METHOD 9079

SCREENING TEST METHOD FOR POLYCHLORINATED BIPHENYLS IN TRANSFORMER OIL

1.0 SCOPE AND APPLICATION

1.1 Method 9079 may be used to screen hydrocarbon based electrical insulating fluids for polychlorinated biphenyls (PCBs) at preset levels of 20, 50, 100, or 500 µg/g. The method is designed to provide screening data outside of a laboratory environment in under 10 minutes, providing a colorimetric indication that the concentration of PCBs is above or below the fixed end point. Screening procedures may significantly reduce the number of samples requiring laboratory testing.

1.2 Chlorines are removed from the PCB molecule using an organo-sodium reagent. The resulting chloride ions are measured using a colorimetric indicator.

1.3 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

2.1 A sample of the oil to be tested is reacted with a mixture of metallic sodium catalyzed with naphthalene and diglyme at ambient temperature. This process converts all organic halogens to their respective sodium halides. All halides in the treated mixture, including those present prior to the reaction, are then extracted into an aqueous buffer, a premeasured amount of mercuric nitrate is added, followed by a solution of diphenylcarbazone as the indicator.

2.2 The color of the solution at the end of the test indicates whether the sample is above or below the preset chlorine level. A yellow end point indicates a concentration greater than the set point of the test and a blue-violet end point indicates a concentration less than the set point of the test.

2.3 The end point at which each of the test kits turns positive is calibrated using Aroclor 1242 standards. Aroclor 1242 provides a conservative end point due to its low chlorine content relative to the other Aroclors used in electrical equipment. A list of Aroclors used in electrical equipment and the PCB concentration that gives a positive indication using the 50 µg/g test kit is given in Table 1.

3.0 INTERFERENCES

3.1 Water present in the sample at more than 2% may cause a low reading. Water present at this high a level results in an obvious change in the sodium reaction and the user should stop the test.

3.2 High sulfur levels (4%) will cause a high bias possibly resulting in a false positive reading. The sample will also smell strongly of sulfur after the sodium reaction.

3.3 Any chlorine contained in the sample will be measured as PCB possibly resulting in a false positive if the total non-PCB chlorine concentration in the sample is greater than the preset end point for the kit.

4.0 APPARATUS AND MATERIALS

Colorimetric test kit: Clor-N-Oil® (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. Reagents should be labeled with appropriate expiration dates.

5.0 REAGENTS

Each commercially available test kit will supply or specify the reagents necessary for successful completion of the test.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

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

6.2 Oil 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.

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 1-4.

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 test. Read all MSDS and warnings included with the kit before starting testing procedure.

WARNING: Mercury waste must be properly disposed.

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 9079 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 A double blind study was conducted using the Clor-N-Oil 50 on 6 spiked transformer oil samples. The spiking concentrations were chosen to be identical to those from three sets of the EPA's Water Pollution (WP) Performance Evaluation Program. They were made in Shell Diala A electrical insulating fluid at the concentrations listed in Table 1.

9.1.1 A total of 38 operators participated in the test, 10 of whom had prior experience with the test method, and 27 of whom had no prior experience using the kits, and 1 of whom had viewed a test demonstration before running the test themselves. This distribution of operators was chosen to verify the robust nature of the method in light of the vast range of experience typical of users in the field.

9.1.2 Each operator was given six random samples containing an unknown concentration of PCBs. Operators recorded their results as greater than or less than 50 µg/g. Only after all of the tests were run were the data collected and compared with the known values.

9.1.3 Out of the 228 tests run, 4 were invalid due to spillage of reagents or improper kit operation resulting in an incomplete test. The test data are presented in Table 2. From these data, it is evident that there is a much higher likelihood of obtaining a false positive reading than a false negative.

9.1.4 The expected certainties estimated from these data are presented in Table 3. The likelihood of obtaining a false positive approaches 90% at 90% of the action level. This reflects the conservative design of the test. At the action level of 50 µg/g, nearly 99% of the samples would be identified correctly as containing 50 µg/g of PCB or greater. These results represent errors due to all sources and therefore represent real world performance of the method by field personnel.

9.1.5 For samples containing 45 µg/g, 37 of 41 gave results >50 ppm.

9.2 The real world performance was investigated in detail by Utah Power & Light Company. They tested the insulating fluid from approximately 200,000 pieces of electrical equipment with the Clor-N-Oil 50 test kit in order to classify them as either PCB or non-PCB (i.e., containing more or less than 50 µg/g). Each piece of equipment was tested once with the test kit. A random sample of 937 of the Clor-N-Oil negatives were tested by Gas Chromatography to confirm the results. The results from the testing are summarized in Table 4. The false negative rate predicted from the data is less than 1% (0.65%). This means that a transformer can be classified as non-PCB with greater than 99% confidence.

10.0 REFERENCES

1. Finch, S.; Lynn, T.B; Lynn T.D.; and Scott, R.P.W., Which Method is the Most Reliable in the Field, EPRI PCB seminar Proceedings October 1991.

2. Mills, David W.; and Rhoads, Kirt W., Clor-N-Oil Test Kit as a PCB Screening Tool 1985. EPRI PCB Seminar Proceedings March 1986. EPRI CS/EA/EL-4480 Product 2028.

3. Rhoads, Kirt W., Clor-N-Oil Test Kit as a Risk Management Tool - An Update 1987. EPRI PCB Seminar Proceedings December 1987.

TABLE 1

SPIKING CONCENTRATIONS FOR Clor-N-Oil PCB Concentrations in Shell Diala A	
Aroclor	Concentration
1242	21.2 µg/g
1242	45.0 µg/g
1254	26.3 µg/g
1254	50.0 µg/g
1260	8.2 µg/g
1260	50.0 µg/g

TABLE 2

ESTIMATED ERROR RATES FOR Clor-N-Oil 50		
PCB Concentration µg/g	Expected False Positive Rate %	Expected False Negative Rate %
8	<3	---
21	2.8	---
26	35	---
45	88	---
50	---	1.4

TABLE 3

GC RETEST RESULTS OF 930 NEGATIVES CLASS INTERVAL (µg/g)									
	0-1	1-5	6-15	16-25	26-46	47-99	100-475	476-999	1000+
Number	768	93	48	5	10	3	3	0	0
Percent	82.6	10	5.2	0.5	1.1	0.3	0.3	0	0

TABLE 4

TEST RESULTS FOR Clor-N-Oil 50 STUDY

Sample ID	Concentration PCB (µg/g)	Aroclor	Field Test Result (µg/g)
1	50	1254	>50
2	26.3	1254	<50
3	50	1260	NA
4	45	1242	>50
5	21	1242	<50
6	8.2	1260	<50
7	21	1242	<50
8	45	1242	<50
9	26.3	1254	<50
10	50	1254	>50
11	8.2	1260	<50
12	50	1260	>50
13	26.3	1254	<50
14	45	1242	>50
15	8.2	1260	<50
16	45	1242	>50
17	50	1260	>50
18	50	1254	>50
19	8.2	1260	<50
20	26.3	1254	<50
21	45	1242	>50
22	21	1242	<50
23	45	1242	<50
24	50	1260	>50
25	21	1242	<50
26	50	1260	>50
27	50	1254	>50

TABLE 4 (cont.)

Sample ID	Concentration PCB (µg/g)	Aroclor	Field Test Result (µg/g)
28	21	1242	<50
29	45	1242	>50
30	50	1254	>50
31	8.2	1260	<50
32	50	1254	>50
33	50	1260	>50
34	50	1254	>50
35	45	1242	>50
36	26.3	1254	<50
37	50	1254	>50
38	26.3	1254	<50
39	50	1260	>50
40	21	1242	<50
41	50	1260	>50
42	8.2	1260	<50
43	26.3	1254	<50
44	8.2	1260	<50
45	21	1242	<50
46	26.3	1254	<50
47	8.2	1260	<50
48	21	1242	<50
49	50	1260	>50
50	50	1260	>50
51	8.2	1260	<50
52	45	1242	<50
53	21	1242	<50
54	26.3	1254	<50
55	21	1242	<50

TABLE 4 (cont.)

Sample ID	Concentration PCB (µg/g)	Aroclor	Field Test Result (µg/g)
56	45	1242	>50
57	8.2	1260	<50
58	50	1254	>50
59	26.3	1254	>50
60	21	1242	<50
61	8.2	1260	<50
62	45	1242	>50
63	50	1254	>50
64	50	1254	>50
65	50	1254	>50
66	50	1260	>50
67	21	1242	<50
68	50	1260	>50
69	50	1260	>50
70	45	1242	<50
71	26.3	1254	<50
72	21	1242	<50
73	8.2	1260	<50
74	8.2	1260	<50
75	50	1260	>50
76	45	1242	<50
77	45	1242	>50
78	21	1242	<50
79	8.2	1260	<50
80	50	1254	>50
81	50	1254	>50
82	26.3	1254	<50
83	26.3	1254	<50

TABLE 4 (cont.)

Sample ID	Concentration PCB (µg/g)	Aroclor	Field Test Result (µg/g)
84	26.3	1254	<50
85	21	1242	<50
86	26.3	1254	<50
87	45	1242	>50
88	50	1254	>50
89	8.2	1260	<50
90	50	1260	>50
91	45	1242	>50
92	8.2	1260	<50
93	50	1260	>50
97	45	1242	>50
95	21	1242	<50
96	50	1254	>50
97	45	1242	>50
98	45	1242	>50
99	45	1242	>50
100	26.3	1254	>50
101	50	1260	>50
102	26.3	1254	>50
103	26.3	1254	<50
104	21	1242	<50
105	26.3	1254	>50
106	50	1260	>50
107	8.2	1260	<50
108	21	1242	<50
109	26.3	1254	<50
110	50	1254	>50
111	21	1242	<50

TABLE 4 (cont.)

Sample ID	Concentration PCB (µg/g)	Aroclor	Field Test Result (µg/g)
112	45	1242	>50
113	26.3	1254	<50
114	50	1260	>50
115	21	1242	<50
116	21	1242	<50
117	26.3	1254	<50
118	8.2	1260	<50
119	50	1254	>50
120	50	1254	>50
121	50	1254	>50
122	26.3	1254	>50
123	45	1242	>50
124	50	1260	>50
125	8.2	1260	<50
126	50	1254	>50
127	8.2	1260	<50
128	45	1242	>50
129	50	1260	>50
130	21	1242	<50
131	26.3	1254	<50
132	50	1260	>50
133	26.3	1254	<50
134	45	1242	>50
135	26.3	1254	<50
136	50	1260	>50
137	50	1260	>50
138	45	1242	>50
139	21	1242	<50

TABLE 4 (cont.)

Sample ID	Concentration PCB (µg/g)	Aroclor	Field Test Result (µg/g)
140	50	1254	>50
141	8.2	1260	<50
142	8.2	1260	<50
143	50	1260	>50
144	8.2	1260	<50
145	45	1242	>50
146	8.2	1260	<50
147	26.3	1254	<50
148	21	1242	<50
149	8.2	1260	<50
150	21	1242	<50
151	50	1254	>50
152	8.2	1260	<50
153	50	1254	>50
154	50	1254	>50
155	21	1242	NA
156	50	1260	NA
157	26.3	1254	<50
158	21	1242	<50
159	26.3	1254	>50
160	8.2	1260	<50
161	50	1254	>50
162	45	1242	>50
163	50	1254	>50
164	50	1254	>50
165	50	1260	>50
166	21	1242	<50
167	50	1254	>50

TABLE 4 (cont.)

Sample ID	Concentration PCB (µg/g)	Aroclor	Field Test Result (µg/g)
168	50	1260	>50
169	45	1242	>50
170	50	1260	<50
171	50	1260	>50
172	21	1242	<50
173	50	1260	>50
174	8.2	1260	<50
175	45	1242	>50
176	8.2	1260	<50
177	8.2	1260	<50
178	45	1242	>50
179	26.3	1254	>50
180	50	1254	>50
181	45	1242	>50
182	45	1242	>50
183	21	1242	<50
184	26.3	1254	>50
185	50	1254	>50
186	21	1242	<50
187	50	126	>50
188	50	1254	>50
189	26.3	1254	<50
190	8.2	1260	<50
191	21	1242	<50
192	8.2	1260	<50
193	45	1242	>50
194	8.2	1260	<50
195	21	1242	<50

TABLE 4 (cont.)

Sample ID	Concentration PCB (µg/g)	Aroclor	Field Test Result (µg/g)
196	26.3	1254	>50
197	50	1260	>50
198	50	1260	>50
199	26.3	1254	>50
200	45	1242	>50
201	21	1242	>50
202	8.2	1260	<50
203	21	1242	<50
204	45	1242	>50
205	50	1254	>50
206	50	1254	>50
207	26.3	1254	NA
208	50	1254	>50
209	50	1254	>50
210	50	1260	>50
211	45	1242	>50
212	21	1242	<50
213	50	1260	>50
214	50	1260	>50
215	8.2	1260	<50
216	8.2	1260	<50
217	50	1260	>50
218	45	1242	>50
219	26.3	1254	>50
220	26.3	1254	>50
221	21	1242	>50
222	45	1242	>50
223	45	1242	>50

TABLE 4 (cont.)

Sample ID	Concentration PCB (µg/g)	Aroclor	Field Test Result (µg/g)
224	45	1242	>50
225	26.3	1254	>50
226	8.2	1260	<50
227	45	1242	>50
228	50	1254	>50