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New Organochlorine Pesticide Water Test Methods Improve Data Quality, Sampling Efficiency and Manual Handling

Introduction

ALS Environmental has made significant advances in sample size reduction, leading the industry towards safer and more sustainable practices. Lower water sample volume requirements can significantly reduce sampling times; and smaller, lighter coolers reduce the risk of strain injuries in the field.

The benefits continue in the laboratory, where the environmental footprint from testing is reduced with lower chemical, consumable and energy requirements. In many cases the research and development that has resulted in lower volume requirements has also led to the additional benefit of improved test method performance.

The implementation of a NEW low-level Organochlorine Pesticide (OCP) and Polychlorinated Biphenyls (PCB) method that allows a reduced sample size is no exception; and the data quality benefits are significant for this accredited[a] method, now offered by ALS Waterloo, Canada.

General Quality Implications

The required detection levels for OCP in water for various regulations in Canada are quite low. To achieve these levels by either GC/MS or GC/ECD, sensitive instrumentation and sample extracts free from interfering compounds are essential. ALS has focused on both of these aspects, working to overcome the sensitivity limitations of GC/MS through optimization of new technology, and modifying sample preparation methodologies to virtually eliminate background interference and carryover risk.

The end result is data that meet stringent detection limit criteria, while at the same time significantly reducing the risk of false positives when analyzing a range of ultra-clean and dirty samples – a challenge for any lab.

What This Means For ALS Clients

Ontario Reg. 153/04, BC Contaminated Sites Regulation (CSR), Alberta Tier 1 (ABT1), and CCME each have specific criteria for Organochlorine Pesticides. ALS Canada has developed a Routine Method that meets O.Reg. 153/04 and the lowest BC CSR criteria with a 100 mL sample size.

To meet the lowest ABT1 and CCME levels, a Trace Method using a 500 mL sample is required. The table on Page 2 shows reporting limits, applicable regulatory standards, and current method performance for the 100 mL Routine Method at or near the detection limit.

In addition, the reporting limits for the 500 mL Trace Method are provided, along with associated regulatory limits that require this method. The Limits of Reporting (LORs) of the Routine and Trace methods meet or surpass routine LORs previously offered by ALS Canada, and require less sample volume.

Routine vs. Trace

The Trace Method may be selected to obtain ultra-low level detection limits, if required to meet the most stringent criteria, or to measure concentrations or monitor trends below regulatory limits.

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OCP Reporting Limits, Applicable Regulatory Standards, and Trace Method Performance

Parameter	Routine (100 mL) LOR ug/L	Recovery at LOR %	Precision at LOR %RSD	O.Reg. 153/04 RDL ug/L	BC CSR Lowest Standard ug/L	Trace (500 mL) LOR ug/L	ABT1 Lowest Guideline ug/L	CCME Lowest Guideline ug/L
aldrin	0.008	110	6.1	0.01	0.009	0.002	NVI	NV
aldrin + dieldrin	0.012	-	-	NV	0.04	0.003	0.7	NV
BHC, alpha-	0.008	110	4.0	NV	0.025	0.005	NV	NV
BHC, beta-	0.008	90	6.1	NV	0.085	0.005	NV	NV
BHC, delta-	0.008	105	5.0	NV	NV	0.005	NV	NV
BHC, gamma- (lindane)	0.008	115	4.8	0.01	0.1	0.005	0.01	0.01
chlordane, alpha-	0.008	117	4.1	NVI	NVI	0.002	NV	NV
chlordane, gamma-	0.008	124	3.2	NVI	NVI	0.002	NV	NV
chlordane, alpha- + gamma-	0.012	-	-	0.06	0.06	0.003	NV	NV
dieldrin	0.008	127	4.9	0.05	0.01	0.002	NVI	NV
endosulfan, alpha-	0.007	115	5.8	NVI	NVI	0.001	NVI	NVI
endosulfan, beta-	0.007	100	8.3	NVI	NVI	0.001	NVI	NVI
endosulfan, alpha- + beta-	0.01	-	-	0.05	0.01	0.0015	0.0031	0.002
endosulfan sulfate	0.008	130	7.3	NV	NV	0.002	NV	NV
endrin	0.01	110	9.4	0.05	0.023	0.005	2.8	NV
endrin aldehyde	0.01	103	10.7	NV	NV	0.005	NV	NV
endrin ketone	0.01	137	8.8	NV	NV	0.005	NV	NV
heptachlor	0.008	109	3.8	0.01	0.035	0.002	NV	NV
heptachlor epoxide	0.008	119	4.0	0.01	0.015	0.002	0.052	NV
hexachlorobenzene	0.008	106	4.9	0.01	0.1	0.002	0.52	NV
hexachlorobutadiene	0.008	86	5.3	0.01	2	0.002	1.3	NV
hexachloroethane	0.008	91	6.2	0.01	3	0.002	NV	NV
methoxychlor	0.008	111	4.5	0.05	20	0.002	0.17	NV
mirex	0.008	130	2.8	NV	0.0085	0.002	NV	NV
DDD, o,p'-	0.004	117	4.1	NVI	NVI	0.0004	NVI	NV
DDD, p,p'-	0.004	106	4.9	NVI	NVI	0.0004	NVI	NV
DDD, total	0.006	-	-	0.05	NVI	NV	NVI	NV
DDE, o,p'-	0.004	116	4.0	NVI	NVI	0.0004	NVI	NV
DDE, p,p'-	0.004	113	3.1	NVI	NVI	0.0004	NVI	NV
DDE, total	0.006	-	-	0.01	NVI	NV	NVI	NV
DDT, o,p'-	0.004	109	5.2	NVI	NVI	0.0004	NVI	NV
DDT, p,p'-	0.004	103	4.7	NVI	NVI	0.0004	NVI	NV
DDT, total	0.006	-	-	0.05	NVI	NV	NVI	NV
DDT and metabolites[1]	0.01	-	-	NV	0.01	NV	93	NV
Average Precision and Accuracy:		**111**	**5.4**					

NV = no value applied
NVI = no value for individual isomer(s), standard/guideline applies to sum
LOR = limit of reporting
RSD = relative standard deviation
RDL = maximum reporting detection limit

[1] DDT and metabolites is the sum of all isomers of DDD, DDE, and DDT.

Recovery and precision values shown reflect method performance at the LOR for the ALS Routine Method, from initial validation results.
These performance measures generally improve at higher concentrations.

Precision and Accuracy Implications

In addition to other benefits, these new methods have resulted in improvements to data quality for both precision and accuracy, which means any trending of pesticide positives will be more accurate, with less 'noise.' The previous OCP method (using 500 mL samples) yielded average precision of 12.5% RSD at 0.010 ug/L. The new Routine OCP Method (using 100 mL samples) shows notable improvement, with average precision of 5.4% RSD at 0.010 ug/L.

This method is also validated and accredited[(a)] for PCB Aroclors with similar improvements in performance. The Routine method easily meets any of the indicated regulatory requirements for Aroclors in water. The table below shows the 100 mL Routine Method performance at the Limit of Reporting.

Parameter	Routine (100 mL) LOR ug/L	Recovery at LOR %	Precision at LOR %RSD	O.Reg. 153/04 RDL* ug/L	BC CSR Lowest Standard ug/L	ABT1 Lowest Guideline ug/L	CCME Lowest Guideline ug/L
Aroclor 1242	0.02	95	3.2	NV	NV	NV	NV
Aroclor 1254	0.02	99	3.4	NV	NV	NV	NV
Aroclor 1260	0.02	117	2.9	NV	NV	NV	NV
Aroclor 1248	0.02			NV	NV	NV	NV
Total PCBs	0.04	104	3.2	0.2	NV	9.4	NV

* RDL = maximum permitted Reporting Detection Limit

Performance of 100 mL Routine Method

This method performance not only allows for lower sample volumes for PCB analysis, but also improves the precision and accuracy of the test, enhancing overall confidence in results.

Sampling Considerations and Sample Containers

When testing to trace levels, exclusion of sediment in the sampling procedure is particularly important. Sediment may provide high bias to water sample results when testing for hydrophobic compounds such as OCPs and PCBs. Groundwater samples should be taken using low-flow techniques to minimize disturbance which can result in uptake of sediment into the sample. Samples may not be filtered for OCP or PCB, as the filtering step will potentially result in low bias through adsorption.

These new ALS methods should only be applied using ALS proofed and validated 100 mL or 500 mL amber glass bottles.

In order to meet applicable criteria, the test method required (Routine or Trace) should be indicated when ordering sample bottles.

[(a)] These new methods are accredited to the ISO 17025:2017 standard at ALS Waterloo by the Canadian Association for Laboratory Accreditation (CALA). Refer to the ALS Waterloo Scope of Accreditation for complete and current details.