



Super Trace PCB analysis to meet ANZECC 99% protection limits with improved precision and accuracy

BACKGROUND

Over the last decade ALS has refined methodologies and used improved instrumentation to reach lower detection limits for trace organics. One of the final challenges for ALS in Australia was achieving the very low trigger levels required for PCBs which are persistent and bio-accumulating and members of the Stockholm convention dirty dozen. One of the challenges in achieving trace Limits of Reporting (LOR) is that reliability at LOR is a combination of the accuracy, precision, signal to noise ratio and system control to remove potential false positives.

ALS had generally offered trace PCB analysis to 0.1 or 0.05µg/L depending on the matrix (e.g. drinking water or other clean matrices). Following a long term method development project, ALS now offers LORs at one tenth of previous limits with Aroclors including 1254 reporting to 5ppt or 0.005µg/L.

This now allows industry to easily meet the ANZECC 99% protection for fresh waters and can be simultaneously analysed with trace OCPs and PAHs (B(a)P - refer to [EnviroMail 99 - OC Pesticide and PAHs for ANZECC 99 percent protection and ADWG compliance.pdf](#))

NEW ALS SUPER ULTRA-TRACE CAPABILITIES

The ALS R&D team has validated this method across multiple water matrices. One pleasing aspect of this methodology is the improved precision, accuracy and quality achieved, thereby delivering increased confidence for practitioners monitoring sensitive environments.

In keeping with ALS manual handling objectives, the LORs are achieved using a standard ALS 500ml Amber bottle and prices will be at about the same level as the ultra-trace rates of 2014 to avoid cost increases.

METHOD AND LOR INFORMATION

Super Trace PCBs pristine water matrices
 EP131B-ST LOR: 0.005 µg/L

STOCKHOLM CONVENTION AND PCB PERSISTENCE

In 1995, The UN called for global action to be taken on POPs, which it defined as "chemical substances that persist in the environment bio-accumulate and present a higher risk of causing adverse effects to human health and the environment". In 2001 agreement was reached in Stockholm to target the reduction of 12 persistent organics that we know today as 'Stockholm convention' pollutants (POPs). PCBs were one of the original dirty dozen and as the name suggest, still exist in many environments. Trace monitoring is used to protect many of the more pristine environments in Australia and many other countries.

ACHIEVING ANZECC 99% TRIGGER LEVELS WITHOUT USING EXPENSIVE HRMS

Table 1 below is familiar to many industry experts. Not all limits could be bettered in the past unless using expensive HRMS techniques. New ALS super trace methods overcome this gap with PCB LORs now reliably below (i.e. half) of the 99% trigger level for fresh waters.

Table 1 - Australian and New Zealand Guidelines for Fresh and Marine Water Quality - October 2000.

Chemical	Trigger values for freshwater (µg/L ⁻¹)				
	Level of protection (% species)				
	99%	95%	90%	80%	
Polychlorinated Biphenyls (PCBs) & Dioxins					
Capacitor 21	B	ID	ID	ID	ID
Aroclor 1016	B	ID	ID	ID	ID
Aroclor 1221	B	ID	ID	ID	ID
Aroclor 1232	B	ID	ID	ID	ID
Aroclor 1242	B	0.3	0.6	1.0	1.7
Aroclor 1248	B	ID	ID	ID	ID
Aroclor 1254	B	0.01	0.03	0.07	0.2
Aroclor 1260	B	ID	ID	ID	ID
Aroclor 1262	B	ID	ID	ID	ID
Aroclor 1268	B	ID	ID	ID	ID

PRECISION, ACCURACY AND LORS FOR PCBs

This new ALS methodology also has improvements in Quality as follows;

- The Previous ALS Super Ultra Trace PCB method had a recovery s of 85% with an average RSD of 7.7% at 50ppt.
- The new Super Trace PCB method has an average recovery of 96% (a range of 89-101% over three matrices) at the LOR of 5ppt. This method also has a very tight overall average RSD of 6.3% and an average MDL of 0.9ppt or 0.0009 µg/L a significant improvement given the ten-fold lower LOR.

This method performance not only provides lower LORs but significant benefits to quality (precision and accuracy) and enhances overall confidence in the results for comparison to guidelines given the lower reporting limits.

REPORTING AND LORS FOR SELECT OCPS AND PAHS

The new PCB EP131B-ST method reports the same seven Aroclors (if identified) and total PCBs. The method uses classical techniques of pattern recognition and identification using the identified Aroclor for quantitation Analytes are shown below with limits and ALS LORs. The ALS objective was reporting an LOR of 0.005µg/L, half the lowest trigger limit of 0.01µg/L. This objective was achieved. Example reporting follows, with good surrogate recovery a feature of this method.

Analytical Results

Sub-Matrix: WATER (Matrix: WATER)		Client sample ID			EP131B-ST	EP131B-ST
		Client sampling date / time			[18-Feb-2016]	[18-Feb-2016]
Compound	CAS Number	LOR	Unit	ES1603632-001	ES1603632-002	
				Result	Result	
EP131B: Polychlorinated Biphenyls (as Aroclors)						
Total Polychlorinated biphenyls	----	0.005	µg/L	<0.005	<0.005	
Aroclor 1016	12674-11-2	0.005	µg/L	<0.005	<0.005	
Aroclor 1221	11104-28-2	0.005	µg/L	<0.005	<0.005	
Aroclor 1232	11141-16-5	0.005	µg/L	<0.005	<0.005	
Aroclor 1242	53469-21-9	0.005	µg/L	<0.005	<0.005	
Aroclor 1248	12672-29-6	0.005	µg/L	<0.005	<0.005	
Aroclor 1254	11097-69-1	0.005	µg/L	<0.005	<0.005	
Aroclor 1260	11096-82-5	0.005	µg/L	<0.005	<0.005	
EP131T: PCB Surrogate						
Decachlorobiphenyl	2051-24-3	0.01	%	107	104	

SAMPLING CONSIDERATIONS, SAMPLE CONTAINERS AND TEST COMMUNICATION

With ultra-trace testing, it is very important to consider whether the samples are likely to be of a 'pristine' nature. Samples should not be submitted containing high levels of TRH and other organics (without discussion) and LORs may be raised for dirty matrices. High levels of TRH will also result in LOR increases. Another key consideration is the impact of even the smallest traces of sediment as discusses in EnviroMail 28 (link follows) [EnviroMail28 Super Ultra-Trace PAHs in water July 2008](#)

This new ALS methodology can only be performed using ALS traceable 500mL Amber bottles. Both PCBs, OC Pesticides and PAHs can be simultaneously extracted for analysis and therefore one bottle will meet both combined analytical needs. Additional bottles should be provided for lab duplicates (1 in 10) and matrix spikes (1 in 20) however for simplicity it is recommended that one sample in every batch have three bottles provided every 10 samples or part thereof. To avoid confusion with other ultra-trace methods please record the method codes EP131B-ST on COCs.

REFERENCES

Australian and New Zealand Guidelines for Fresh and Marine Water Quality - October 2000