

# Analysis of EDB, 123-TCP, and DBCP

By USEPA Method 524.3

Eclipse 4760 Purge and Trap and the 4100 Autosampler

## Introduction

Many laboratories analyze 1,2-Dibromoethane (EDB), 1,2,3-Trichloropropane (123-TCP), and 1,2-Dibromo-3-chloropropane (DBCP) in drinking water by USEPA Method 504.1 or USEPA 8011. These methods require a micro-extraction step and analysis with dual column gas chromatography with dual electron capture detectors. These compounds may also be analyzed by USEPA Method 524.3 using purge and trap sample concentration followed by gas chromatographic separation with mass spectrometric detection (GC/MS) and selective ion monitoring (SIM). This approach is simpler since there is no sample prep and has the advantage of mass spec selectivity while yielding equivalent, if not better reporting limits than the GC/ECD method.

## Methodology

Method 524.3 was followed using the instrumentation and conditions listed below. A seven-point calibration from 20 ppt to 2000 ppt was run. Initial demonstrations of capability (IDC) were run by analyzing seven replicates of 100 ppt and also 250 ppt standard. Seven replicates at 20 ppt were analyzed to determine the half range for the prediction interval of results ( $HR_{PIR}$ ). All standards and QC samples were prepared using the ascorbic acid/maleic acid preservative required by the method.



**Table 1. Instrument Configuration and Operating Conditions**

Purge-and-Trap	Eclipse 4760 P & T Sample Concentrator	Autosampler	4100 Water/Soil Sample Processor
Trap	#10 trap; Tenax® / Silica gel / CMS	System Gas	Zero grade nitrogen
Purge Gas	Zero grade Helium at 45 mL/min	Purge Gas	Zero grade helium
Purge Time	10 min	LV20 Pressure	8.0 psi
Spurge Mount Temperature	45 °C	Loop-based Time Settings	Default
Sample Temperature (purge)	45 °C	Rinse Water	80 °C
Sample Temperature (bake)	45 °C	Soil Sample Transfer	150 °C
Desorb Time	0.5 min	Soil Oven	150 °C
Bake Time	4 min	Soil Lift Station	45 °C
OI #10 Trap Temperature	Ambient during purge 180 °C during desorb pre-heat 190 °C during desorb 210 °C during bake		
Water Management	120 °C during purge Ambient during desorb 240 °C during bake		
Transfer Line Temperature	140 °C		
Six-port Valve Temperature	140 °C		

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Using USEPA Method 524.3

4100 Sample Processor Methods		
Sample Type	Waters Only	Blanks Only
Vial Cap Color	Blue	Green
Needle Rinses	1	0
SAM A (µL)	5	5
SAM B (µL)	0	0
SAM C (µL)	0	0
SAM D (µL)	0	0
Purge Time (min)	10.0	10.0
Desorb Time (min)	0.5	0.5
P&T Rinses	3	0
Rinse Water	Hot	Hot
Water Stir Time (min)	0.0	
Water Settle Time (sec)	0	
Soil Add Water to Vial (#loops)		
Soil Pre-Heat Stir		
Soil Pre-Heat/Purge Temp (°C)		
Soil Stir During Purge		

Gas Chromatograph	Agilent 7890A
Column	Restek Rtx-VMS 20 meter, 0.18 mm ID, 1.0 µm film
Carrier Gas	Zero grade helium
Inlet Temperature	250 °C
Inlet Liner	Agilent Ultra Inert, 1 mm straight taper
Column Flow Rate	0.6 mL/min
Split Ratio	30:1
Oven Program	Hold at 40 °C for 1 min 18 °C/minute to 180 °C 40 °C/minute to 220 °C Hold at 220 °C for 3 min Total GC Run is 12.78 min

Mass Spectrometer	Agilent 5975C	
Mode	SIM 100 msec dwell time	
SIM Compounds		
Group 1	1,2-Dibromoethane	m/z 107,109
Group 2	Chlorobenzene-d5	m/z 82,117,119
Group 3	4-Bromofluorobenzene	m/z 95,174,176
Group 4	1,2-3-Trichloropropane	m/z 75,77,100
Group 5	1,4-Dichlorobenzene-d4	m/z 115,150,152
Group 6	1,2-Dichlorobenzene-d4	m/z 115,150,152
Group 7	1,2-Dibromo-3-Chloropropane	m/z 75,155,157
Scans/Second	5.19	
Solvent Delay	1.4 min	
Transfer Line Temperature	250 °C	
Source Temperature	300 °C	
Quadrupole Temperature	200 °C	
Draw Out Plate	6 mm	

### Results and Discussion

See Tables 2 through 6 for calibration data and USEPA Method 524.3 Quality Control data. The calibration must be validated by calculating the concentration of the analytes for each of the points used to generate the curve by use of the regression equations.

**Table 2. Calibration Data**

Analyte	Compound	Avg RF	% RSD
1	Chlorobenzene-d5 (IS)	N/A	N/A
2	1,2-Dibromoethane	0.340	4.15
3	1,4-Dichlorobenzene-d4 (IS)	N/A	N/A
4	4-Bromofluorobenzene (SS)	0.796	1.82
5	1,2,3-Trichloropropane	0.783	4.02
6	1,2-Dichlorobenzene-d4 (SS)	1.011	3.19
7	1,2-Dibromo-3-chloropropane	0.210	15.62

**Table 3. Calibration Acceptance and Validation - 5 mL of 524.3 Standard**

[Results Signal 1]	20 ppt Std	20 ppt % Rec	50 ppt Std	50 ppt % Rec	100 ppt Std	100 ppt % Rec	250 ppt Std	250 ppt % Rec	500 ppt Std	500 ppt % Rec	1000 ppt Std	1000 ppt % Rec	2000 ppt Std	2000 ppt % Rec
Chlorobenzene-d5 (IS)	500	100%	500	100%	500	100%	500	100%	500	100%	500	100%	500	100%
1,2-Dibromo-ethane	22.12	111%	55.36	111%	106.70	106%	272.89	109%	523.82	105%	1027.31	103%	1976.83	99%
1,4-Dichloro-benzene-d4 (IS)	500	100%	500	100%	500	100%	500	100%	500	100%	500	100%	500	100%
4-Bromofluoro-benzene (SS)	498.29	100%	495.99	99%	492.50	99%	493	99%	494.24	99%	513.75	103%	512.23	102%
1,2,3-Trichloro-propane	21.91	110%	55.88	112%	104.63	105%	270.33	108%	515.01	103%	1022.98	102%	1981.82	99%
1,2-Dichloro-benzene-d4 (SS)	507.62	102%	497.94	100%	493.47	99%	498.87	100%	501.48	100%	500.97	100%	499.64	100%
1,2-Dibromo-3-chloropropane	21.61	108%	60.02	120%	106.29	106%	271.18	108%	517.04	103%	1029.68	103%	1977.67	99%

**Table 4. Initial Demonstration of Capability at 100 ppt**

100 ppt	IDC 1	IDC 2	IDC 3	IDC 4	IDC 5	IDC 6	IDC 7	Precision % RSD ≤ 20 %	Accuracy % Recovery + - 20%
Chlorobenzene-d5 (IS)	500	500	500	500	500	500	500	N/A	100.00
1,2-Dibromoethane	101.99	107.32	104.85	105.05	104.78	103.67	106.75	1.71	104.92
1,4-Dichlorobenzene-d4 (IS)	500	500	500	500	500	500	500	N/A	100.00
4-Bromofluorobenzene (SS)	504.76	495.08	499.53	496.20	496.45	501.74	501.78	0.72	99.87
1,2,3-Trichloropropane	101.32	106.91	105.83	106.21	106.04	105.8	107.82	1.95	105.70
1,2-Dichlorobenzene-d4 (SS)	496.66	505.69	508.38	506.60	515.47	512.23	510.79	1.19	101.59
1,2-Dibromo-3-chloropropane	111.68	106.24	103.11	100.32	100.82	97.36	98.10	4.91	102.52

**Table 5. Initial Demonstration of Capability at 250 ppt**

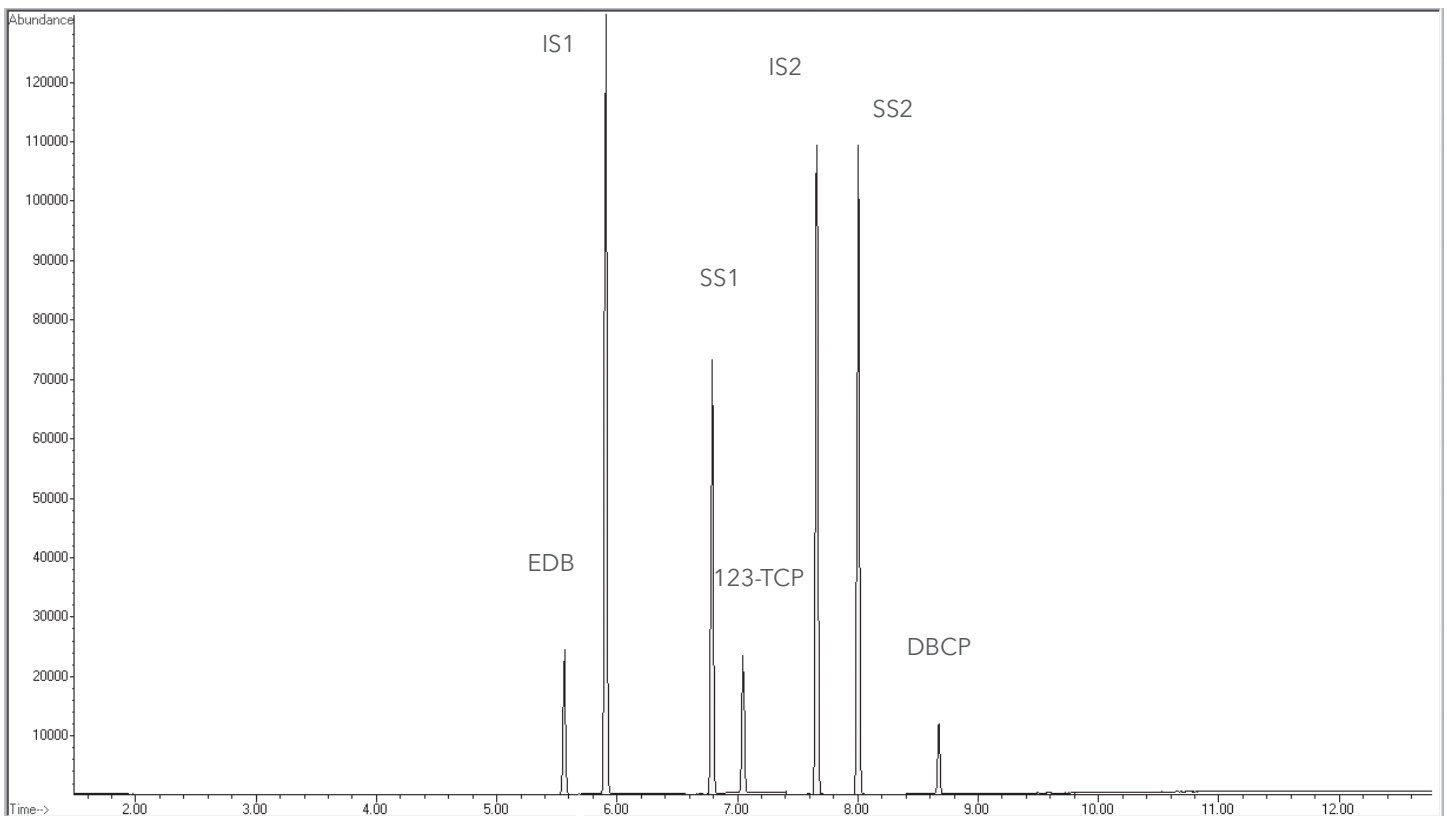
250 ppt	IDC 1	IDC 2	IDC 3	IDC 4	IDC 5	IDC 6	IDC 7	Precision % RSD ≤ 20 %	Accuracy % Recovery + - 20%
Chlorobenzene-d5 (IS)	500	500	500	500	500	500	500	N/A	100.00
1,2-Dibromoethane	270.31	273.31	279.05	277.28	274.09	270.32	268	1.46	109.28
1,4-Dichlorobenzene-d4 (IS)	500	500	500	500	500	500	500	N/A	100.00
4-Bromofluorobenzene (SS)	494.02	499.7	496.26	495.66	504.14	496.54	501.51	0.73	99.65
1,2,3-Trichloropropane	270.27	276.10	282.09	276.90	281.39	271.80	273.69	1.64	110.41
1,2-Dichlorobenzene-d4 (SS)	517.41	511.79	517.66	513.69	522.48	522.66	522.64	0.86	103.67

1,2-Dibromo-3-chloropropane	263.33	260.60	279.67	261.18	268.73	265.01	253.61	3.06	105.84
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**Table 6. Minimum Reporting Level (MRL) Confirmation**

20 ppt	PIR 1	PIR 2	PIR 3	PIR 4	PIR 5	PIR 6	PIR 7	Mean	STD DEV	HR <sub>PIR</sub>	(4.0 ppt = True Value) Upper PIR Limit <= 150%	Lower PIR Limit >= 50%
Chlorobenzene-d5 (IS)	500	500	500	500	500	500	500	N/A	N/A	N/A	N/A	N/A
1,2-Dibromoethane	22.87	22.51	22.10	22.22	22.20	21.95	22.86	22.39	0.37	1.45	119.21	104.66
1,4-Dichlorobenzene-d4 (IS)	500	500	500	500	500	500	500	N/A	N/A	N/A	N/A	N/A
4-Bromofluorobenzene (SS)	509.84	498.94	504.58	498.39	502.51	511.53	505.63	504.49	5.02	19.91	104.88	96.92
1,2,3-Trichloropropane	21.63	21.88	21.81	21.90	22.28	22.33	21.59	21.92	0.29	1.15	115.33	103.84
1,2-Dichlorobenzene-d4 (SS)	481.10	496.06	506.4	498.39	511	510.32	497.02	500.04	10.43	41.33	108.27	91.74
1,2-Dibromo-3-chloropropane	23.19	22.28	20.81	21.01	20.98	21.14	23.53	21.85	1.14	4.53	131.91	86.58

**Figure 1. 250 ppt Standard**



## Conclusions

This analysis by Method 524.3 using GC/MS SIM offers a fast, accurate alternative to Method 504.1 and 8011 and easily meets low reporting levels. The preservative for this method can build up in the purge and trap, so care must be taken to run cleaning blanks, rinsing the sparger between samples, and occasionally rinsing the sample pathway.

## References

**USEPA Method 524.3** Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry, Version 1.0 June 2009

**USEPA Method 504.1** 1,2-Dibromoethane (EDB), 1,2-Dibromo-3-chloro-propane (DBCP), and 1,2,3-Trichloropropane (123TCP) in Water by Microextraction and Gas Chromatography, Revision 1.1 Edited 1995

**USEPA Method 8011** 1,2-Dibromoethane and 1,2-Dibromo-3-Chloropropane by Microextraction and Gas Chromatography, Revision 0 July 1992



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