

Simultaneous analysis of residual contaminants in drinking water using Programmed Temperature Vaporization (PTV) for Large Volume Injection (LVI) and GC-MS/MS

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1. Overview

Control and prevention of environmental pollution have emerged as paramount global concerns. Activities like industrial discharge, agricultural run-off, and improper waste disposal introduce a wide range of contaminants into various water bodies including drinking water.

Despite the multiple treatments and filtration processes that drinking water (Figure 1) undergoes before reaching consumers, the accurate quantitation of the target contaminants is of great importance. This involves tedious extraction and concentration processes followed by instrumental analysis.



Figure 1 Drinking Water

2. Introduction

This study aimed to develop a single analytical method for the simultaneous analysis of over 60 residual contaminants, including Poly-Aromatic Hydrocarbons (PAH), Poly-Chlorinated Biphenyls (PCB), and pesticides, in drinking water. Traditionally, multiple methods are being employed for analyzing these contaminants. Most common ones include, extraction using solvents in large separating funnel or SPE cartridge followed by volume concentration by evaporation. These methods require huge sample and solvent quantities. Due to these factors like large sample size handling, concentration steps etc., method may show poor reproducibility.

By utilizing Programmed Temperature Vaporization (PTV) for Large Volume Injection (LVI), the method significantly reduces the initial sample amount required for preparation, pre-treatment steps, and solvent consumption compared to conventional approaches, while eliminating the need for final sample concentration. The optimized procedure achieved limits of quantification (LOQs) as low as 0.005 ppb and 0.05 ppb, demonstrating high sensitivity without compromising efficiency.

Validation parameters like specificity, linearity, recovery and precision were studied as per SANTE guidelines^[1]. For linearity and recovery study, matrix match calibration was used.

3. Materials and methods

For this study, reference standard mixtures of over 60 contaminants regulated in drinking water were procured from Restek® Corporation. It included PAH, PCB and GC amenable pesticides.

Drinking water sample was extracted and used to prepare matrix-matched calibration standards and spiked samples. This method was validated for criteria mentioned in SANTE Guidelines.

GCMS-TQ8040 NX (Figure 2), manufactured by Shimadzu Corporation Japan, was used to quantify residual contaminants in drinking water sample.

3-1. Method development

Instrumental method was developed based on chromatographic and mass spectrometric parameters. Use of Smart Environmental Database (for PAH, PCB) and Smart Pesticides Database Ver.2 (for Pesticides) enabled quick instrumental method optimization for higher throughput. For all the contaminants, 1 target and 2 reference MRM transitions were included in the method. Shimadzu's 'LabSolutions Insight' software was used for data processing, which helped in evaluating validation parameters with ease. This greatly reduced the method development and optimization time. Pretreatment method involved use of small sample quantity and simple extraction process for better and consistent recoveries.

3-2. Sample and standard preparation

Locally purchased drinking water sample was used for this study. 30 mL of sample was extracted using 3 mL of dichloromethane (DCM) solvent along with pre-heated sodium chloride (AR grade) in optimized proportion to maximize recoveries of contaminants at trace level. The mixture was vortexed for 2 minutes. The mixture was allowed to settle. Following this, 2 mL of lower DCM layer was collected

with gas tight syringe and transferred into glass vial. To this, 50 mg anhydrous sodium sulphate was added and mixed well. Then the supernatant layer of DCM was removed, filled in GC vial and injected. During extraction itself, the sample was concentrated 10 times avoiding additional evaporation & reconstitution steps. All samples were analyzed as per conditions shown in table 1.

Preparation of solvent standard concentration levels

The PAH, PCB, and multi-residue pesticides mixtures were mixed and diluted using ethyl acetate to prepare 'Standard Mixture Stock Solution' of about 1000 ppb. From this, seven concentration levels of 0.25 ppb to 100 ppb were prepared using DCM.

Preparation of matrix matched standard linearity levels

The blank sample extract (matrix blank) prepared as per section 3-2 was used as a diluent. It was spiked with above solvent standard levels to prepare matrix match linearity of 0.025 to 10 ppb. It was ensured that the v/v proportion of matrix blank and solvent standard is kept same to keep the matrix effect constant throughout the linearity levels.

Preparation of spike samples (Recovery samples)

To determine the extraction efficiency of the method, recovery study was conducted. For this, 30 mL sample was spiked with 'Standard Mixture Stock Solution' to prepare recovery samples of 0.005 and 0.05 ppb. The spiked analytes were then extracted, analyzed and quantified against matrix matched linearity to study their recoveries. As the sample is 10 times concentrated during extraction, the actual concentration injected on instrument will be 10-fold.

3-3. Analytical Conditions

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Table 1 Instrument configuration and Analytical Conditions: GC-MS/MS									
System Configuration									
Instrument	: GCMS-TQ8040 NX								
Auto-injector	: AOC TM -20i + s								
Column	: SH-I-5SiI MS (30 m × 0.25 mm I.D., df = 0.25 μ m) (P/N:221-75954-30)								
Liner	: Restek® Topaz PTV Liner, with wool								
GC									
Injection Type	: PTV								
Injector temp. program	: 45 °C (0.50 min), 400 °C/min to 330 °C (63 min), -400 °C/min to 45°C (0.28 min)								
Column oven temp	: 35 °C (5 min), 30 °C/min to 120 °C (0 min), 4 °C/min to 330 °C (3.67 min)								
Run time	: 55 min								
Injection mode	: Split (Split Ratio: 5)								
Injection volume	: 50 μL (5 μL x 10 Multi-Injection Counts)								
Carrier gas	: He								
Column Flow	: 2.00 mL/min (Constant mode)								
MS									
Interface temp.	: 330 °C								
Ion source temp.	: 250 °C								
Ionization mode	: EI								
Solvent cut time	: 7.5 min								
Loop Time	: 0.3 sec								

Table 2 Summary results

14 Phensanthrene 21,230 28,00-18,00 28 0,9761 0,005 87,08 13,26	avi	e 2 Summary results							
2 Accenaphthylence 13.766 15.70-10-10.10 20 9.9770 0.05 118.92 4.65 5 Fluorene 16.38 165.10-1610 20 0.9392 NA* 12 Anthracene 20.937 178.10-152.10 20 0.9396 NA* 12 Anthracene 21.230 28.800-180.00 20.936.10 10.05 87.08 18.25 6 Fluoranthene 21.230 28.800-180.00 20 9.936.10 0.05 120.02 3.62 22 Beracyllthroache 35.421 228.10-220.10 20 0.939.0 0.05 118.42 3.49 55 Beracyllthroache 41.315 222.00-280.0 20 0.931.0 0.05 118.43 3.49 56 Beracyllthroache 41.35 222.00-280.0 20 0.931.0 0.05 118.43 11.66 58 Beracyllthryence 42.675 287.00-260.0 20 9.931.0 0.	ID	Compound Name		•	CE	(r²)			
1.	1	Napthalene	9.677	128.10>102.10	20	0.4136	NA*	-	-
15 Filter 1.5.28 1.5.10 1.5.28 1.5.10 1.5.28 1.5.20 1.5.	2	Acenaphthylene	13.766	152.10>126.10	28	0.9770	0.05	119.00	4.65
12	3	Acenaphthene	14.464	153.10>151.10	28	0.9982	0.05	118.92	4.27
14 Nemarthrene	5	Fluorene	16.538	165.10>163.10	28	0.9392		-	-
87 December 1988 Pyreme 1988 2001 1998 2005 1002 3.65 100	12			178.10>152.10	20			-	-
28 Pyrne 28 28 200,10 190,10 30 0,982 0,005 120,002 3,62 200,05 200,05 119,52 5,29	14								
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40 DDT-o,p' 41 DDT-o,p' 42 Endosulfan II (beta isomer) 43 1.378 194.90>160.00 8 0.9947 0.05 117.46 6.58 43 DDD-o,p' 44 DDD-p,p' 45 235.00>165.00 24 0.9896 0.005 118.73 3.51 46 DDD-p,p' 47 Endosulfan sulfate 48 DDT-p,p' 49 271.80>236.00 18 0.9900 0.005 118.32 5.11 48 DDT-p,p' 40 271.80>236.00 18 0.9900 0.005 118.32 5.11 48 DDT-p,p' 40 271.80>236.00 18 0.9900 0.005 113.49 5.26 41 2.40lorobiphenyl (BZ #1) 14.756 188.00>153.10 10 0.9883 0.05 116.54 3.72 42 2.50lorobiphenyl (BZ #18) 19.233 222.00>152.00 25 0.9814 0.05 119.93 3.88 43 2.2°,5-Trichlorobiphenyl (BZ #18) 21.158 178.10>152.10 20 0.9649 0.005 106.63 2.57 42 2.4°,5-Trichlorobiphenyl (BZ #31) 23.242 258.00>151.00 50 0.9878 0.005 119.09 10.83 42 2.2°,5,5'-Tetrachlorobiphenyl (BZ #44) 25.820 289.90>219.90 25 0.9871 0.05 119.87 1.10 42 2.2°,3,5'-Tetrachlorobiphenyl (BZ #60) 27.762 289.90>219.90 25 0.9845 0.005 119.39 4.30 42 2.2',3,5'-Pentachlorobiphenyl (BZ #101) 30.018 327.90>229.90 42 2.2',3,5'-Pentachlorobiphenyl (BZ #101) 30.018 327.90>229.90 43 2.2',3,5'-G-Hexachlorobiphenyl (BZ #151) 31.018 361.90>289.90 44 2.2',3,4,5,5'-Hexachlorobiphenyl (BZ #151) 31.018 361.90>289.90 45 2.2',3,4,4',5,5'-Hexachlorobiphenyl (BZ #166) 33.178 369.0>289.90 46 2.2',3,4,4',5,5'-Hexachlorobiphenyl (BZ #137) 33.821 361.90>289.90 47 30 0.9953 0.005 118.74 5.87 48 2.2',3,4,4',5,6'-Hexachlorobiphenyl (BZ #183) 34.569 395.80>323.80 30 0.9953 0.005 118.04 4.21 49 2.2',3,4,4',5,6'-Hexachlorobiphenyl (BZ #181) 34.787 395.80>323.80 30 0.9953 0.005 118.04 4.21 40 0.9880 0.005 118.04 40 0.9880 0	36	Dieldrin	30.004	188.10>132.10	18	0.9827		115.55	
42 Endosulfan II (beta isomer) 31.378 194.90>160.00 8 0.9947 0.05 117.46 6.58 43 DDD-o,p' 32.026 235.00>165.00 24 0.9896 0.005 118.73 3.51 44 DDD-p,p' 32.128 235.00>165.00 24 0.9908 0.005 118.32 5.11 47 Endosulfan sulfate 33.294 271.80>236.90 18 0.9900 0.005 106.75 8.74 48 DDT-p,p' 33.655 235.00>165.00 24 0.9830 0.005 113.49 5.26 4 2-Chlorobiphenyl (BZ #1) 14.756 188.00>153.10 10 0.9883 0.05 116.54 3.72 8 2,3-Dichlorobiphenyl (BZ #15) 19.233 222.00>152.10 25 0.9814 0.05 119.93 3.88 13 2,2',5-Trichlorobiphenyl (BZ #18) 21.158 178.10>152.10 25 0.9814 0.05 119.93 18.80 13 2,2',5-Trichlorobiphenyl (BZ #31) 23.242 258.00>151.00 50 0.9878 0.005 119.09 10.83 20 2,2',5,5'-Tetrachlorobiphenyl (BZ #44) 25.820 289.90>219.90 25 0.9871 0.05 119.87 1.10 21 2,2',3,5'-Tetrachlorobiphenyl (BZ #60) 32,3',4,4'-Tetrachlorobiphenyl (BZ #60) 32,3',4,5-Pentachlorobiphenyl (BZ #101) 30.018 327.90>292.90 15 0.9929 0.005 103.97 17.04 32,3,3',4,6-Pentachlorobiphenyl (BZ #101) 30.018 327.90>292.90 15 0.9929 0.005 118.74 5.87 42,2',3,5,5'-Hexachlorobiphenyl (BZ #151) 31.018 361.90>289.90 30 0.9892 0.005 118.74 5.87 42,2',3,4,4',5,5'-Hexachlorobiphenyl (BZ #137) 33.821 361.90>289.90 25 0.9845 0.005 118.74 5.87 46 2,2',3,4,4',5,5'-Hexachlorobiphenyl (BZ #153) 32.645 361.90>289.90 30 0.9892 0.005 118.74 5.87 42,2',3,4,4',5,5'-Hexachlorobiphenyl (BZ #137) 33.821 361.90>289.90 25 0.9853 0.005 118.74 5.87 42,2',3,4,4',5,5'-Hexachlorobiphenyl (BZ #137) 33.821 361.90>289.90 25 0.9853 0.005 118.04 4.21 4.22',3,4,4',5,5'-Hexachlorobiphenyl (BZ #137) 33.821 361.90>289.90 25 0.9853 0.005 118.04 4.21 4.22',3,4,4',5,5'-Hexachlorobiphenyl (BZ #137) 34.869 395.80>323.80 30 0.9953 0.005 118.02 318.02 32,3',4,4',5-Heptachlorobiphenyl (BZ #137) 34.6750 358.80>323.80 30 0.9902 0.005 118.22 2.89 2.2',3,4,4',5,6'-Heptachlorobiphenyl (BZ #137) 34.6750 358.80>323.80 30 0.9902 0.005 118.22 2.89	38	DDE-p,p'	30.194	246.00>176.00	30	0.9732	0.005	116.93	4.66
43 DDD-o,p' 32.026 235.00>165.00 24 0.9896 0.005 118.73 3.51 44 DDD-p,p' 32.128 235.00>165.00 24 0.9908 0.005 118.32 5.11 47 Endosulfan sulfate 33.294 271.80>236.90 18 0.9900 0.005 106.75 8.74 48 DDT-p,p' 33.655 235.00>165.00 24 0.9830 0.005 113.49 5.26 42 - Chlorobiphenyl (BZ #1) 14.756 188.00>153.10 10 0.9883 0.05 116.54 3.72 8.23-Dichlorobiphenyl (BZ #18) 19.233 222.00>152.10 25 0.9814 0.05 119.93 3.88 13 2,2',5-Trichlorobiphenyl (BZ #18) 21.158 178.10>152.10 20 0.9649 0.005 106.63 2.57 16 2,4',5-Trichlorobiphenyl (BZ #31) 23.242 258.00>150.00 50 0.9878 0.005 119.87 1.10 12.21 2,2',3,5'-Tetrachlorobiphenyl (BZ #44) 25.820 289.90>219.90 25 0.9871 0.05 119.87 1.10 5.22 82 2,3,4,4'-Tetrachlorobiphenyl (BZ #60) 27.762 289.90>219.90 25 0.9845 0.005 119.39 4.30 32.23,3',4,5-Pentachlorobiphenyl (BZ #101) 30.018 327.90>25.90 25 0.9845 0.005 119.15 2.98 32.23,3',4,5-Pentachlorobiphenyl (BZ #101) 30.018 327.90>25.90 25 0.9865 0.05 119.15 2.98 12.23,3',4,5-Pentachlorobiphenyl (BZ #151) 31.018 361.90>289.90 21.90 0.005 118.74 5.87 2.2',4,5,5'-Hexachlorobiphenyl (BZ #151) 31.018 361.90>289.90 21.90 10.005 118.74 5.87 4.5 2.2',4,4',5,5'-Hexachlorobiphenyl (BZ #153) 32.645 361.90>289.90 21.90 30 0.9896 0.005 118.74 5.87 4.5 2.2',4,4',5,5'-Hexachlorobiphenyl (BZ #137) 33.821 361.90>289.90 25 0.9853 0.005 118.04 4.21 5.87 4.2',3,4',5,5'-Hexachlorobiphenyl (BZ #137) 33.821 361.90>289.90 25 0.9853 0.005 118.04 4.21 5.87 4.2',3,4',5,5'-Hexachlorobiphenyl (BZ #138) 34.569 395.80>32.80 30 0.9953 0.005 118.04 4.21 5.84 5.2',3,4,4',5-Hexachlorobiphenyl (BZ #137) 33.821 361.90>289.90 25 0.9853 0.005 118.04 4.21 5.87 4.2',3,4',5,5'-Hexachlorobiphenyl (BZ #137) 33.821 361.90>289.90 25 0.9853 0.005 118.04 4.21 5.87 5.2',3,4',5,5'-Hexachlorobiphenyl (BZ #138) 34.569 395.80>32.80 30 0.9953 0.005 118.04 4.21 5.42 5.44 5.44 5.54 5.44	40	DDT-o,p'	30.525	235.00>165.00	24	0.9939	0.005	118.30	4.86
44 DDD-p,p' 32.128 235.00>165.00 24 0.9908 0.005 118.32 5.11 47 Endosulfan sulfate 33.294 271.80>236.90 18 0.9900 0.005 106.75 8.74 48 DDT-p,p' 33.655 235.00>165.00 24 0.9830 0.005 113.49 5.26 4 2-Chlorobiphenyl (BZ #1) 14.756 188.00>153.10 10 0.9883 0.05 116.54 3.72 8 2,3-Dichlorobiphenyl (BZ #18) 19.233 222.00>152.10 25 0.9814 0.05 119.93 3.88 13 2,2',5-Frichlorobiphenyl (BZ #18) 21.158 178.10>152.10 20 0.9649 0.005 106.63 2.57 16 2,4',5-Trichlorobiphenyl (BZ #31) 23.242 258.00>151.00 50 0.9878 0.005 119.09 10.83 20 2,2',3,5,5'-Tetrachlorobiphenyl (BZ #44) 25.820 289.90>219.90 25 0.9871 0.05 119.87 1.10 21 2,2',3,4',5-Pentachlorobiphenyl (BZ #97) 28.858 327.90>255.90 25 0.9965 0.05<	42	Endosulfan II (beta isomer)	31.378	194.90>160.00	8	0.9947	0.05	117.46	6.58
47 Endosulfan sulfate 33.294 271.80>236.90 18 0.9900 0.005 106.75 8.74 48 DDT-p,p' 33.655 235.00>165.00 24 0.9830 0.005 113.49 5.26 4 2-Chlorobiphenyl (BZ #1) 14.756 188.00>153.10 10 0.9883 0.05 116.54 3.72 8 2,3-Dichlorobiphenyl (BZ #5) 19.233 222.00>152.10 25 0.9814 0.05 119.93 3.88 13 2,2',5-Trichlorobiphenyl (BZ #18) 21.158 178.10>152.10 20 0.9649 0.005 106.63 2.57 16 2,4',5-Trichlorobiphenyl (BZ #31) 23.242 258.00>151.00 50 0.9878 0.005 119.09 10.83 20 2,2',5,5'-Tetrachlorobiphenyl (BZ #44) 25.820 289.90>219.90 25 0.9871 0.05 119.87 1.10 21 2,2',3,5'-Tetrachlorobiphenyl (BZ #44) 25.820 289.90>219.90 30 0.9778 0.005 119.39 4.30 33 2,2',3',4,5-Pentachlorobiphenyl (BZ #97) 28.858 327.90>255.90 25 0.9845 0.005 119.39 4.30 33 2,2',4,5,5'-Pentachlorobiphenyl (BZ #101) 30.018 327.90>292.90 15 0.9965 0.05 119.15 2.98 37 2,2',4,5,5'-Pentachlorobiphenyl (BZ #101) 30.018 327.90>292.90 15 0.9929 0.005 103.97 17.04 39 2,3,3',4,6-Pentachlorobiphenyl (BZ #109) 30.459 325.90>255.90 30 0.9982 0.005 116.81 2.96 41 2,2',3,5,5'-Hexachlorobiphenyl (BZ #151) 31.018 361.90>289.90 25 0.9648 0.005 118.74 5.87 45 2,2',4,4',5,5'-Hexachlorobiphenyl (BZ #153) 32.645 361.90>289.90 25 0.9863 0.005 118.74 5.87 46 2,2',3,4',5,5'-Hexachlorobiphenyl (BZ #133) 32.645 361.90>289.90 25 0.9863 0.005 118.74 5.87 46 2,2',3,4',5,5'-Hexachlorobiphenyl (BZ #133) 33.2645 361.90>289.90 25 0.9863 0.005 118.74 5.87 47 2,2',3,4',5,5'-Hexachlorobiphenyl (BZ #137) 33.821 361.90>289.90 25 0.9853 0.005 118.04 4.21 50 2,2',3,4',5,5'-Hexachlorobiphenyl (BZ #183) 34.569 395.80>323.80 30 0.9953 0.005 116.71 4.78 51 2,2',3,4',5,5'-Heptachlorobiphenyl (BZ #187) 34.787 395.80>323.80 0 0.9902 0.005 115.42 2.89 54 2,2',3,3',4,5'-Heptachlorobiphenyl (BZ #187) 34.787 395.80>323.80 0 0.9910 0.005 115.42 5.44	43	DDD-o,p'	32.026	235.00>165.00	24	0.9896	0.005	118.73	3.51
48 DDT-p,p¹ 33.655 235.00>165.00 24 0.9830 0.005 113.49 5.26 4 2-Chlorobiphenyl (BZ #1) 14.756 188.00>153.10 10 0.9883 0.05 116.54 3.72 8 2,3-Dichlorobiphenyl (BZ #5) 19.233 222.00>152.10 25 0.9814 0.05 119.93 3.88 13 2,2¹,5-Trichlorobiphenyl (BZ #18) 21.158 178.10>152.10 20 0.9649 0.005 106.63 2.57 16 2,4¹,5-Trichlorobiphenyl (BZ #31) 23.242 258.00>151.00 50 0.9878 0.005 119.09 10.83 20 2,2¹,5,5¹-Tetrachlorobiphenyl (BZ #52) 24.907 289.90>219.90 25 0.9871 0.05 119.87 1.10 21 2,2¹,3,5¹-Tetrachlorobiphenyl (BZ #44) 25.820 289.90>219.90 25 0.9871 0.05 119.39 4.30 33 2,2¹,3¹,4,5-Pentachlorobiphenyl (BZ #60) 27.762 289.90>219.90 25 0.9845 0.005 119.39 4.30 33 2,2¹,3¹,4,5-Pentachlorobiphenyl (BZ #101) 30.018 327.90>292.90 15 0.9965 0.05 119.15 2.98 37 2,2¹,4,5,5¹-Pentachlorobiphenyl (BZ #101) 30.018 327.90>292.90 15 0.9929 0.005 103.97 17.04 39 2,3,3¹,4,6-Pentachlorobiphenyl (BZ #109) 30.459 325.90>255.90 30 0.9892 0.005 116.81 2.96 41 2,2¹,3,5,5¹,6-Hexachlorobiphenyl (BZ #153) 32.645 361.90>289.90 25 0.9648 0.005 118.74 5.87 45 2,2¹,4,4¹,5,5¹-Hexachlorobiphenyl (BZ #137) 33.821 361.90>289.90 25 0.9853 0.005 118.79 2.55 46 2,2¹,3,4',5,5¹-Hexachlorobiphenyl (BZ #137) 33.821 361.90>289.90 25 0.9853 0.005 118.04 4.21 50 2,2¹,3,4,4¹,5-Hexachlorobiphenyl (BZ #183) 34.569 395.80>323.80 30 0.9953 0.005 118.04 4.21 50 2,2¹,3,4',5,5¹-Hexachlorobiphenyl (BZ #183) 34.569 395.80>323.80 30 0.9953 0.005 118.22 2.89 54 2,2¹,3,4',5,5¹-Hetachlorobiphenyl (BZ #187) 34.787 395.80>323.80 30 0.9910 0.005 115.42 5.44	44	DDD-p,p'	32.128	235.00>165.00	24	0.9908	0.005	118.32	5.11
4 2-Chlorobiphenyl (BZ #1) 14.756 188.00>153.10 10 0.9883 0.05 116.54 3.72 8 2,3-Dichlorobiphenyl (BZ #5) 19.233 222.00>152.10 25 0.9814 0.05 119.93 3.88 13 2,2',5-Trichlorobiphenyl (BZ #18) 21.158 178.10>152.10 20 0.9649 0.005 106.63 2.57 16 2,4',5-Trichlorobiphenyl (BZ #31) 23.242 258.00>151.00 50 0.9878 0.005 119.09 10.83 20 2,2',5,5'-Tetrachlorobiphenyl (BZ #52) 24.907 289.90>219.90 25 0.9871 0.05 119.87 1.10 12.2 2,3,3,5'-Tetrachlorobiphenyl (BZ #44) 25.820 289.90>219.90 30 0.9778 0.005 117.11 5.22 28 2,3,4,4'-Tetrachlorobiphenyl (BZ #60) 27.762 289.90>219.90 25 0.9845 0.005 119.39 4.30 33 2,2',3,4,5-Pentachlorobiphenyl (BZ #101) 30.018 327.90>25.90 25 0.9965 0.05 119.15 2.98 37 2,2',4,5,5'-Pentachlorobiphenyl (BZ #101) 30.018 327.90>29.20 15 0.9929 0.005 103.97 17.04 39 2,3,3',4,6-Pentachlorobiphenyl (BZ #101) 30.459 325.90>25.90 25 0.9882 0.005 116.81 2.96 116.81 2.96 12.2',3,5',5',6-Hexachlorobiphenyl (BZ #151) 31.018 361.90>28.99.0 30 0.9918 0.005 118.74 5.87 2,2',4,4',5,5'-Hexachlorobiphenyl (BZ #153) 32.645 361.90>28.99.0 30 0.9918 0.005 118.74 5.87 2,2',4,4',5,5'-Hexachlorobiphenyl (BZ #146) 33.178 359.90>28.99.0 25 0.9853 0.005 118.79 2.55 0.81 49 2,2',3,4',5,5'-Hexachlorobiphenyl (BZ #137) 33.821 361.90>28.99.0 25 0.9853 0.005 118.04 4.21 50 2,2',3,4',5,5'-Hexachlorobiphenyl (BZ #138) 34.569 395.80>323.80 30 0.9992 0.005 118.04 4.21 50 2,2',3,4',5,5'-G-Heptachlorobiphenyl (BZ #188) 34.569 395.80>323.80 30 0.9992 0.005 118.22 2.89 54 2,2',3,4',5,5'-G-Heptachlorobiphenyl (BZ #187) 34.787 395.80>323.80 30 0.9992 0.005 115.42 5.44	47	Endosulfan sulfate	33.294	271.80>236.90	18	0.9900	0.005	106.75	8.74
8 2,3-Dichlorobiphenyl (BZ #5) 19.233 222.00>152.10 25 0.9814 0.05 119.93 3.88 13 2,2',5-Trichlorobiphenyl (BZ #18) 21.158 178.10>152.10 20 0.9649 0.005 106.63 2.57 16 2,4',5-Trichlorobiphenyl (BZ #31) 23.242 258.00>151.00 50 0.9878 0.005 119.09 10.83 20 2,2',5,5'-Tetrachlorobiphenyl (BZ #52) 24.907 289.90>219.90 25 0.9871 0.05 119.87 1.10 119.87 1.10 12.2,2',3,5'-Tetrachlorobiphenyl (BZ #44) 25.820 289.90>219.90 30 0.9778 0.005 117.11 5.22 28 2,3,4,4'-Tetrachlorobiphenyl (BZ #60) 27.762 289.90>219.90 25 0.9845 0.005 119.39 4.30 2,2',3,4,5-Pentachlorobiphenyl (BZ #97) 28.858 327.90>255.90 25 0.9965 0.05 119.15 2.98 37 2,2',4,5,5'-Pentachlorobiphenyl (BZ #101) 30.018 327.90>292.90 15 0.9929 0.005 103.97 17.04 29.3,3',4,6-Pentachlorobiphenyl (BZ #109) 30.459 325.90>255.90 30 0.9892 0.005 116.81 2.96 116.81 2.96 12.2',3,5,5',6-Hexachlorobiphenyl (BZ #151) 31.018 361.90>289.90 30 0.9918 0.005 118.74 5.87 2,2',4,4',5,5'-Hexachlorobiphenyl (BZ #153) 32.645 361.90>289.90 30 0.9896 0.005 118.79 2.55 0.81 49 2,2',3,4',5,5'-Hexachlorobiphenyl (BZ #137) 33.821 361.90>289.90 25 0.9853 0.005 118.04 4.21 50 2,2',3,4',5,5'-Heptachlorobiphenyl (BZ #137) 33.821 361.90>289.90 25 0.9853 0.005 118.04 4.21 50 2,2',3,4',5,5'-Heptachlorobiphenyl (BZ #183) 34.569 395.80>323.80 30 0.9953 0.005 118.22 2.89 54 2,2',3,4',5,5'-Heptachlorobiphenyl (BZ #187) 34.787 395.80>323.80 30 0.9902 0.005 115.42 5.44	48	DDT-p,p'	33.655	235.00>165.00	24	0.9830	0.005	113.49	5.26
13 2,2',5-Trichlorobiphenyl (BZ #18) 21.158 178.10>152.10 20 0.9649 0.005 106.63 2.57 16 2,4',5-Trichlorobiphenyl (BZ #31) 23.242 258.00>151.00 50 0.9878 0.005 119.09 10.83 20 2,2',5,5'-Tetrachlorobiphenyl (BZ #52) 24.907 289.90>219.90 25 0.9871 0.05 119.87 1.10 21 2,2',3,5'-Tetrachlorobiphenyl (BZ #44) 25.820 289.90>219.90 30 0.9778 0.005 117.11 5.22 28 2,3,4,4'-Tetrachlorobiphenyl (BZ #60) 27.762 289.90>219.90 25 0.9845 0.005 119.39 4.30 33 2,2',3',4,5-Pentachlorobiphenyl (BZ #97) 28.858 327.90>255.90 25 0.9965 0.05 119.15 2.98 37 2,2',4,5,5'-Pentachlorobiphenyl (BZ #101) 30.018 327.90>292.90 15 0.9929 0.005 103.97 17.04 39 2,3,3',4,6-Pentachlorobiphenyl (BZ #109) 30.459 325.90>255.90 30 0.9892 0.005 116.81 2.96 41 2,2',3,5,5',6-Hexachlorobiphenyl (BZ #151) 31.018 361.90>289.90 30 0.9918 0.005 118.74 5.87 45 2,2',4,4',5,5'-Hexachlorobiphenyl (BZ #153) 32.645 361.90>289.90 25 0.9648 0.005 118.79 2.55 46 2,2',3,4',5,5'-Hexachlorobiphenyl (BZ #137) 33.821 361.90>289.90 25 0.9853 0.005 118.04 4.21 50 2,2',3,4,4',5-Hexachlorobiphenyl (BZ #183) 34.569 395.80>323.80 30 0.9953 0.005 116.71 4.78 51 2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ #187) 34.787 395.80>323.80 30 0.9910 0.005 115.42 5.44	4	2-Chlorobiphenyl (BZ #1)	14.756	188.00>153.10	10	0.9883	0.05	116.54	3.72
16 2,4',5-Trichlorobiphenyl (BZ #31) 23.242 258.00>151.00 50 0.9878 0.005 119.09 10.83 20 2,2',5,5'-Tetrachlorobiphenyl (BZ #52) 24.907 289.90>219.90 25 0.9871 0.05 119.87 1.10 21 2,2',3,5'-Tetrachlorobiphenyl (BZ #44) 25.820 289.90>219.90 30 0.9778 0.005 117.11 5.22 28 2,3,4,4'-Tetrachlorobiphenyl (BZ #60) 27.762 289.90>219.90 25 0.9845 0.005 119.39 4.30 32,2',3',4,5-Pentachlorobiphenyl (BZ #97) 28.858 327.90>255.90 25 0.9965 0.05 119.15 2.98 37 2,2',4,5,5'-Pentachlorobiphenyl (BZ #101) 30.018 327.90>292.90 15 0.9929 0.005 103.97 17.04 39 2,3,3',4,6-Pentachlorobiphenyl (BZ #109) 30.459 325.90>255.90 30 0.9892 0.005 116.81 2.96 41 2,2',3,5,5',6-Hexachlorobiphenyl (BZ #151) 31.018 361.90>289.90 30 0.9918 0.005 118.74 5.87 45 2,2',4,4',5,5'-Hexachlorobiphenyl (BZ #153) 32.645 361.90>289.90 25 0.9648 0.005 118.79 2.55 46 2,2',3,4',5,5'-Hexachlorobiphenyl (BZ #137) 33.821 361.90>289.90 25 0.9853 0.005 118.04 4.21 50 2,2',3,4,4',5,5'-Hexachlorobiphenyl (BZ #138) 34.569 395.80>323.80 30 0.9953 0.005 118.04 4.21 50 2,2',3,4,4',5,5'-Hexachlorobiphenyl (BZ #187) 34.787 395.80>323.80 30 0.9902 0.005 118.22 2.89 54 2,2',3,4',5,5'-Heptachlorobiphenyl (BZ #170) 36.750 358.80>323.80 0 0.9910 0.005 115.42 5.44	8	2,3-Dichlorobiphenyl (BZ #5)	19.233	222.00>152.10	25	0.9814	0.05	119.93	3.88
20 2,2',5,5'-Tetrachlorobiphenyl (BZ #52) 24.907 289.90>219.90 25 0.9871 0.05 119.87 1.10 21 2,2',3,5'-Tetrachlorobiphenyl (BZ #44) 25.820 289.90>219.90 30 0.9778 0.005 117.11 5.22 28 2,3,4,4'-Tetrachlorobiphenyl (BZ #60) 27.762 289.90>219.90 25 0.9845 0.005 119.39 4.30 33 2,2',3',4,5-Pentachlorobiphenyl (BZ #97) 28.858 327.90>255.90 25 0.9965 0.05 119.15 2.98 37 2,2',4,5,5'-Pentachlorobiphenyl (BZ #101) 30.018 327.90>292.90 15 0.9929 0.005 103.97 17.04 39 2,3,3',4,6-Pentachlorobiphenyl (BZ #109) 30.459 325.90>255.90 30 0.9892 0.005 116.81 2.96 41 2,2',3,5,5',6-Hexachlorobiphenyl (BZ #151) 31.018 361.90>289.90 30 0.9918 0.005 118.74 5.87 45 2,2',4,4',5,5'-Hexachlorobiphenyl (BZ #153) 32.645 361.90>289.90 25 0.9648 0.005 118.79 2.55 46 2,2',3,4',5,5'-Hexachlorobiphenyl (BZ #146) 33.178 359.90>289.90 30 0.9896 0.005 119.53 0.81 49 2,2',3,4,4',5-Hexachlorobiphenyl (BZ #137) 33.821 361.90>289.90 25 0.9853 0.005 118.04 4.21 50 2,2',3,4,4',5-Hexachlorobiphenyl (BZ #183) 34.569 395.80>323.80 30 0.9953 0.005 118.22 2.89 54 2,2',3,4',5,5'-Hetachlorobiphenyl (BZ #187) 34.787 395.80>323.80 30 0.9902 0.005 115.42 5.44	13	2,2',5-Trichlorobiphenyl (BZ #18)	21.158	178.10>152.10	20	0.9649	0.005	106.63	2.57
21 2,2',3,5'-Tetrachlorobiphenyl (BZ #44) 25.820 289.90>219.90 30 0.9778 0.005 117.11 5.22 289.30>219.90 25 0.9845 0.005 119.39 4.30 32,2',3',4,5-Pentachlorobiphenyl (BZ #97) 28.858 327.90>255 0.9845 0.005 119.15 2.98 37 2,2',4,5,5'-Pentachlorobiphenyl (BZ #101) 30.018 327.90>292.90 15 0.9965 0.05 119.15 2.98 39 2,3,3',4,6-Pentachlorobiphenyl (BZ #109) 30.459 325.90>255.90 30 0.9892 0.005 103.97 17.04 39 2,3,3',4,6-Pentachlorobiphenyl (BZ #151) 31.018 361.90>289.90 30 0.9892 0.005 116.81 2.96 41 2,2',3,5,5',6-Hexachlorobiphenyl (BZ #153) 32.645 361.90>289.90 30 0.9918 0.005 118.74 5.87 45 2,2',4,4',5,5'-Hexachlorobiphenyl (BZ #146) 33.178 359.90>289.90 30 0.9896 0.005 119.53 0.81 49 2,2',3,4,4',5-Hexachlorobiphenyl (BZ #137) 33.821 361.90>289.90 25 0.9853 0.005 118.04 4.21 50 2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ #183) 34.569 395.80>323.80 30 0.9902 0.005 118.22 2.89 54 2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ #170) 36.750 358.80>32.80 0 0.9810 0.005 115.42 5.44	16	2,4',5-Trichlorobiphenyl (BZ #31)	23.242	258.00>151.00	50	0.9878	0.005	119.09	10.83
28 2,3,4,4'-Tetrachlorobiphenyl (BZ #60) 27.762 289.90>219.90 25 0.9845 0.005 119.39 4.30 32,2',3',4,5-Pentachlorobiphenyl (BZ #97) 28.858 327.90>255.90 25 0.9965 0.05 119.15 2.98 37 2,2',4,5,5'-Pentachlorobiphenyl (BZ #101) 30.018 327.90>292.90 15 0.9929 0.005 103.97 17.04 39 2,3,3',4,6-Pentachlorobiphenyl (BZ #109) 30.459 325.90>255.90 30 0.9892 0.005 116.81 2.96 41 2,2',3,5,5',6-Hexachlorobiphenyl (BZ #151) 31.018 361.90>289.90 30 0.9918 0.005 118.74 5.87 45 2,2',4,4',5,5'-Hexachlorobiphenyl (BZ #153) 32.645 361.90>289.90 25 0.9648 0.005 118.79 2.55 46 2,2',3,4',5,5'-Hexachlorobiphenyl (BZ #146) 33.178 359.90>289.90 30 0.9896 0.005 119.53 0.81 49 2,2',3,4,4',5-Hexachlorobiphenyl (BZ #137) 33.821 361.90>289.90 25 0.9853 0.005 118.04 4.21 50 2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ #183) 34.569 395.80>323.80 30 0.9953 0.005 118.22 2.89 54 2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ #187) 34.787 395.80>323.80 0 0.9810 0.005 115.42 5.44	20	2,2',5,5'-Tetrachlorobiphenyl (BZ #52)	24.907	289.90>219.90	25	0.9871	0.05	119.87	1.10
33 2,2',3',4,5-Pentachlorobiphenyl (BZ #97) 28.858 327.90>255.90 25 0.9965 0.05 119.15 2.98 37 2,2',4,5,5'-Pentachlorobiphenyl (BZ #101) 30.018 327.90>292.90 15 0.9929 0.005 103.97 17.04 39 2,3,3',4,6-Pentachlorobiphenyl (BZ #109) 30.459 325.90>255.90 30 0.9892 0.005 116.81 2.96 41 2,2',3,5,5',6-Hexachlorobiphenyl (BZ #151) 31.018 361.90>289.90 30 0.9918 0.005 118.74 5.87 45 2,2',4,4',5,5'-Hexachlorobiphenyl (BZ #153) 32.645 361.90>289.90 25 0.9648 0.005 118.79 2.55 46 2,2',3,4',5,5'-Hexachlorobiphenyl (BZ #146) 33.178 359.90>289.90 30 0.9896 0.005 119.53 0.81 49 2,2',3,4,4',5-Hexachlorobiphenyl (BZ #137) 33.821 361.90>289.90 25 0.9853 0.005 118.04 4.21 50 2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ #183) 34.569 395.80>323.80 30 0.9953 0.005 116.71 4.78 51 2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ #187) 34.787 395.80>323.80 30 0.9902 0.005 115.42 5.44	21	2,2',3,5'-Tetrachlorobiphenyl (BZ #44)	25.820	289.90>219.90	30	0.9778	0.005	117.11	5.22
37 2,2',4,5,5'-Pentachlorobiphenyl (BZ #101) 30.018 327.90>292.90 15 0.9929 0.005 103.97 17.04 39 2,3,3',4,6-Pentachlorobiphenyl (BZ #109) 30.459 325.90>255.90 30 0.9892 0.005 116.81 2.96 41 2,2',3,5,5',6-Hexachlorobiphenyl (BZ #151) 31.018 361.90>289.90 30 0.9918 0.005 118.74 5.87 45 2,2',4,4',5,5'-Hexachlorobiphenyl (BZ #153) 32.645 361.90>289.90 25 0.9648 0.005 118.79 2.55 46 2,2',3,4',5,5'-Hexachlorobiphenyl (BZ #146) 33.178 359.90>289.90 30 0.9896 0.005 119.53 0.81 49 2,2',3,4,4',5-Hexachlorobiphenyl (BZ #137) 33.821 361.90>289.90 25 0.9853 0.005 118.04 4.21 50 2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ #183) 34.569 395.80>323.80 30 0.9953 0.005 116.71 4.78 51 2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ #187) 34.787 395.80>323.80 30 0.9902 0.005 118.22 2.89 54 2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ #170) 36.750 358.80>323.80 0 0.9810 0.005 115.42 5.44	28	2,3,4,4'-Tetrachlorobiphenyl (BZ #60)	27.762	289.90>219.90	25	0.9845	0.005	119.39	4.30
39 2,3,3',4,6-Pentachlorobiphenyl (BZ #109) 30.459 325.90>255.90 30 0.9892 0.005 116.81 2.96 41 2,2',3,5,5',6-Hexachlorobiphenyl (BZ #151) 31.018 361.90>289.90 30 0.9918 0.005 118.74 5.87 45 2,2',4,4',5,5'-Hexachlorobiphenyl (BZ #153) 32.645 361.90>289.90 25 0.9648 0.005 118.79 2.55 46 2,2',3,4',5,5'-Hexachlorobiphenyl (BZ #146) 33.178 359.90>289.90 30 0.9896 0.005 119.53 0.81 49 2,2',3,4,4',5-Hexachlorobiphenyl (BZ #137) 33.821 361.90>289.90 25 0.9853 0.005 118.04 4.21 50 2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ #183) 34.569 395.80>323.80 30 0.9953 0.005 116.71 4.78 51 2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ #187) 34.787 395.80>323.80 30 0.9902 0.005 118.22 2.89 54 2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ #170) 36.750 358.80>323.80 0 0.9810 0.005 115.42 5.44	33	2,2',3',4,5-Pentachlorobiphenyl (BZ #97)	28.858	327.90>255.90	25	0.9965	0.05	119.15	2.98
41 2,2',3,5,5',6-Hexachlorobiphenyl (BZ #151) 31.018 361.90>289.90 30 0.9918 0.005 118.74 5.87 45 2,2',4,4',5,5'-Hexachlorobiphenyl (BZ #153) 32.645 361.90>289.90 25 0.9648 0.005 118.79 2.55 46 2,2',3,4',5,5'-Hexachlorobiphenyl (BZ #146) 33.178 359.90>289.90 30 0.9896 0.005 119.53 0.81 49 2,2',3,4,4',5-Hexachlorobiphenyl (BZ #137) 33.821 361.90>289.90 25 0.9853 0.005 118.04 4.21 50 2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ #183) 34.569 395.80>323.80 30 0.9953 0.005 116.71 4.78 51 2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ #187) 34.787 395.80>323.80 30 0.9902 0.005 118.22 2.89 54 2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ #170) 36.750 358.80>323.80 0 0.9810 0.005 115.42 5.44	37	2,2',4,5,5'-Pentachlorobiphenyl (BZ #101)	30.018	327.90>292.90	15	0.9929	0.005	103.97	17.04
45 2,2',4,4',5,5'-Hexachlorobiphenyl (BZ #153) 32.645 361.90>289.90 25 0.9648 0.005 118.79 2.55 46 2,2',3,4',5,5'-Hexachlorobiphenyl (BZ #146) 33.178 359.90>289.90 30 0.9896 0.005 119.53 0.81 49 2,2',3,4,4',5-Hexachlorobiphenyl (BZ #137) 33.821 361.90>289.90 25 0.9853 0.005 118.04 4.21 50 2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ #183) 34.569 395.80>323.80 30 0.9953 0.005 116.71 4.78 51 2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ #187) 34.787 395.80>323.80 30 0.9902 0.005 118.22 2.89 54 2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ #170) 36.750 358.80>323.80 0 0.9810 0.005 115.42 5.44	39	2,3,3',4,6-Pentachlorobiphenyl (BZ #109)	30.459	325.90>255.90	30	0.9892	0.005	116.81	2.96
46 2,2',3,4',5,5'-Hexachlorobiphenyl (BZ #146) 33.178 359.90>289.90 30 0.9896 0.005 119.53 0.81 49 2,2',3,4,4',5-Hexachlorobiphenyl (BZ #137) 33.821 361.90>289.90 25 0.9853 0.005 118.04 4.21 50 2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ #183) 34.569 395.80>323.80 30 0.9953 0.005 116.71 4.78 51 2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ #187) 34.787 395.80>323.80 30 0.9902 0.005 118.22 2.89 54 2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ #170) 36.750 358.80>323.80 0 0.9810 0.005 115.42 5.44	41	2,2',3,5,5',6-Hexachlorobiphenyl (BZ #151)	31.018	361.90>289.90	30	0.9918	0.005	118.74	5.87
49 2,2',3,4,4',5-Hexachlorobiphenyl (BZ #137) 33.821 361.90>289.90 25 0.9853 0.005 118.04 4.21 50 2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ #183) 34.569 395.80>323.80 30 0.9953 0.005 116.71 4.78 51 2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ #187) 34.787 395.80>323.80 30 0.9902 0.005 118.22 2.89 54 2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ #170) 36.750 358.80>323.80 0 0.9810 0.005 115.42 5.44	45	2,2',4,4',5,5'-Hexachlorobiphenyl (BZ #153)	32.645	361.90>289.90	25	0.9648	0.005	118.79	2.55
50 2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ #183) 34.569 395.80>323.80 30 0.9953 0.005 116.71 4.78 51 2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ #187) 34.787 395.80>323.80 30 0.9902 0.005 118.22 2.89 54 2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ #170) 36.750 358.80>323.80 0 0.9810 0.005 115.42 5.44	46	2,2',3,4',5,5'-Hexachlorobiphenyl (BZ #146)	33.178	359.90>289.90	30	0.9896	0.005	119.53	0.81
51 2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ #187) 34.787 395.80>323.80 30 0.9902 0.005 118.22 2.89 54 2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ #170) 36.750 358.80>323.80 0 0.9810 0.005 115.42 5.44	49	2,2',3,4,4',5-Hexachlorobiphenyl (BZ #137)	33.821	361.90>289.90	25	0.9853	0.005	118.04	4.21
54 2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ #170) 36.750 358.80>323.80 0 0.9810 0.005 115.42 5.44	50	2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ #183)	34.569	395.80>323.80	30	0.9953	0.005	116.71	4.78
	51	2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ #187)	34.787	395.80>323.80	30	0.9902	0.005	118.22	2.89
57 2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ #206) 42.190 463.80>393.70 30 0.9891 0.005 116.37 2.38	54	2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ #170)	36.750	358.80>323.80	0	0.9810	0.005	115.42	5.44
	57	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ #206)	42.190	463.80>393.70	30	0.9891	0.005	116.37	2.38

* Analytes incurred in large amounts in blanks sample, hence LOQ could not be determined

PAH Pesticides PCB



Figure 2. Shimadzu GCMS-TQ8040 NX

4. Results

Validation parameters like linearity, recovery and precision were studied to establish LOQs. The summary results are shown in Table 2.

4-1. Linearity

For linearity study and quantifying spiked samples, matrix matched calibration standards were used. Multilevel calibration curve included 0.025, 0.05, 0.1, 0.25, 0.5, 1, 2.5 and 5 ppb concentration levels. All calibration standards were found within 80 to 120% accuracy range which is well within the criteria mentioned in SANTE guidelines.

4-2. Recovery

Recovery was evaluated by quantifying spiked samples at 0.005 and 0.05 ppb (six spiked samples at each level) against matrix matched linearity. Mean recoveries were found to be within 80-120% at LOQ level (Refer Table 2). As mentioned previously, spiked samples were concentrated 10 times, so that final concentrations were comparable to 0.05 and 0.5 ppb of linearity runs.

4-3. Reproducibility (% RSD_R)

Reproducibility experiment for recoveries was performed on 6 different spiked samples at 0.005, and 0.05 ppb concentration levels. The % RSD of 6 spiked samples at their respective LOQ level was found to be less than 20 % (Refer Table 2).

4-4. LOQ

Out of 61 contaminants analyzed, this method successfully achieved LOQs of 0.005 and 0.05 for 36 and 22 contaminants, respectively. Remaining three contaminants all from PAH category were incurred in high amounts in blanks, hence their validation parameters could not be studied. List of LOQs of individual contaminants is shown in Table 2.

5. Conclusion

- > This study shows that the extraction of reduced sample quantity combined with LVI of 50 μL on GC-MS/MS system is a reliable and efficient tool to simultaneously quantify residual contaminants like PAH, PCB and pesticides in drinking water sample.
- ➤ Also, highly sensitive Shimadzu GC-MS/MS allows trace level detection even without the need of evaporating & concentrating the sample. This helps in ruggedness resulting in reproducible detection of analytes.
- ➤ The combination of sensitive instrument and reproducible extraction method enables its use in testing laboratories for residual contaminants analysis in drinking water.

6. References

1. Guidance document on analytical quality control and method validation procedures for pesticide residues and analysis in food and feed. SANTE/11312/2021