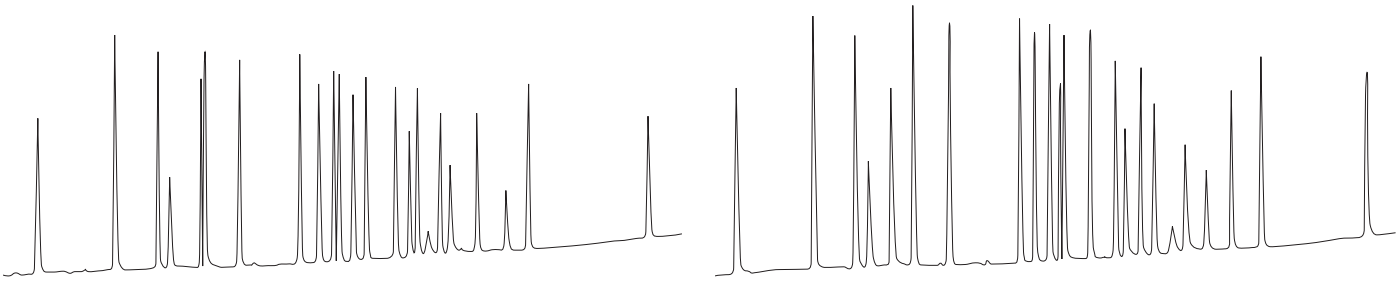
EPA Method 8081A



PRIMARY COLUMN

Column: Zebron ZB-5
Dimensions: 30m x 0.53mm ID x 1.5µm df
Order No.: 7HK-G002-28
Injection: Pulsed Splitless @ 200°C, 1µL
Carrier Gas: Helium 65cm/sec
Oven Program: 120°C (hold 1min), 120-300°C @ 9°C/min (hold 4min)
Detector: Electron Capture (ECD) @ 300°C

CONFIRMATION COLUMN

Column: Zebron ZB-35
Dimensions: 30m x 0.53mm ID x 1.0µm df
Order No.: 7HK-G003-22
Injection: Pulsed Splitless @ 200°C, 1µL
Carrier Gas: Helium 65cm/sec
Oven Program: 120°C (hold 1min), 120-300°C @ 9°C/min (hold 4min)
Detector: Electron Capture (ECD) @ 300°C

ZB-5 Primary Column			ZB-35 Confirmation Column		
Sample Analyte	Ret Time (min)	On Column Conc. (ng)	Sample Analyte	Ret Time (min)	On Column Conc. (ng)
Tetrachloro-m-xylene	8.22	0.0513	Tetrachloro-m-xylene	8.29	0.0529
alpha-BHC	9.96	0.0486	alpha-BHC	9.86	0.0506
gamma-BHC	10.89	0.0487	gamma-BHC	10.70	0.0508
beta-BHC	11.13	0.0481	beta-BHC	10.96	0.0499
delta-BHC	11.84	0.0564	delta-BHC	11.39	0.0597
Heptachlor	11.95	0.0455	Heptachlor	11.89	0.0503
Aldrin	12.70	0.0481	Aldrin	12.61	0.0496
Heptachlor epoxide	14.04	0.0492	Heptachlor epoxide	14.04	0.0497
gamma-Chlordane	14.46	0.0474	gamma-Chlordane	14.32	0.0498
alpha-Chlordane	14.79	0.0480	alpha-Chlordane	14.62	0.0512
Endosulfan I	14.89	0.0473	4,4'-DDE	14.84	0.0490
4,4'-DDE	15.21	0.0456	Endosulfan I	14.92	0.0507
Dieldrin	15.50	0.0465	Dieldrin	15.45	0.0491
Endrin	16.15	0.0447	Endrin	15.95	0.0493
4,4'-DDD	16.45	0.0445	4,4'-DDD	16.14	0.0470
Endosulfan II	16.61	0.0453	Endosulfan II	16.44	0.0487
4,4'-DDT	17.11	0.0469	4,4'-DDT	16.74	0.0596
Endrin aldehyde	17.34	0.0451	Endrin aldehyde	17.35	0.0496
Endosulfan sulfate	17.92	0.0480	Methoxychlor	17.78	0.0548
Methoxychlor	18.59	0.0462	Endosulfan sulfate	18.28	0.0544
Endrin ketone	19.05	0.0464	Endrin ketone	18.87	0.0514
Decachlorobiphenyl	21.68	0.0479	Decachlorobiphenyl	20.99	0.0525

Special thanks to STL - Buffalo, NY for chromatographic data and technical information

EPA Method 8081A

Method 8081A is a dual-column analysis for chlorine containing pesticides in which one column, ZB5, is used for analysis; while a separate column of alternate selectivity, ZB35 or ZB1701, is used to confirm the presence of target analytes. A specialized ECD detector, which has a high sensitivity for Chlorine containing compounds, is used to achieve method detection levels in the low ppb range. The difficulty of this method is two fold: First, finding conditions under which two columns will resolve all of the 22 most commonly targeted compounds and second, maintaining a system that is devoid of activity to prevent breakdown of the sensitive compounds such as Endrin, DDT, and Methoxychlor.

One of the major problems in the method is the breakdown of Endrin, DDT, and Methoxychlor. The breakdown is due to active silanol sites in the system. Free Silanol groups are present in even the most deactivated glass. The concentration of free Silanols will increase as the column ages due to a loss of stationary phase or deactivation agent, resulting in higher and higher activity. Samples that are overly acidic or basic, or that are considered dirty will cause the deactivation to diminish faster, resulting in increased activity.

If low recoveries or breakdown are observed, check the injection port. Start by replacing the liners, septa, and cutting a few inches off the head of the guard column. It is recommended that liners used contain silanized glass wool to trap particulates such as small septa plugs or unvolatilized residue from the injections. This practice will lower the amount of maintenance required on all parts of the system past the injection liner. If the problem persists perform injection port maintenance again, but this time replace the gold disk at the bottom of the injection port (HP Split/Splitless injectors only).

Once all other variables have been eliminated, the column should be investigated as a source of activity. The degradation activity is usually more pronounced in the first 4" of any column, which is why the chromatography will improve after the column has been trimmed. If the column is still good, the most common ways to reduce activity are to trim 4"-12" from the inlet end of the column, bake it out at 10°C above the methods highest temperature, or the columns maximum temperature, for up to 2 hours, or in extreme cases to solvent rinse the column.

ZB-5

		15 Meter		30 Meter		60 Meter	
ID (mm)	df (µm)	order no.	price	order no.	price	order no.	price
	0.10	7EG-G002-02		7HG-G002-02		7KG-G002-02	
0.25	0.25	7EG-G002-11		7HG-G002-11		7KG-G002-11	
	0.50			7HG-G002-17			
	1.00	7EG-G002-22		7HG-G002-22		7KG-G002-22	
	0.10			7HM-G002-02			
0.32	0.25	7EM-G002-11		7HM-G002-11		7KM-G002-11	
	0.50			7HM-G002-17			
	1.00	7EM-G002-22		7HM-G002-22		7KM-G002-22	
	0.50	7EK-G002-17		7HK-G002-17			
0.53	1.50	7EK-G002-28		7HK-G002-28		7KK-G002-28	
	3.00	7EK-G002-36		7HK-G002-36			
	5.00			7HK-G002-39			

ZB-35

0.25	0.25	7EG-G003-11		7HG-G003-11			
	0.50	7EG-G003-17		7HG-G003-17			
0.32	0.25			7HM-G003-11		7KM-G003-11	
	0.50			7HK-G003-17			
0.53	1.00	7EK-G003-22		7HK-G003-22			

ZB-1701

0.25	0.25	7EG-G006-11		7HG-G006-11		7KG-G006-11	
0.32	0.25	7EM-G006-11		7HM-G006-11		7KM-G006-11	
New Dimension		7HK-G006-22		30m x 0.53mm x 1.0µm			