Organochlorine Pesticides and PCBs by GC
EPA 608

Facility Name:_____VELAP ID_____

Assessor Name:_____Analyst Name:_____Inspection Date_____

Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Records Examined: SOP Number/ Revision/ Date _____ Analyst:_____ Sample ID: _____ Date of Sample Preparation:_____ Date of Analysis:_____					
Was granular sodium sulfate heated at 400°C for four hours in a shallow tray prior to use?	6.7				
Was Florisil PR grade (60/100 mesh), purchased as "activated at 1250°F", stored in the dark, and activated prior to use at 130°C for at least 16 hours?	6.8				
Was triple distilled mercury used?	6.9				
Was activated copper powder used?	6.10				
Were standards stored at 4°C protected from light?	6.11.2				
Were a minimum of three concentration levels used as calibration standards?	7.2.1 7.3.1				
Was one of the calibration standards near, but above, the MDL?	7.2.1 7.3.1				
When internal standards were used, were response factors (RFs) calculated for each compound, and only if RSD was <10%, was an average RF used for calculations? (Alternatively, results can be used to plot a calibration curve of response ratios.)	7.3.2				
Were one or more calibration standards verified each day to be within ±15% of the predicted responses? (Alternatively, a fresh calibration can be performed.)	7.4				
Before using any cleanup procedure, was a series of calibration standards processed through the procedure, to validate elution patterns and the absence of interferences from the reagents?	7.6				
Notes/Comments:					

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For INITIAL DEMONSTRATION OF CAPABILITY (IDC), was a QC check containing each analyte of interest at the specified concentrations (per Table 3) in acetone obtained from the EPA and analyzed to demonstrate analyst accuracy and precision?	8.1, 8.2.1, 8.2.2, Table 3				
For the IDC, were four replicates of the QC check done with average recoveries and standard deviations calculated?	8.2.4				
For the IDC, did all average recoveries for analytes fall within acceptance criteria from Table 3?	8.2.5, Table 3				
Were 10% of the samples from each sample site spiked? (For labs that analyze 1-10 samples per month, at least one spiked sample per month is required.)	8.3				
Were spike recovery criteria defined per Table 3 or 4?	8.3.3				
If spikes failed, were QC check standards containing the failed parameters prepared and analyzed to pass Table 3?	8.4				
Were records of wastewater spike recovery data updated on a regular basis (i.e., after 5-10 new measurements)? These records include average percent recovery and standard deviation of the percent recovery.	8.5				
Were samples collected in glass containers?	9.1				
Were all samples iced at 4°C from collection until extraction?	9.2				
Were samples that were not extracted within 72 hours adjusted to a pH within 5.0-9.0 using sulfuric acid and/or sodium hydroxide?	9.2				
If aldrin was to be measured, were samples checked for residual chlorine and dechlorinated with sodium thiosulfate?	9.2				
Were samples extracted within seven days of collection and completely analyzed within 40 days of extraction?	9.3				

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Were the menisci on the sample containers marked for later determination of sample volume, and the samples poured into 2 liter separatory funnels?	10.1				
Were sample volumes recorded to the nearest 5 mL?	10.9				
Were sample bottles rinsed with 60 mL of methylene chloride, and the methylene chloride added to the separatory funnels?	10.2				
Were the separatory funnels shaken for 2 minutes, and were the phases allowed to separate for at least 10 minutes?	10.2				
If emulsions formed that were more than one-third the volume of the solvent layer, were mechanical techniques employed to complete the phase separation?	10.2				
Were the methylene chloride layers from the extraction collected in 250 mL erlenmeyer flasks?	10.2				
Were the above three steps repeated twice more?	10.3				
Were the combined extracts filtered through drying columns containing sodium sulfate, and were the flasks rinsed with 20-30 mL of methylene chloride?	10.5				
Were extracts concentrated to 1 mL in a K-D concentrator at 60-65°C?	10.6				
Was 50 mL of hexane added to the concentrated extract, and the extract concentrated further at 80°C for 5-10 minutes?	10.7				
Were extracts transferred to Teflon-sealed screw cap vials if to be stored for longer than two days?	10.8				
Cleanup and Separation- *Cleanup may not be necessary for a relatively clean sample matrix (11.1)*					
Was Florisil placed into a chromatographic column with 1-2 cm of anhydrous sodium sulfate on top?	11.2.1				
After transferring sample extract into the column, was the extract container rinsed twice with 1-2 mL of hexane?	11.2.3				

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Were columns rinsed with 200 mL of 6% ethyl ether in hexane, 200 mL of 15% ethyl ether in hexane, and then 200 mL of 50% ethyl ether in hexane?	11.2.4				
Were the cleaned up samples concentrated as before prior to analysis, with the temperature at 85°C?	11.2.5				
To prevent sulfur interference, were samples cleaned up with 1-3 drops of mercury or with activated copper powder?	11.3				
GC analysis					
Was the system calibrated daily?	12.2				
If internal standards were used, were the internal standards added to sample extracts and mixed thoroughly immediately before injection?	12.3				
Did the laboratory record the injected volume to the nearest 0.05 µL, the total extract volume, and the resulting peak size in area or peak height units?	12.4				

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Table 3—QC Acceptance Criteria—Method 608

Parameter	Test conc. (µg/L)	Limit for s (µg/L)	Range for $\bar{X}$ (µg/L)	Range for P, P_s %)
Aldrin	2.0	0.42	1.08 - 2.24	42 - 122
α-BHC	2.0	0.48	0.98 - 2.44	37 - 134
β-BHC	2.0	0.64	0.78 - 2.60	17- 147
δ-BHC	2.0	0.72	1.01 - 2.37	19 - 140
γ-BHC	2.0	0.46	0.86 - 2.32	32 - 127
Chlordane	50	10.0	27.6 - 54.3	45 - 119
4,4'-DDD	10	2.8	4.8 - 12.6	31 - 141
4,4'-DDE	2.0	0.55	1.08 - 2.60	30 - 145
4,4'-DDT	10	3.6	4.6 - 13.7	25 - 160
Dieldrin	2.0	0.76	1.15 - 2.49	36 - 146
Endosulfan I	2.0	0.49	1.14 - 2.82	45 - 153
Endosulfan II	10	6.1	2.2 - 17.1	D - 202
Endosulfan Sulfate	10	2.7	3.8 - 13.2	26 - 144
Endrin	10	3.7	5.1 - 12.6	30 - 147
Heptachlor	2.0	0.40	0.86 - 2.00	34 - 111
Heptachlor epoxide	2.0	0.41	1.13 - 2.63	37 - 142
Toxaphene	50.0	12.7	27.8 - 55.6	41 - 126
PCB-1016	50	10.0	30.5 - 51.5	50 - 114
PCB-1221	50	24.4	22.1 - 75.2	15 - 178
PCB-1232	50	17.9	14.0 - 98.5	10 - 215
PCB-1242	50	12.2	24.8 - 69.6	39 - 150
PCB-1248	50	15.9	29.0 - 70.2	38 - 158
PCB-1254	50	13.8	22.2 - 57.9	29 - 131
PCB-1260	50	10.4	18.7 - 54.9	8 - 127

s = Standard deviation of four recovery measurements, in µg/L (Section 8.2.4).
$\bar{X}$ = Average recovery for four recovery measurements, in µg/L (Section 8.2.4).
P, P_s = Percent recovery measured (Section 8.3.2, Section 8.4.2).
D = Detected; result must be greater than zero.

NOTE: These criteria are based directly upon the method performance data in Table 4. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 4.

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Table 4—Method Accuracy and Precision as Functions of Concentration—Method 608

Parameter	Accuracy, as recovery, X′ (µg/L)	Single analyst precision, s_r' (µg/L)	Overall precision, S′ (µg/L)
Aldrin	0.81C+0.04	0.16$\bar{X}$ - 0.04	0.20$\bar{X}$ - 0.01
α-BHC	0.84C+0.03	0.13$\bar{X}$+0.04	0.23$\bar{X}$ - 0.00
β-BHC	0.81C+0.07	0.22$\bar{X}$ - 0.02	0.33$\bar{X}$ - 0.05
δ-BHC	0.81C+0.07	0.18$\bar{X}$+0.09	0.25$\bar{X}$+0.03
γ-BHC	0.82C - 0.05	0.12$\bar{X}$+0.06	0.22$\bar{X}$+0.04
Chlordane	0.82C - 0.04	0.13$\bar{X}$+0.13	0.18$\bar{X}$+0.18
4,4'-DDD	0.84C+0.30	0.20$\bar{X}$ - 0.18	0.27$\bar{X}$ - 0.14
4,4'-DDE	0.85C+0.14	0.13$\bar{X}$+0.06	0.28$\bar{X}$ - 0.09
4,4'-DDT	0.93C - 0.13	0.17$\bar{X}$+0.39	0.31$\bar{X}$ - 0.21
Dieldrin	0.90C+0.02	0.12$\bar{X}$+0.19	0.16$\bar{X}$+0.16
Endosulfan I	0.97C+0.04	0.10$\bar{X}$+0.07	0.18$\bar{X}$+0.08
Endosulfan II	0.93C+0.34	0.41$\bar{X}$ - 0.65	0.47$\bar{X}$ - 0.20
Endosulfan Sulfate	0.89C - 0.37	0.13$\bar{X}$+0.33	0.24$\bar{X}$+0.35
Endrin	0.89C - 0.04	0.20$\bar{X}$+0.25	0.24$\bar{X}$+0.25
Heptachlor	0.69C+0.04	0.06$\bar{X}$+0.13	0.16$\bar{X}$+0.08
Heptachlor epoxide	0.89C+0.10	0.18$\bar{X}$ - 0.11	0.25$\bar{X}$ - 0.08
Toxaphene	0.80C+1.74	0.09$\bar{X}$+3.20	0.20$\bar{X}$+0.22
PCB-1016	0.81C+0.50	0.13$\bar{X}$+0.15	0.15$\bar{X}$+0.45
PCB-1221	0.96C+0.65	0.29$\bar{X}$ - 0.76	0.35$\bar{X}$ - 0.62
PCB-1232	0.91C+10.79	0.21$\bar{X}$ - 1.93	0.31$\bar{X}$+3.50
PCB-1242	0.93C+0.70	0.11$\bar{X}$+1.40	0.21$\bar{X}$+1.52
PCB-1248	0.97C+1.06	0.17$\bar{X}$+0.41	0.25$\bar{X}$ - 0.37
PCB-1254	0.76C+2.07	0.15$\bar{X}$+1.66	0.17$\bar{X}$+3.62
PCB-1260	0.66C+3.76	0.22$\bar{X}$ - 2.37	0.39$\bar{X}$ - 4.86

X′ = Expected recovery for one or more measurements of a sample containing a concentration of C, in µg/L.
s_r' = Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in µg/L.
S′ = Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in µg/L.
C = True value for the concentration, in µg/L.
$\bar{X}$ = Average recovery found for measurements of samples containing a concentration of C, in µg/L.

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