stdev)***	***2.2 (1.0)***	***8.2 (0.5)***
Foam U			
Foam/Location	**Statistical Parameter**	**% Melamine**	**% TDCP**
Piece 1	Average	1.2	0
	Range	1.1 -1.3	0
	Standard Deviation	0.1	0
Piece 2	Average	1.3	0
	Range	1.1 – 1.5	0
	Standard Deviation	0.1	0
Piece 3	Average	1.2	0
	Range	1.1 1.4	0
	Standard Deviation	0.1	0
Corners	Average	1.3	0
Sides	Average	1.2	0
Center	Average	1.2	0
All	***Average (Stdev)***	***1.2 (0.1)***	***0***

Table 4. Foams R and S Chemical Analysis Results

Foam Designation	Aliquot	%P	%FM-550®		% Melamine	%DE-71® based on HPLC
			Based on P	Based on HPLC		
R	1	0.23	5.3	Not tested	0	Not Tested
R	2	0.24	5.6	Not tested	0	Not Tested
R	SBC1	0.26	6.1	3.3	Not Tested	3.0
R	SBC2	0.26	6.0	3.3	Not Tested	2.9
R	SBC3	0.30	7.0	3.4	Not Tested	3.0
R	SBC4	0.26	6.7	3.2	Not Tested	3.0
R	SBC5	0.27	6.5	3.2	Not Tested	3.2
R	SBC6	0.30	7.1	3.2	Not Tested	2.9
R	SBC7	0.29	6.7	3.1	Not Tested	3.0
R	SBC8	0.28	6.5	3.3	Not Tested	3.0
R	SBC9	0.30	7.1	3.4	Not Tested	3.0
R	SBC10	0.27	6.2	3.5	Not Tested	2.9
R Average		**0.28**	**6.4***	**3.3**	**0**	**3.0**
			% FM-550®		**% Melamine**	**% TDCP**
S	1	0.24	0		0	6.9
S	2	0.25	0		0	6.3
S Average		**0.25**	**0**		**0**	**6.6**

***** Disagreement of phosphorus content and FM-550® content indicate likelihood of additional phosphorus compound present. HPLC data is believed to be more representative of FM-550® content.

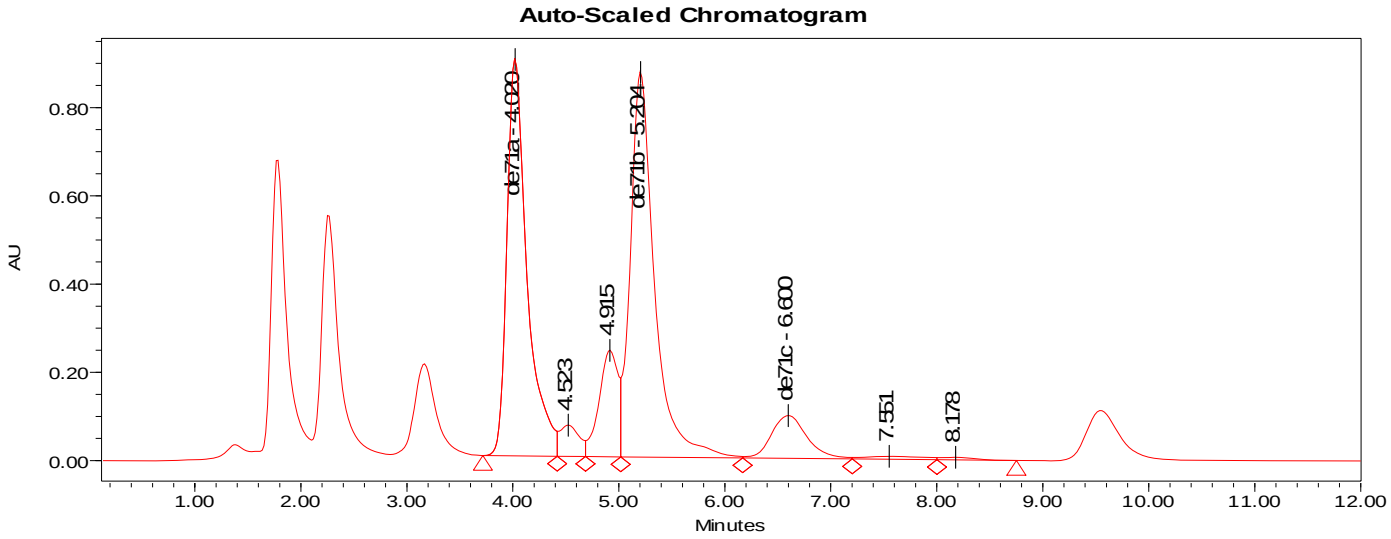
Table 5. Foam Z Chemical Analysis Results

Foam Designation	Slab No.	Location	%P	%FM-550®		% Melamine
				Based on P	Based on HPLC	
Z	1	A	0.26	6.1	Not tested	2.4
Z	1	B	0.26	6.0	Not tested	2.5
Z	1	C	0.26	6.0	Not tested	2.6
Z	1	D	0.26	6.0	Not tested	3.3
Z	1	E	0.26	6.1	Not tested	3.2
Z	1	F	0.27	6.4	Not tested	3.2
Z	1	G	0.27	6.2	Not tested	2.8
Z	1	H	0.26	6.1	Not tested	2.9
Z	2	A	0.27	6.2	Not tested	3.0
Z	2	B	0.3	6.9	Not tested	2.6
Z	2	C	0.25	5.9	Not tested	2.7
Z	2	D	0.25	5.9	Not tested	2.9
Z	2	E	0.26	6.0	Not tested	2.3
Z	2	F	0.26	6.1	Not tested	2.2
Z	3	A	0.26	6.1	Not tested	2.3
Z	3	B	0.27	6.2	Not tested	2.9
Z	3	C	0.27	6.2	Not tested	2.7
Z	3	D	0.26	6.1	Not tested	2.2
Z	3	E	0.25	5.9	Not tested	2.3
Z	3	F	0.26	6.1	Not tested	2.6
Z	4	A	0.24	5.7	Not tested	2.9
Z	4	B	0.25	5.9	Not tested	2.8
Z	4	C	0.26	6.0	Not tested	3.3
Z	4	D	0.26	6.0	Not tested	2.9
Z	4	E	0.27	6.2	Not tested	3.0
Z	4	F	0.26	6.0	Not tested	2.8
Z	4	G	0.27	6.2	Not tested	3.3
Z	4	H	0.28	6.4	Not tested	2.9
Z	SBC	1	0.28	6.4	5.9	Not tested
Z	SBC	2	0.29	6.7	6.2	Not tested
Z	SBC	3	0.29	6.8	6.0	Not tested
Z	SBC	4	0.31	7.3	5.5	Not tested
Z	SBC	5	0.29	6.8	5.9	Not tested
Z	SBC	6	0.29	6.8	5.9	Not tested
Z	SBC	7	0.32	7.4	6.0	Not tested
Z	SBC	8	0.31	7.1	6.8	Not tested
Z	SBC	9	0.31	7.3	6.0	Not tested
Z	SBC	10	0.30	6.9	6.1	Not tested
Average (stdev)			**0.26(0.01)**	**6.2 (0.4)**	**6.0 (0.5)**	**2.8 (0.3)**

Figure 3a. HPLC Chromatograph of R foam extract analyzed for DE-71®

Sample Information

SampleName	r1	Sample Type	Unknown
Vial	54	Date Acquired	4/18/05 10:07:52 AM
Injection	1	Acq Method Set	de71ms
Injection Volume	5.00 ul	Processing Method	de71pm
Channel	996	Date Processed	4/19/05 8:02:38 AM
Run Time	12.0 Minutes		



Component Results

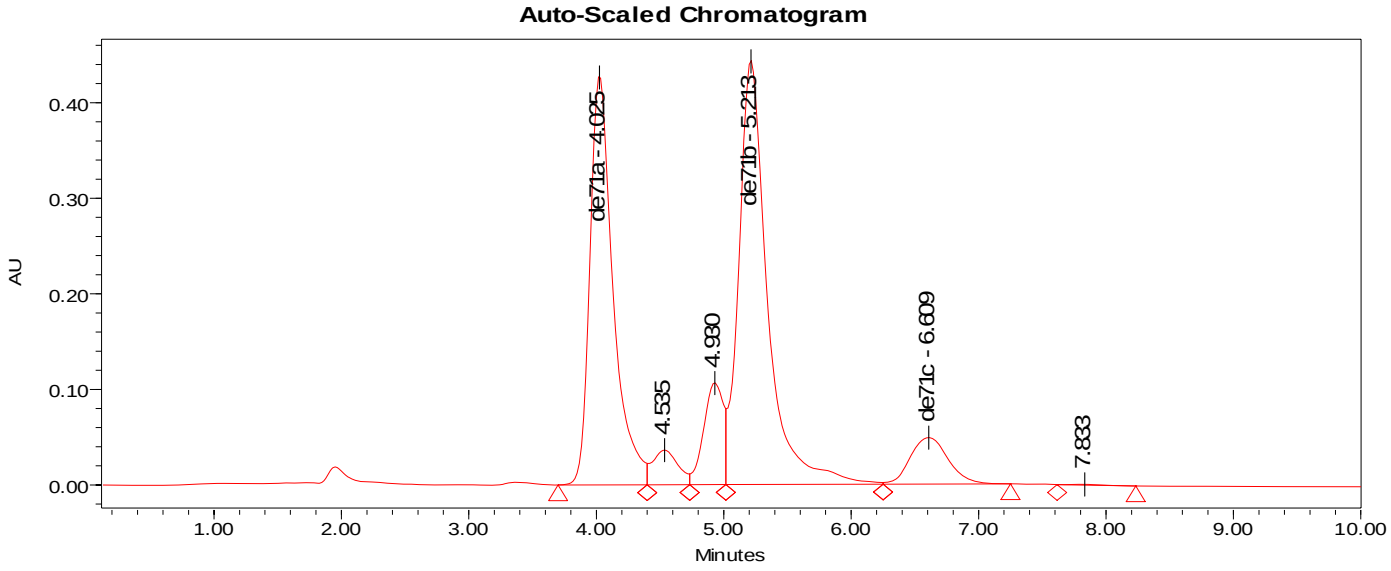
	Name	RT	Area	Height	Amount	Units
1	de71a	4.020	11915815	900281	409.770	
2	de71b	5.204	13796759	873119	364.451	
3	de71c	6.600	2252851	96971	365.397	

Figure 3b. HPLC Chromatograph of DE-71® standard

Current Date 5/4/05

Sample Information

SampleName	s3	Sample Type	Standard
Vial	51	Date Acquired	4/18/05 9:35:10 AM
Injection	1	Acq Method Set	de71ms
Injection Volume	5.00 ul	Processing Method	de71pm
Channel	996	Date Processed	4/19/05 8:02:36 AM
Run Time	10.0 Minutes		



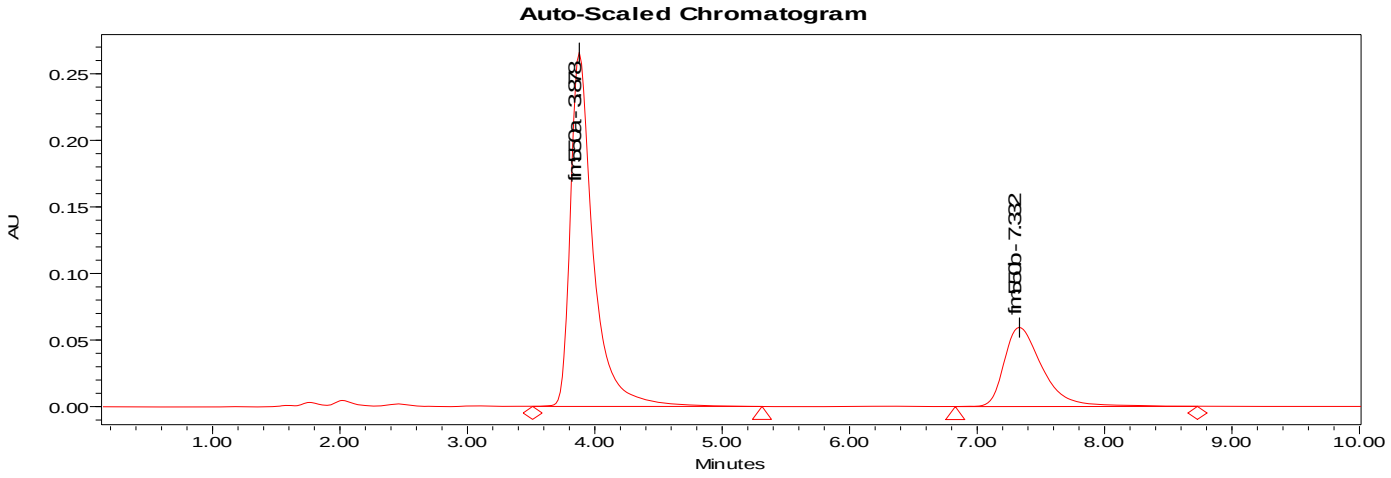
Component Results

	Name	RT	Area	Height	Amount	Units
1	de71a	4.025	5411988	429114	188.000	
2	de71b	5.213	6963186	443815	188.000	
3	de71c	6.609	1008083	48788	188.000	

Figure 3c. HPLC chromatograph of FM-550® standard

Sample Information

SampleName	400 ppm	Sample Type	Standard
Vial	15	Date Acquired	4/6/05 11:39:15 AM
Injection	1	Acq Method Set	dbms
Injection Volume	2.00 ul	Processing Method	fm550highconc
Channel	996	Date Processed	4/7/05 8:32:54 AM
Run Time	10.0 Minutes		



Component Results

	Name	RT	Area	Height	Amount	Units
1	Peak1	3.090				
2	fm550a	3.878	3313972	265668	400.000	
3	fm550b	7.332	1242145	59444	400.000	

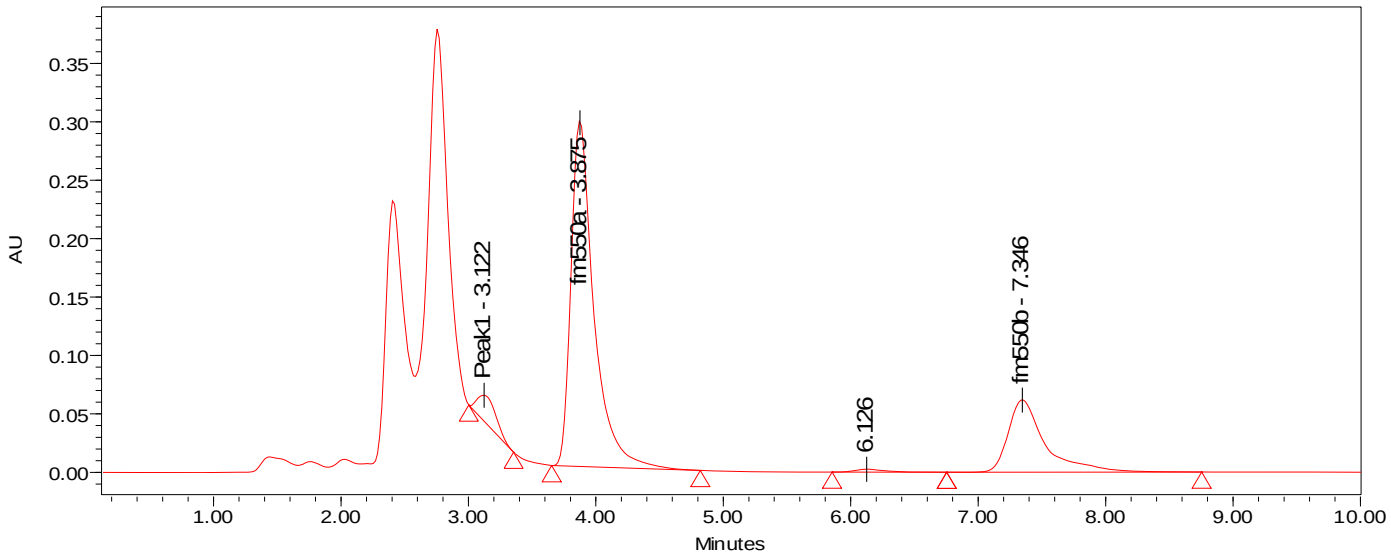
Figure 3d. HPLC chromatograph of extract of R foam analyzed for FM-550®

Current Date 5/4/05

Sample Information

SampleName	R1	Sample Type	Unknown
Vial	25	Date Acquired	4/6/05 2:43:57 PM
Injection	1	Acq Method Set	dbms
Injection Volume	2.00 ul	Processing Method	fm550highconc
Channel	996	Date Processed	4/7/05 8:24:48 AM
Run Time	10.0 Minutes		

Auto-Scaled Chromatogram



Component Results

	Name	RT	Area	Height	Amount	Units
1	Peak1	3.122	236747	22069		
2	fm550a	3.875	3694833	295909	448.155	
3	fm550b	7.346	1240258	61806	405.174	

Figures 4. GC-MS chromatographs and mass spectra of FM-550® and DE-71®

Figure 4a: FM-550 Chromatogram

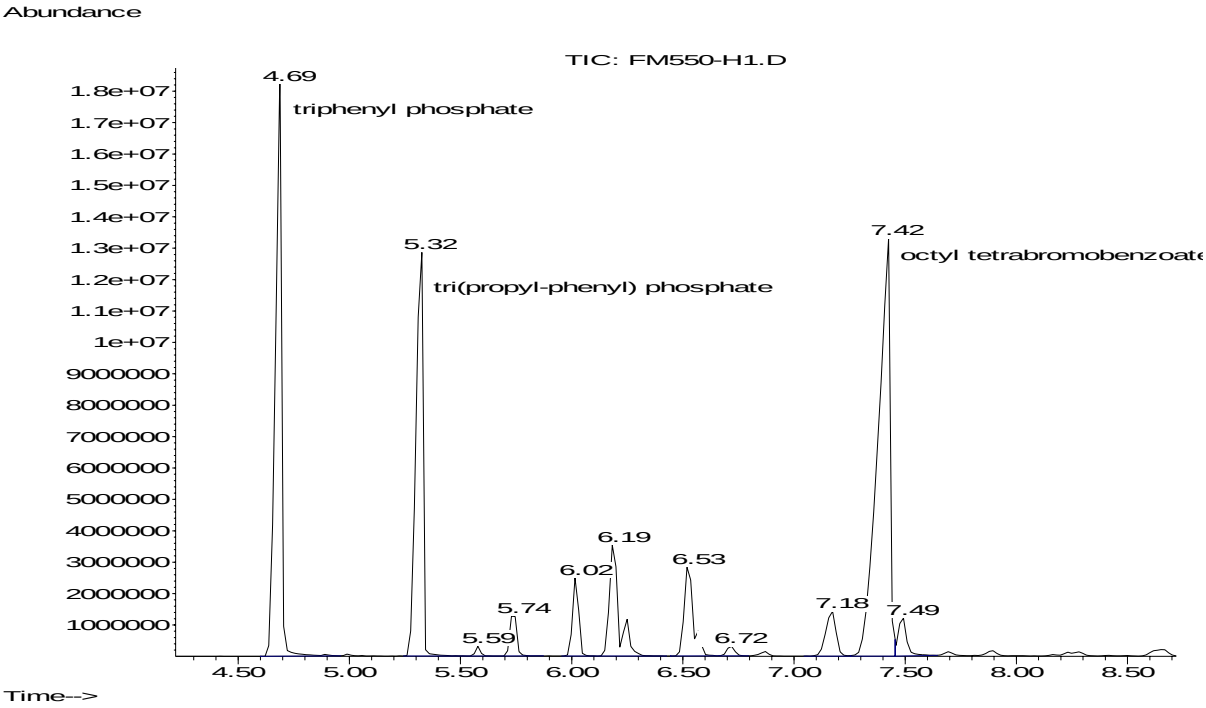


Figure 4b: Mass Spectrum for 4.671 minutes, triphenyl phosphate

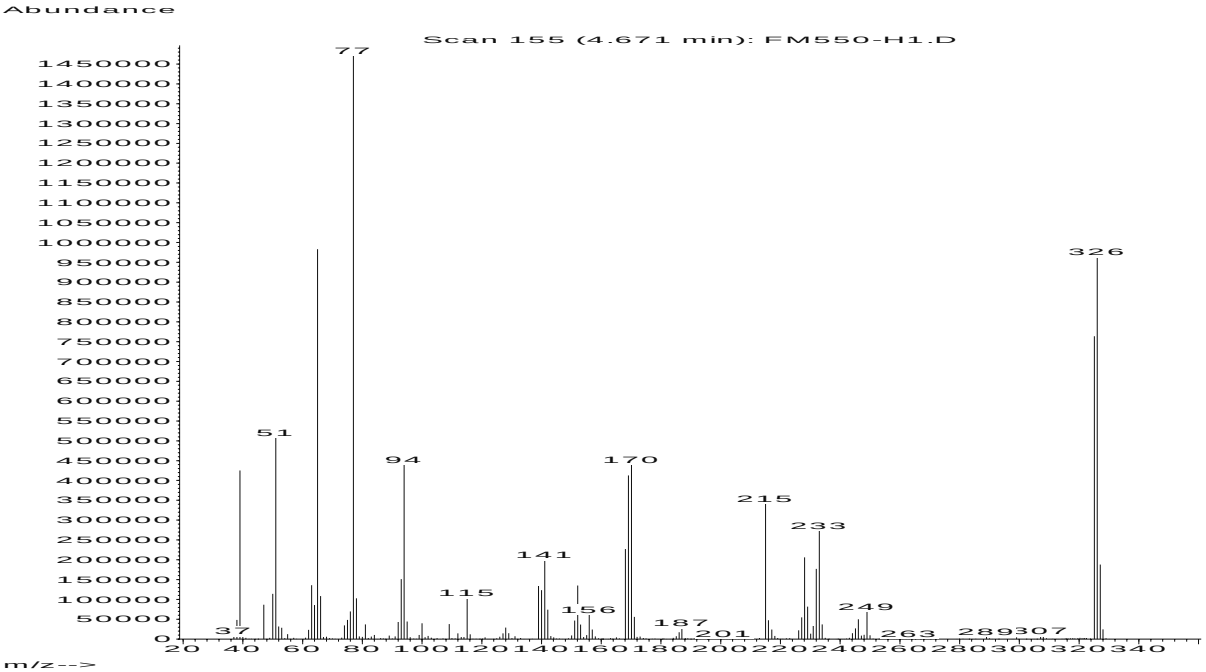


Figure 4c: Mass Spectrum for 5.326 minutes, tri-(propyl-phenyl) phosphate

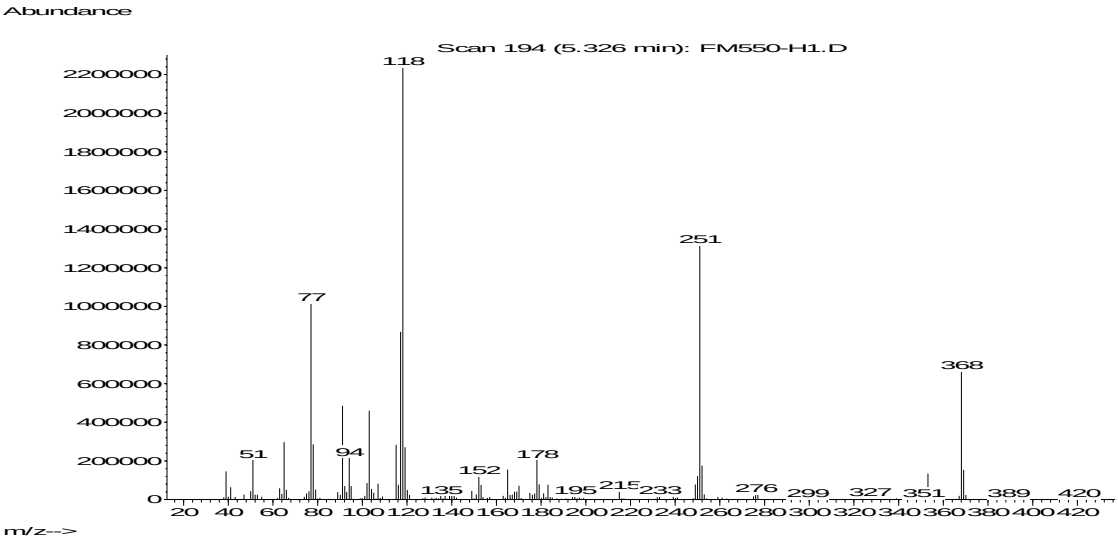


Figure 4d: Mass Spectrum for 7.408 minutes, octyl tetrobromobenzoate

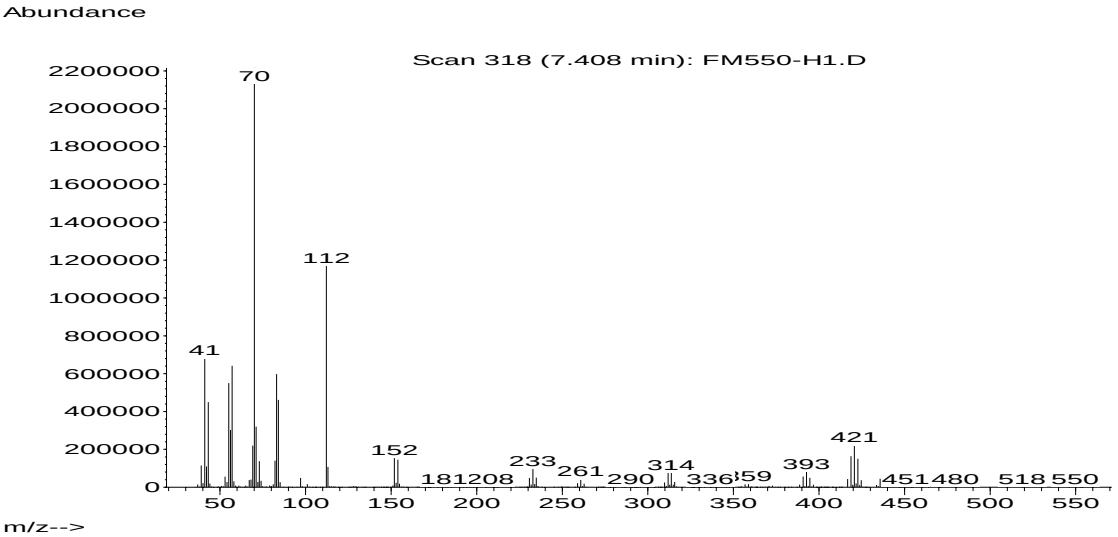


Figure 4e: DE-71® Chromatogram

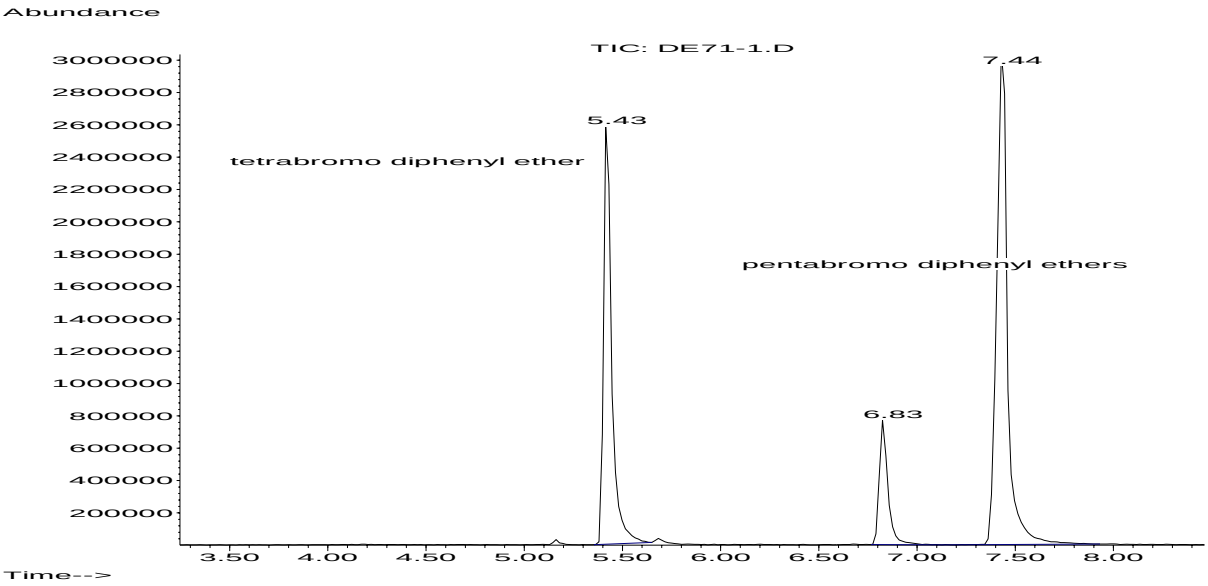


Figure 4f: Mass Spectrum for 5.431 minutes, tetrabromo diphenyl ether

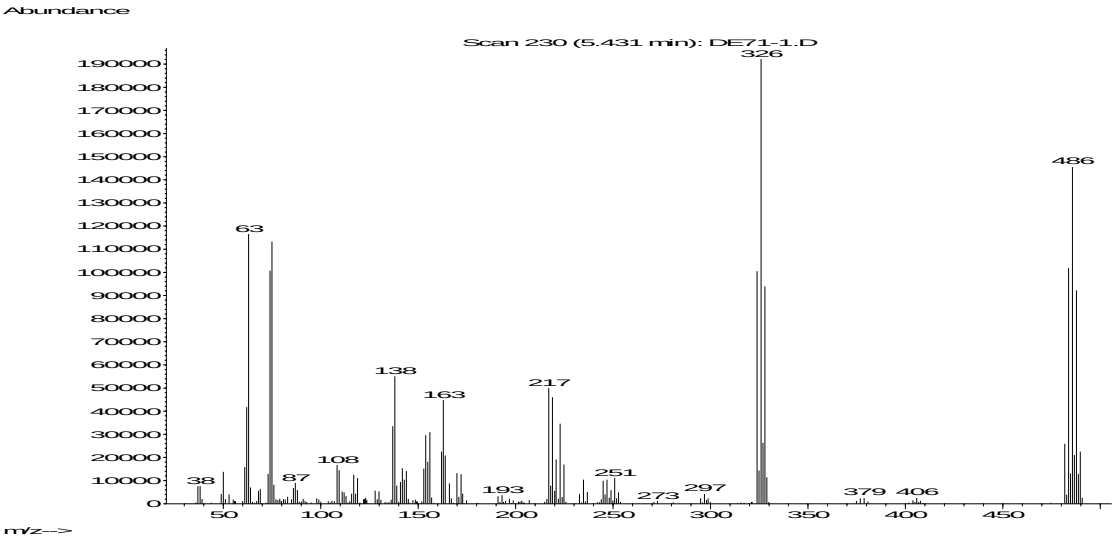


Figure 4g: Mass Spectrum for 6.825 minutes, pentabromo diphenyl ether

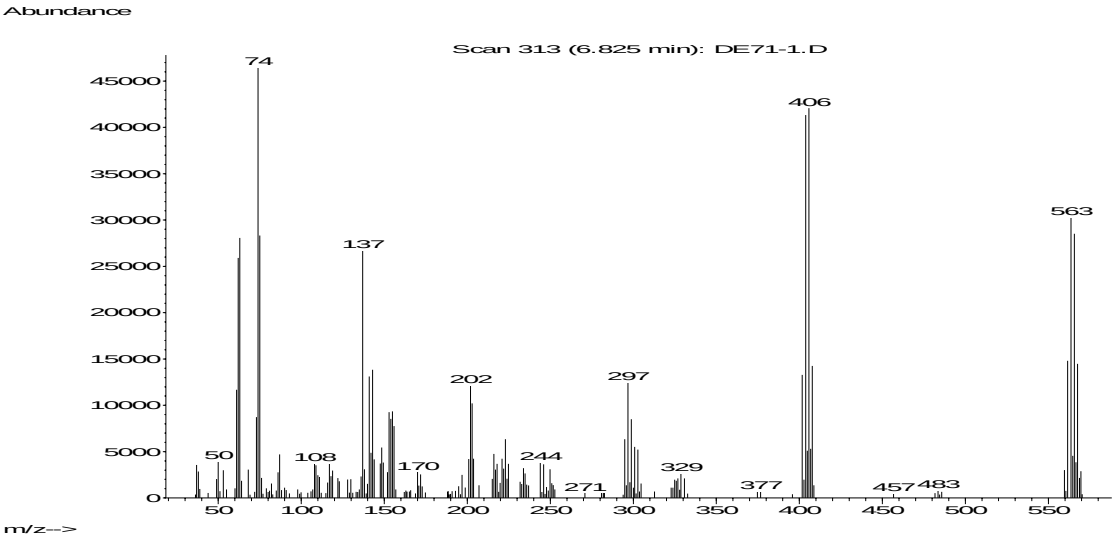


Figure 4h: Mass Spectrum for 7.412 minutes, pentabromo diphenyl ether