METHOD 9078

SCREENING TEST METHOD FOR POLYCHLORINATED BIPHENYLS IN SOIL

1.0 SCOPE AND APPLICATION

1.1 The method may be used to determine the amount of PCB (polychlorinated biphenyl) contamination in soils such as sand, gravel, loam, sediment, and clay, assuming that PCBs are the sole source of organic halogens in the sample.

1.2 This electrochemical method is designed to provide quantitative field results over a range of 2 to 2000 µg/g PCBs, significantly cutting down on the number of samples requiring laboratory testing.

1.3 Chlorines are removed from the PCB molecule using an organo-sodium reagent. The resulting chloride ions are measured using a chloride specific electrode. Analysts must identify the type of Aroclor contamination in order to use this as a quantitative method.

1.4 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

A sample of the soil to be tested is extracted with a hydrocarbon based solvent. The resulting extract is filtered to remove moisture and inorganic salts. The dried extract is reacted with metallic sodium and a catalyst to strip chloride from any PCB that may be present. The resulting chloride ions are extracted into an aqueous buffer solution where they are detected using a chloride ion specific electrode.

CAUTION: Some of the reagents used with this testing procedure contain flammable solvents, dilute acids, and metallic sodium. Wear gloves and safety glasses while performing tests. Read all MSDS and warnings included with the instrument before starting testing procedure.

3.0 INTERFERENCES

3.1 This procedure is sensitive to any chlorinated compound that is preferentially soluble in a non-polar solvent. When analyzing for PCBs, the presence of other chlorinated organics will result in a high bias. Iodine and bromine containing compounds will affect results if present in significant quantities. Wet or dry samples may be run, but results for all samples are calculated on a wet-weight basis. In one evaluation study (Table 1), 1.4% of the measurements were false negatives.

3.2 Inorganic chlorides should not interfere using this method if the sample is extracted with organic solvent.

4.0 APPARATUS AND MATERIALS

Electrochemical PCB test kit: L2000® PCB/Chloride Analyzer, (Dexsil Corporation, One Hamden Park Drive, Hamden, CT), or equivalent. Each commercially available test kit will supply or specify the apparatus and materials necessary for successful completion of the test.

5.0 REAGENTS

Each commercially available test kit will supply or specify the reagents necessary for successful completion of the test. Reagents should be labeled with appropriate expiration dates.

6.0 SAMPLE COLLECTION AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Sec. 4.1.

6.2 Soil samples may be contaminated, and should therefore be considered hazardous and handled accordingly. All samples should be collected using a sampling plan that addresses the considerations discussed in Chapter Nine.

6.3 To achieve accurate analyses, soil samples should be well homogenized prior to testing. PCBs are generally not evenly distributed in a soil sample and extensive mixing must be done to assure consistency.

7.0 PROCEDURE

Follow the manufacturer's instructions for the test kit being used. Those test kits used must meet or exceed the performance specifications indicated in Tables 1 and 2.

8.0 QUALITY CONTROL

8.1 Follow the manufacturer's instructions for quality control procedures specific to the test kit used. Additionally, guidance provided in Chapter One should be followed.

8.2 Use of replicate analyses, particularly when results indicate concentrations near the action level, is recommended to refine information gathered with the kit.

8.3 Method 9078 is intended for field or laboratory use. The appropriate level of quality assurance should accompany the application of this method to document data quality.

9.0 METHOD PERFORMANCE

9.1 146 soil samples from a PCB contaminated site were analyzed. There were 114 individual samples and 32 field duplicates. Each sample was analyzed using both the L2000 and GC/MS. The L2000 analyses were performed on-site in a mobile lab and the PCBs were analyzed as Aroclor 1242. Laboratory analyses were performed on splits of the same samples. The results from the analyses are presented in Table 1.

9.2 After applying accepted statistical methods to account for the detection limit difference between the two methods the data were evaluated to determine the acceptability of the L2000 method. A matched-pair students t-test performed on the L2000 and CLP GC/MS data results in a t value of 0.2141. This is well below the critical value (1.645 @ 0.05) for rejecting the null hypothesis indicating that there is no statistical difference between the data pairs. An analysis of the data for outliers identified only 2 data points whose residuals were greater than 3 standard deviations (10 and 5 respectively). Both points were determined to be in error using other evidence and were eliminated from the data set. A linear regression analysis of the remaining data results in a correlation coefficient of 0.95 and a positive intercept of 10.98 µg/g. The slope of 0.985 was not statistically different from 1 and the intercept was not statistically different from 0.

9.3 The relative percent difference (RPD) calculated from all valid duplicates greater than the L2000 detection limit of 2 µg/g for each method resulted in a mean RPD of 19% for the L2000 data and a mean RPD of 43% for the CLP GC-MS method. A Dunnett's test shows that this is statistically significant.

9.4 In a second study, soil samples contaminated with Aroclor 1260 were taken during a site cleanup. The samples were split and sent for lab analysis by Method 8082 as well as analysis by the L2000 in the field. The results are reported in Table 2. A linear regression analysis of the data resulted in a correlation coefficient of 0.995, a slope of 1.048 and an intercept of -1.48 µg/g indicating that the L2000 is accurate compared to the lab method. A calculation of the relative percent difference for data, where duplicates were run within a method, results in a lower RPD for the L2000 indicating a tighter data spread and better repeatability.

10.0 REFERENCE

1. Griffin, Roger D. Application of a New PCB Field Analysis Technique for Site Assessment. Proceedings of Hazmacon '92 March - April 1992.

TABLE 1

COMPARISON OF L2000 AND GC/MS RESULTS FROM SPLIT SAMPLES
Summary of Results

Sample Number	L2000 (µg/g)	GC/MS (µg/g)	Results Agree?
1	ND	0.593	Yes
3	ND	0.114	Yes
4	23.6	6.71	Yes
6	ND	0.679	Yes
7	ND	0.552	Yes
8	3.9	2	Yes
9	6.9	1.3	Yes
10	5.1	0.172	Yes
11	2.7	1.15	Yes
15	9.4	9.13	Yes
15D	12.5	9.84	Yes
16	484	2110	Yes
17	6.5	2.55	Yes
18	382	45.4	Yes
19	71.1	6.7	False Pos.
23	48.8	20.8	Yes
25	3.5	11.7	Yes
32	36	47.6	Yes
33	ND	6	Yes
34	14.4	34	Yes
36	>2000	816	Yes
38	778	1030	Yes
40	5.7	4.25	Yes
43	4.1	1.69	Yes
43D	3.6	1.74	Yes
50	ND	3.6	Yes
50D	ND	4.4	Yes
52	9.3	4.21	Yes
53	25.7	0.958	False Pos.
54	5.1	0.516	Yes
55	4.4	2.4	Yes
59	ND	7.9	Yes
60	2.3	0.624	Yes
60D	4.4	0.577	Yes
61	549	580	Yes

TABLE 1 (cont.)

Sample Number	L2000 (µg/g)	GC/MS (µg/g)	Results Agree?
62	111	2.35	False Pos.
64	172	19	Yes
65	ND	3.1	Yes
66	2.1	1.98	Yes
67	7.5	0.081	Yes
68	8	0.504	Yes
69	5.8	ND	Yes
69D	4.4	ND	Yes
73	37	15.8	Yes
74	22	13.3	Yes
75	61	23	Yes
76	82	46.7	Yes
78	21	2.27	Yes
79	148	42.8	Yes
80	ND	3.8	Yes
84	7.6	1.16	Yes
84D	10.9	1.08	False Pos.
85	593	428	Yes
85D	596	465	Yes
88	ND	2.7	Yes
88D	ND	1.77	Yes
89	ND	45	False Neg.
90	2	1.01	Yes
90D	ND	1.4	Yes
91	1650	1630	Yes
91D	1608	1704	Yes
92	3.14	1.21	Yes
92D	3.4	ND	Yes
95	20.6	17.5	Yes
95D	20.1	31.2	Yes
100	384	177	Yes
100D	363	167	Yes
101	8.3	1.21	Yes
102	6.3	293	False Neg.
102D	5	1.77	Yes
103	75.2	40.3	Yes
104	4.1	7.66	Yes

TABLE 1 (cont.)

Sample Number	L2000 (µg/g)	GC/MS (µg/g)	Results Agree?
107	161	14.1	Yes
108	6.1	3.84	Yes
109	P	ND	Yes
109D	10.3	ND	False Pos.
111	20	ND	False Pos.
112	240	315	Yes
113	21.8	14.9	Yes
114	107	66.3	Yes

NOTE: 75 out of 146 samples are reported in Table 1. Samples that were found to be ND for both the L2000 kit and the GC/MS determination were not reported. The determination of a "false negative" result for the L2000 technique is based on an action level of 10 µg/g. If another action limit is chosen, the rate of false negative results may differ. Similarly, a "false positive" result for the L2000 technique is indicated when the L2000 results are above 10 µg/g and the GC/MS results are "ND" or below 10 µg/g, or when the results of the L2000 techniques are higher than the GC/MS results by more than two orders of magnitude.

ND = Not detected
6 False positives: ND - 14.1 ppm by GC/MS
2 False negatives: 2.7 - 293 ppm by GC/MS
71 Non-detects: ND - 2.5 ppm by GC/MS

TABLE 2

COMPARISON OF L2000 AND GC/EC RESULTS FROM SPLIT SAMPLES
Summary of Results

Sample Number	Method 8082 (µg/g)	L2000 Results (µg/g)
1	83	79/76
2	21	22
3	12	14
4	300/375	357/326/327
5	29	27
6	106/134	116/117
7	3	7.6
8	9.3	7.2
9	1.5	5.2
10	99	93
11	7/9	13
12	3.6	12
13	4.2/6.2	2.9
14	290	254/265