



# EnviroMail 19 *Canada - December 2019*

## Trace GC/MS/MS 100 mL OC Pesticide Method Raises the Bar for Data Quality

### Introduction

ALS Environmental Canada is introducing a new Organochlorine Pesticide (OCP) test method for waters, using state-of-the-art GC/MS/MS triple quadrupole instrumentation to achieve trace level detection limits with near-zero chance of false positives, using 100 mL sample bottles. This new method raises the bar for data quality over traditional test methods that use either single quadrupole GC/MS or GC/ECD (Electron Capture Detection).

The unmatched sensitivity of our optimized GC/MS/MS triple quad method easily meets the lowest Canadian standards and guidelines for OCPs in water without the need for large 500 mL or 1 L sample volumes, which further advances the ALS commitment to sample size reduction to support faster and easier sample collection in the field with less potential for strain injuries to samplers.

ALS announced data quality and sample size reduction improvements to our OCP water methods in June 2019 with [EnviroMail 14](#), when we introduced our OCP method for routine Limits of Reporting (LORs) using 100 mL sample bottles by traditional single quadrupole GC/MS. Our routine single quad method remains suitable for most common regulatory requirements and for most water samples, but our new GC/MS/MS method achieves detection limits up to 10 times lower, meeting the lowest of Canadian water quality standards and guidelines using the same 100 mL sample size as our routine method, while introducing significant new data quality benefits.

### What is Triple Quad GC/MS/MS?

Both GC/MS and GC/MS/MS use gas chromatography to separate organic substances in time using a capillary chromatographic column interfaced to a mass spectrometric (MS) detector. Both techniques use an electron beam to ionize and fragment organic molecules as they elute from the GC column, with detection of mass-specific molecular fragments. But with GC/MS/MS, a technique called *Multiple Reaction*

*Monitoring* (MRM) provides far greater selectivity and sensitivity. With MRM, a precursor ion is isolated in the first stage of the MS, which is then further fragmented in a collision cell (2<sup>nd</sup> stage), with detection of unique “daughter” ions in the third stage of the spectrometer. GC/MS/MS eliminates most interferences that can occur with GC/MS because it only detects substances that can be ionized to produce the correct precursor ion, *and* where that precursor ion can be further fragmented to the correct secondary ion. Detection of these highly unique MRM “transitions” virtually guarantees the correct identification and accurate quantitation of each targeted analyte.

Parameter	Trace (100 mL) LOR ug/L	O.Reg. 153/04 RDL ug/L	BC CSR Lowest Standard ug/L	ABT1 Lowest Guideline ug/L	CCME Lowest Guideline ug/L
aldrin	0.002	0.01	0.009	NVI	NV
aldrin + dieldrin	0.003	NV	0.04	0.7	NV
BHC, alpha-	0.005	NV	0.025	NV	NV
BHC, beta-	0.005	NV	0.085	NV	NV
BHC, delta-	0.005	NV	NV	NV	NV
BHC, gamma- (lindane)	0.005	0.01	0.1	0.01	0.01
chlordane, alpha- + gamma-dieldrin	0.003	0.06	0.06	NV	NV
endosulfan, alpha- + beta-endosulfan sulfate	0.0015	0.05	0.01	0.0031	0.002
endrin	0.005	0.05	0.023	2.8	NV
endrin aldehyde	0.005	NV	NV	NV	NV
endrin ketone	0.005	NV	NV	NV	NV
heptachlor	0.002	0.01	0.035	NV	NV
heptachlor epoxide	0.002	0.01	0.01	0.052	NV
hexachlorobenzene	0.002	0.01	0.1	0.52	NV
hexachlorobutadiene	0.002	0.01	2	1.3	1.3
hexachloroethane	0.002	0.01	3	NV	NV
methoxychlor	0.002	0.05	20	0.17	NV
mirex	0.002	NV	0.0085	NV	NV
DDD, o,p'-	0.0004	NVI	NVI	NVI	NV
DDD, p,p'-	0.0004	NVI	NVI	NVI	NV
DDD, total	0.0006	0.05	NVI	NVI	NV
DDE, o,p'-	0.0004	NVI	NVI	NVI	NV
DDE, p,p'-	0.0004	NVI	NVI	NVI	NV
DDE, total	0.0006	0.01	NVI	NVI	NV
DDT, o,p'-	0.0004	NVI	NVI	NVI	NV
DDT, p,p'-	0.0004	NVI	NVI	NVI	NV
DDT, total	0.0006	0.05	NVI	NVI	NV
DDT and metabolites <sup>1</sup>	0.001	NV	0.01	93	NV

NV = no value applied  
NVI = no value for individual isomer(s), standard/guideline applies to sum  
LOR = limit of reporting  
RDL = maximum reporting detection limit  
<sup>1</sup> DDT and metabolites is the sum of all isomers of DDD, DDE, and DDT.

*Trace OCP Water Reporting Limits & Canadian Regulatory Standards*

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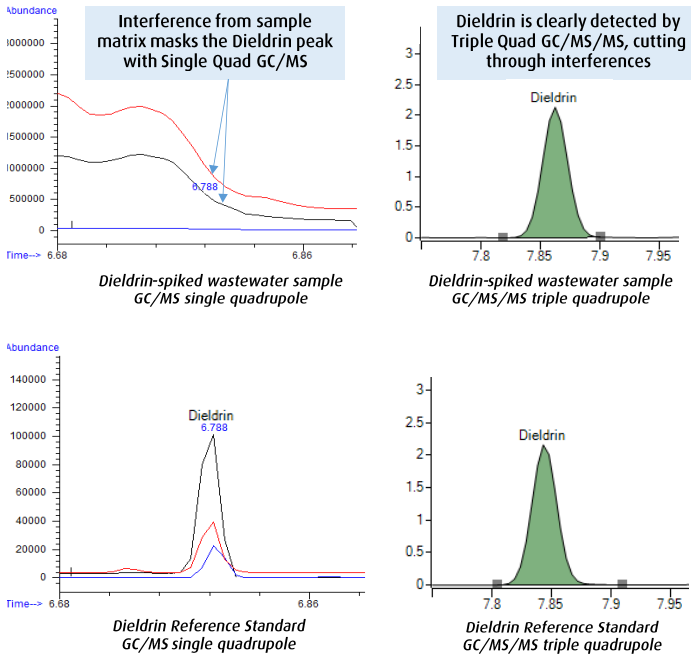


Figure 1: Effect of Interferences on GC/MS vs GC/MS/MS (same sample)

## Data Quality Benefits of GC/MS/MS

Traditional single quadrupole GC/MS provides good sensitivity and good selectivity from common interferences found in surface waters or drinking waters, but where ultra-trace detection limits are required, or for complex matrices such as wastewaters and some groundwaters where many potential interferences may be present, GC/MS/MS gives the most reliable results using the smallest possible sample size.

Figure 1 shows an example of single quadrupole GC/MS interferences for the measurement of Dieldrin in a wastewater sample, where these same interferences are easily resolved by triple quad GC/MS/MS. The bottom images show mass chromatograms from the wastewater sample, which has been “spiked” (fortified) with Dieldrin. In this example, the single quadrupole GC/MS data shows a major interference, which would lead to a non-detected test result with an elevated LOR, where the GC/MS/MS triple quad easily detects Dieldrin in the same sample with no interference.

In addition to its use for trace level detection limits, the GC/MS/MS method can also resolve interferences encountered during testing of difficult samples by our routine GC/MS method, however some complex matrix samples (e.g. wastewaters) may require dilution with corresponding increases to our trace LORs.

## Sampling Considerations

Water samples for our Trace OCP method should be collected using 100 mL amber glass sample bottles which have been validated for suitability by ALS. For groundwater sampling, exclusion of sediment is important to avoid a potential for high bias of hydrophobic OCPs, which may be sorbed to particulate matter. Low flow sampling techniques are recommended to minimize disturbance and uptake of sediment. Sample filtration is not permitted for OCPs due to potential sorption to filtration media. Chlorinated waters should be preserved with sodium thiosulfate to reduce any residual free chlorine. Holding time for this test is 7 days (14 days for Ontario MOE applications).



Figure 2: New 100 mL bottle and old 500 mL bottle

ISO 17025 accreditation for the GC/MS/MS trace method is pending (expected January 2020). Refer to the ALS Waterloo [CALA Scope of Accreditation](#) for current status and details.