NWRI CONTRIBUTION 89-129

ANALYSIS OF CONTAMINATED FUELS

by

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May 1989

MANAGEMENT PERSPECTIVE

During the recent concern of possible contaminants in fuels, a random number of these collected samples were analyzed using the GC congener specific PCB methodology. The samples were also analyzed for chlorobenzenes and OCs contained in the non-polar eluate of the silica gel cleanup step. Since any PCB contamination would be expected in the form of industrial mixtures of the congeners known as aroclors, only Aroclor elution patterns were considered. On this basis, Aroclors were found in seven of the samples and their concentrations ranged from 58 to 349 ppb, significantly below the 1 ppm value that is the current regulatory limit used by other analysts. Using the congener method, it is possible to measure PCBs down to ppb level. The lower level of detection of congener method will allow 0.01 L of Aroclor waste added to a large tanker truck to be detected.

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PERSPECTIVE-GESTION

À la suite des récentes inquiétudes liées à la présence éventuelle de contaminants dans les carburants, on a soumis un nombre choisi au hasard de ces échantillons recueillis à une méthode de CPG d'analyse des congénères spécifiques des PCB. On a aussi effectué sur ces échantillons un dosage des chlorobenzènes et des composés organochlorés contenus dans l'éluat non polaire de la phase de nettoyage par silica gel. Comme on s'attendrait à trouver toute contamination par PCB dans les formes de mélanges industriels de congénères connues sous le nom d'aroclors, on n'a étudé que les configurations d'élution d'Aroclor. On a trouvé des aroclors dans sept des échantillons, et leurs concentrations variaient de 58 à 349 parties par milliard, soit bien en-dessous de la valeur de 1 partie par million qui est actuellement le seuil utilisé par d'autres analystes. Par la méthode des congénères, on peut mesurer des concentrations de PCB de l'ordre de la partie par milliard. Le taux le plus bas de la méthode de détection par les congénères permet de détecter la présence de 0,01 l de résidus d'Aroclor dans un gros camion-citerne.

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ABSTRACT

The GC methodology for PCB congeners, which also analyzes for chlorobenzenes and organochlorines eluted in the non-polar eluate of the silica gel cleanup, was used to analyze suspected fuel samples for PCBs and OCs. A total of 91 fuel samples were analyzed as were three intentionally spiked samples, eight duplicates, two secondary standards, four waste samples containing PCBs, three quality control samples and seven calibration standards. The calibration standards analyzed randomly during the analysis, exhibited a high degree of reproducibility. Only seven of the samples contained PCBs, but at concentrations well below the 1 ppm level. Also no chlorobenzenes or other OCs were found in the samples.

RÉSUMÉ

On a analysé par la méthode de CPG pour les congénères des PCB, qui dose également les chlorobenzènes et composés organochlorés élués dans l'éluat non polaire du nettoyage au silica gel, des échantillons de carburants où l'on soupçonnait la présence de PCB et d'OC. On a analysé un total de 91 échantillons de carburants, de même que 3 échantillons volontairement dopés, huit doubles, deux étalons secondaires, quatre échantillons de résidus contenant des PCB, trois échantillons de contrôle de la qualité et sept solutions-étalons. Les solutions-étalons, analysées au hasard au cours de l'opération, ont montré un haut degré de reproductibilité. Seulement sept échantillons contenaient des PCB, mais à des concentrations bien inférieures au seuil de 1 partie par million. De plus, on n'a trouvé ni chlorobenzènes ni composés organochlorés dans les échantillons.

1.0 INTRODUCTON

The recent awareness of the possibility of trans-border shipments of reconstituted fuels containing toxic wastes resulted in a number of activities being initiated to prevent any present or future shipments. After determining what constitutes an added contaminant in a fuel, the most important aspect is the ability to measure it accurately about the concentration levels equal to or below the regulatory limit. When dealing with petroleum and its products, another factor becomes important. Simple automobile fuels contain many additives, which may be mistaken for possible contaminants. Indeed, once crude oil leaves its reservoir, chemicals are usually added to assist in the ease of transporting it to refineries at which point other chemicals are added to aid the refining process. Also, chemicals are added to fuels at the refinery which assist in their combustion. Many of these additives will be left in the residue of the refining process (#6 fuel or bunker C). The art of the analyst is to distinguish between non-contaminants added to fuels by the petroleum industry, indigenous interfering constituents of the fuels and contaminants added by individuals who violate toxic waste disposal protocols and endanger the environment.

The major concern in the past few months was the possible presence of PCBs and organochlorine compounds in the reconstituted fuels. Techniques were available within the various laboratories of Conservation and Protection which could accurately monitor these compounds. These laboratories processed over 500 samples. The National Water Quality Laboratory was involved with the technology transfer of a new GC congener specific PCB method (Scott & Onuska, 1989) when the samples began to arrive. Most of the calibration of the method was complete but several difficulties involved with the computer software were not resolved, but it was decided that samples could be analyzed and any of the difficulties that impinged on the results could be resolved later by re-analyzing the result files. In addition, the PCB congener method also determines certain OCs.

Analysis for chlorinated hydrocarbons, which include PCBs, uses an electron capture detector (ECD) whose response to these compounds allows detection at the pg/μL concentration level. This detector responds to structural features of molecules of which the aromatic-chlorine moiety is only one example. Lighter fuels like gasoline will contain more additives, many of which will have heteroatoms (non-carbon) and other structural features which will cause ECD response. Heavier fuels will contain molecules containing heteroatoms indigenous to the oil and additives which may be EC active. Accordingly, chromatography of non contaminated fuels is very complex and any added contaminants will increase its complexity. To facilitate the interpretation of the chromatograms, dual capillary column chromatography was used (Scott and Misunus, 1988). This technique enhances the chemists' ability to confirm the presence of OCs and PCBs.

As any PCBs in contaminated fuels are anticipated to be present as an Aroclor (combinations of various congeners), and these Aroclors would not likely have undergone environmental degradation, a full Aroclor pattern is expected. Accordingly, the identification of a few isolated congeners does not denote the presence of PCBs. For a definitive confirmation of PCB contamination, at least 10 peaks common to an Aroclor must be present, with 80% of these peaks giving rise to the same concentration (within a factor of 2) as determined from the results from both columns. Also, one congener contributing over 50% of the total PCB concentration is not considered as an accurate identification.

This report presents the results of the analysis of 91 individual suspect samples plus standards, duplicates and quality control solutions. More samples could not be run as only one gas chromatograph was calibrated to run the methodology plus the fact that all documentation had to be done manually. Automation of this aspect, which is a vital component of such analysis is only now beginning.

2.0 METHOD

Samples were collected from various locations across Canada and shipped to the Conservation and Protection (C&P) laboratory in that region along with the proper documentation. These samples including those received by the Wastewater Technology Centre (WTC) laboratory underwent the following cleanup. A weighed aliquot of the sample (1.5 to 3 g was placed in a test tube and dissolved in 7 mL of hexane (Burdick and Jackson). A glass column (12 mm id x 350 mm), plugged at one end with clean silanized glass wool and filled with a 25 mm layer of Na_2SO_4, an 80 mm layer of silica gel (deactivated with 3% water), and with another 25 mm layer of Na_2SO_4, was washed with 30 mL of hexane which was discarded. The oil in hexane solution was transferred to the top of the column and the test tube was rinsed three times with 5 mL of hexane and the rinses added to the top of the column. An additional 20 mL of hexane was then passed through the column and the entire volume collected in a flask, to which a few boiling chips were added. Then a three stage Snyder column was attached and 3 mL of isooctane (Burdick and Jackson) added to the top of the column. This assembly was placed on a heating mantle and the solvent allowed to evaporate at a setting of 35°C to a final volume of 3 mL. The residue was quantitatively transferred to a test tube with hexane using rinses of 3 mL four times. The contents of the test tube was reduced to 2 mL at 35°C under nitrogen. A 0.5 to 1.0 mL aliquot of this was placed in a 1 mL vial, capped and was then ready for analysis. Of the 300 samples prepared by the WTC personnel, 91 samples were selected for the PCB congener analysis. In addition, there were a number of duplicates, spiked samples and quality assurance samples. Spiked samples were prepared by taking a known weight of the original sample and adding a known aliquot of standard PCB mixture. Each was cleaned up by the method outlined above.

Standard for spikes and for quality control were supplied by the Quality Control Project of Research and Applications Branch. To prepare the spiked sample, 1.0 mL of the standard was added to 3 g of a

sample and treated as above but with the final volume being 10 mL. Other standards were chromatographed. One standard was the one continually used in establishing the PCB congener methodology. This was run twice every 16 runs, and the WTC standard was run once.

The chromatographic conditions are presented in (Scott and Onuska, 1989) and more fully in (Scott, Onuska and Kohli, 1988). In short, an HP 5890 gas chromatograph equipped with dual EC detectors, dual 3393A integrators, splitless injection, and an HP 7673 automatic sampler was used for all analyses. Using this setup allows the analyst to make a single injection and have the sample split between two columns. The gas chromatograph and ancillary equipment was controlled by the LAS routine of the Real Time Executive software on an HP 1000 mini computer.

3.0 RESULTS

List in Table are the values obtained from the standards used in establishing the PCB congener methodology which were interspersed among the fuel samples. In addition, similar analyses conducted after all the fuel analyses were complete, so that any impurity from the fuels would be purged from the system are included. The listed values include the sums of the individual congeners as well as the number of congeners detected in an analysis. The low value of the coefficient of variance indicates that the sums are reproducible over time, a mark of a good method as well as good chromatography. This assures the analyst that the results of samples analyzed in the intervals between the standards are as accurate as one can expect. The results for the individual congeners are not listed here because of the large amount of space required for such a table. However, the agreement for congeners between the different analysis is very good, the coefficient of variance ranging between 1% to 10% for all the congeners.

Table 2 lists the results for the oil samples. The results for the entries {1} to {7} are high, all having potential PCB concentrations over 1 ppm. A compound having the same retention times as congener 3, (p-chlorobiphenyl), on both capillary columns is the major contributor to these high values. If congener 3 was present, other congeners in Aroclor 1221 and 1016 (1242) would also be detected. This is not the case. The total number of confirmed congeners present in these samples is low, not exceeding 14. Removing the contribution of congener 3 from the total, produces the values enclosed in the boxes in the first set of results in Table 2. These samples were checked for PCBs using GC-MS techniques after concentrating the sample by a factor of fifty times. No evidence for congener 3 was found.

Entries {106} to {111} illustrate the effect that would be expected for fuels tainted with PCBs. Three of these samples are regular samples and have very little suspected PCB congeners present, indeed, only entry {106} has one suspected congener. In the corresponding spiked samples there are about 58 contributing congeners, with a calculated PCB concentration of 1 ppm. The number of contributing congeners agree reasonably well with the results of the standard used as spiking solution which is shown in entries {104} and {105}.

Using the criteria outlined above eliminates samples for PCB contamination in {1} through {46}; {59} through {70}; {73} to {77}; {79}; {82} to {89} and {92} to {102}. In sample {48} there are 30 contributing congeners whose combined concentrations sum to 877 ppb. Another aliquot of the original sample was cleaned up and analyzed. The results fom this sample are shown in entry {49} where the number of contributing congeners is lower as is the total concentration. The remaining samples, {71}, {72}, {78}, {80}, {81}, {90} and {91} are listed in Table 3, reported with the concentrations of each congener present in each sample. These samples show evidence of PCB presence in the range 50 to 320 ppb of PCB but these concentrations are considerably lower than the limit of 1 ppm. In the chromatograms of the fuels, the peaks reported in this table are generally minor peaks in a more

complicated pattern. However the heights of each particular congener reported in Table 3 are greater than found in the lowest level of the secondary calibration solution dilutions (28 ppb) used to establish the linearity of the method (Scott, Onuska and Kohli, 1988). The levels reported here are quite low and elute close to other ECD peaks found in the same sample, therefore it is difficult to estimate the Aroclor responsible for the PCB presence, but an estimate of Aroclor 1254 or 1260 could be made. This is based on the number of later eluting peaks in most of these samples. Also, because of the low concentrations, it would be difficult to obtain confirmation using another technique such as GC-MS.

Table 4 contains the results from a typical waste sample contaminated with PCBs. The samples reported here had to be diluted 200 times relative to the other samples and standards so that the signals would not overload the detector. About 50 confirmed congeners per sample were identified with an average concentration of 615.4 ppm PCB found in the samples. A maximum of four congeners in any one set of results had values that were dissimilar on the two capillary columns for the same analysis. As the PCB concentrations were well above the ppm level, the pattern of the congeners were easy to discern over the background ECD activity. The pattern observed is similar to the pattern generated by most Aroclor 1254 solutions.

Table 5 lists the results from the quality assurance samples that were provided to all laboratories doing the contaminated fuel analysis. In discussions with those involved in the quality control it was discerned that the stock solution values are probably high but the two values for the spiked fuel are very close to the anticipated results.

For all samples, duplicates, and EP standard, the vials had been previously sampled. Accordingly, each sample had the vial septum penetrated. Some loss of material or solvent could have taken place. Also, in the small volumes contained in the vials, material from the septum could be deposited from the initial needle penetration, resulting

in negative peaks. This can affect the integration. No effort was made to recap, as any possible error source had already been introduced, and recapping at that time would only introduce additional problems.

The PCB congener methodology is also capable of detecting organochlorines found in the non-polar extract of the silica gel cleanup method. These include 1,2,3-, 1,3,5-, 1,2,4-trichlorobenzene, 1,2,3,4-tetrachlorobenze, pentachlorobenzene, hexachlorobenzene, hexachlorocyclobutadiene, heptachlor, aldrin, octachlorostyrene, p,p'-DDE, endrin aldehyde, o,p'-DDT, p,p'-DDD, p,p'-DDT, photomirex, and mirex. Review of all the output data showed there was no confirmed presence of any of the above compounds in any of the samples.

ACKNOWLEDGEMENTS

The authors wish to thank the many laboratory staff who spent long hours preparing the samples for analysis. In addition, the efforts of Rob Sampson and John Neate, who as laboratory managers, facilitated all arrangements that allowed prompt and facile analysis of the samples.

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TABLE 1

STANDARD PCB SOLUTION

Sample #	TEC15002	OIL23019	OIL22002	OIL020012	OIL021007	OIL22013
Total(pg/uL) =	2290	2206	2102	2254	2231	2217
# congeners	80	81	74	76	75	74

Sample #	OIL23002	TEC13002	TEC13013	TEC14002	TEC14009
Total(pg/uL) =	2040	2229	2517	2326	2206
# congeners	77	79	78	81	77

Mean (pg/uL)	Std Dev	CV
2233	158	0.07

TABLE 2

SUMMARY OF ANALYSIS FOR FUEL SAMPLES

	{1}	{2}	{3}	{4}	{5}	{6}	{7}
Sample #	ORCG006	ORCG002	ORCG007	ORGC003	ORCG03D	ORGC005	ORGC004
Conc. (ppb)	2620	5536	515	5445	5445	5810	3666
Without the contribution of congener 003	475	652	62.9	528	528	695	531
# of congeners	6	9	14	10	11	10	10
# of dissimilar congeners	2	3	5	4	5	4	5

	{8}	{9}	{10}	{11}	{12}	{13}	{14}
Sample #	ORCG04D	43008	43009	43007	36807	36820	36813
Conc. (ppb)	127	63.4	285	13.7	25.9	17.9	73.3
# of congeners	11	7	8	7	8	6	10
# of dissimilar congeners	2	5	3	4	7	2	4

	{15}	{16}	{17}	{18}	{19}	{20}	{21}
Sample #	36898	43005	36811	43010	43010D	43011	43015
Conc. (ppb)	7.69	15.4	63.7	318.4	349	145.7	683.1
# of congeners	6	6	10	20	11	11	12
# of dissimilar congeners	5	3	3	6	5	7	2

	{22}	{23}	{24}	{25}	{26}	{27}	{28}
Sample #	36810	43016	43074	43077	43077D	43012	43076
Conc. (ppb)	38	191.4	229.4	66.7	165	351	95.7
# of congeners	10	12	13		11	15	14
# of dissimilar congeners	9	5	3	4	4	3	10

	{29}	{30}	{31}	{32}	{33}	{34}	{35}
Sample #	43075	ORCG001	ORCG001	PB3	PB1-06	PB-1	43017
Conc. (ppb)	65.3	297	113.7	209	49.1	93.6	245.8
# of congeners	6	6	11	17	7	22	14
# of dissimilar congeners	3	1	4	4	2	10	1

	{36}	{37}	{38}	{39}	{40}	{41}	{42}
Sample #	43018	ORGC014	ORGC015	ORGC016	ORCG016D	ORCG017	ORCG018
Conc. (ppb)	332	33	172	401	316.8	45	30
# of congeners	10	6	5	6	9	5	7
# of dissimilar congeners	5	1	1	1	3	1	2

TABLE 2 (Cont.)

SUMMARY OF ANALYSIS FOR FUEL SAMPLES

	{43}	{44}	{45}	{46}	{47}	{48}	{49}
Sample #	43013	43020	43020D	43021	43021D	43025	43025D
Conc. (ppb)	330	693	359	2.3	142.6	877	207
# of congeners	12	7	9	43	10	30	1
# of dissimilar congeners	3	1	3	2	2	4	5

	{50}	{51}	{52}	{53}	{54}	{55}	{56}
Sample #	43019	43019D	PB1-13	PB1-14	PB1-15	PB1-16	PB-2
Conc. (ppb)	1586	9372	168	211	30.8	19.8	69.3
# of congeners	5	5	24	21	9	11	11
# of dissimilar congeners	4	2	2	1	5	1	3

	{57}	{58}	{59}	{60}	{61}	{62}	{63}
Sample #	PB1-07	PB4D	PB1-05	PB1-05D	PB1-12	36828	PB1-08
Conc. (ppb)	3.92	27.6	24.1	20.8	74.1	633	13.3
# of congeners	1	8	4	8	11	27	10
# of dissimilar congeners	1	2	2	3	4	5	5

	{64}	{65}	{66}	{67}	{68}	{69}	{70}
Sample #	LS44000	LS43999	PB3-02	PB3-02D	LS439919	LS43831	LS43831D
Conc. (ppb)	19.2	23.9	21.6	42	105	92	408
# of congeners	16	22	7	6	18	11	10
# of dissimilar congeners	5	4	1	2	0	3	0

	{71}	{72}	{73}	{74}	{75}	{76}	{77}
Sample #	LB1-14	LB1-08	ORCG020	PB3-03	LS43833	LS43921	43013
Conc. (ppb)	340	271	90	15.98	9.89	8.37	177
# congeners	23	24	16	14	15	16	13
# of dissimilar congeners	1	1	9	3	8	1	10

	{78}	{79}	{80}	{81}	{82}	{83}	{84}
Sample #	LS43832	PB3-01	LS43998	LB1-13	LB1-11	LB1-19	LB1-04
Conc. (ppb)	77	55	58	123	31.7	67.8	15.9
# congeners	37	11	19	21	10	16	7
# of dissimilar congeners	3	2	0	2	0	0	1

TABLE 2 (Cont.)

SUMMARY OF ANALYSIS FOR FUEL SAMPLES

	{85}	{86}	{87}	{88}	{89}	{90}	{91}
Sample #	LB1-17	LB1-21	LB1-16	LB1-23	LS43996	LB1-18	LB1-24
Conc. (ppb)	25.1	103	42.6	106.9	3.8	139	132
# congeners	10	14	10	14	12	20	20
# of dissiliar congeners	1	2	0	5	1	1	0

	{92}	{93}	{94}	{95}	{96}	{97}	{98}
Sample #	EPORW2	EPORW3	LS43921	LB1-22	LB1-01	LB1-15	36827
Conc. (ppb)	7.42	3.1	50	107	162	71.8	410
# congeners	6	9	13	12	18	23	13
# of dissimilar congeners	1	0	1	0	0	3	6

	{99}	{100}	{101}	{102}	{103}	{104}	{105}
						EPS	EPS
Sample#	LB1-06	LB1-05	LB1-02	LB1-12	LB1-07	STD	STD.
Conc. (ppb)	31.8	42.4	86.7	25.7	116	1110	1048
# congeners	5	11	14	11	19	57	55
# of dissimilar congeners	0	1	0	0	0	0	0

	{106}	{107}	{108}	{109}	{110}	{111}
		SPIKED		SPIKED		SPIKED
Sample #	LB1-03	LB1-03	LB1-22	LB1-22	LB1-09	LB1-09
Conc. (ppb)	20.8	1128	107	710	59.2	1241
# congeners	11	57	12	59	10	60
# of dissimilar congeners	1	0	0	0	0	0

TABLE 3

CONCENTRATION OF CONGENERS in pg IMPINGING ON DETECTOR

Congener Number	Sample # {71} LB1-14	Sample # {72} LB1-08	Sample # {78} LS43832	Sample # {80} LS43998	Sample # {81} LB1-13	Sample # {90} LB1-18	Sample # {91} LB1-24
3			15.52				
Mono Total	0	0	15.52	0	0	0	0
4(+10)			2.99	14.35			
5(+8)			6.83	6.96		25.19	29.28
6	41.64		7.51			12.01	
7		13.67	0.96	5.22		5.06	
12(+13)	126.8		0.53				
Di Total	168.44	13.67	18.82	26.53	0	42.26	29.28
16(+32B)			0.88				
17			1.67				
18			4.42				
19						12.71	
22			0.82				
25	10.13				7.78	3.18	
26			0.64				
29		6.61			4.26		
Tri Total	10.13	6.61	8.43	0	12.04	15.89	0
40			0.58				
41(+64+71)			1.85				
42					0.71		
44			3.84				
45	5.10		0.67		2.62	1.05	
46					2.51		
47(+48A)	2.81						0.68
48						1.26	
49			1.19				
52			1.91				
70(+76)					0.68		
Tetra Total	7.91	0	10.04	0	6.52	2.31	0.68
82						2.20	
83						1.49	1.88
84	0.65						
87			0.86				1.62
89						0.94	1.44
95(+66B)		0.61					
101			3.57	1.30	0.60		
105(+132)	5.81	10.29	1.29	2.87	6.74		
110			1.32				
118			1.51			2.87	
PentaTotal	6.46	10.29	9.16	4.17	7.34	7.5	4.94
128(+182)	5.15	10.67	0.57	1.29			5.58
135(+144)							3.18
137	5.11	5.83			5.71		

TABLE 3 (CONT.)

	Sample # {71} LB1-14	Sample # {72} LB1-08	Sample # {78} LS43832	Sample # {80} LS43998	Sample # {81} LB1-13	Sample # {90} LB1-18	Sample # {91} LB1-24
138		10.45	1.00	1.99			
141	1.72	2.93		0.68		1.74	1.92
153						5.47	6.64
156(+171)	9.39	14.49	0.70		11.29	8.34	8.04
158						3.40	3.46
167	4.79	6.28				4.66	4.49
Hex Total	21.37	44.37	2.27	3.96	17.00	18.95	28.82
170	10.44		0.33	2.35	12.87		
172		9.00					
174		9.79	0.37	1.07			4.42
175			0.45				
177	2.82	4.90	0.24		3.35		
178	4.37	6.21			5.38	4.33	4.44
180	6.88	20.68	0.93	1.71	7.66		6.97
183	4.94	8.54					5.01
185		5.93			5.15	4.22	4.21
189			0.16	1.00			
190		4.93					
191	1.56	2.20		0.32	1.98		
Hepta Tot.	20.57	72.18	2.15	4.10	23.52	8.55	25.05
194	3.25	6.07		0.25	3.93		1.86
195		22.51	0.41	1.56	17.49		
196(+203A)	7.07	13.19	0.43	1.40			5.65
199		3.66					
200						7.30	
201	6.97	11.51	0.37	1.32			
205	1.33	1.89			1.64	1.01	0.71
Octa Total	18.62	58.83	1.21	4.53	23.06	8.31	8.22
206	1.91	3.68	0.38	0.16	2.96		
207				0.13			
Nona Total	1.91	3.68	0.38	0.29	2.96	0	0
Tot.(pg/uL)	=255.41	209.63	67.98	43.58	92.44	103.77	96.99

TABLE 4

RESULTS FOR CONTAMINATED WASTE SAMPLE

Sample #	EPORHM1	EPORHM2	EPORHM3	EPORHM5	EPORHM4
Conc.(ppm)	549.2	796.6	479.5	606.5	645.3
# congeners	49	53	46	50	51
# of dissimilar congeners	3	2	4	3	4

TABLE 5

RESULTS FOR QUALITY ASSURANCE SAMPLES

Sample #	TEC13004	TEC13005	TEC13006
Description	Stock Solution	Spiked Fuel	Spiked Fuel
Conc. (pg/ul)	789.63	979.1	1008.15
		65.3 ug/g	67.2 ug/g
# congeners	74	76	80