

METHOD 4040

SOIL SCREENING FOR TOXAPHENE BY IMMUNOASSAY

1.0 SCOPE AND APPLICATION

1.1 Method 4040 is a procedure for screening soils to determine whether toxaphene (CAS Registry 8001-35-2) is present at concentrations above 0.5 µg/g. Method 4040 provides an estimate for the concentration of toxaphene by comparison against standards.

1.2 In cases where the exact concentration of toxaphene is required, additional techniques (i.e., gas chromatography (Method 8081) or gas chromatography/mass spectrometry (Method 8270)) should be used.

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 Test kits are commercially available for this method. The manufacturer's directions should be followed.

2.2 In general, the method is performed using an extract of a soil sample. Filtered extracts may be stored cold, in the dark. An aliquot of the extract and an enzyme-toxaphene conjugate reagent are added to an immobilized toxaphene antibody. The enzyme-toxaphene conjugate "competes" with toxaphene present in the sample for binding to the immobilized toxaphene antibody. The enzyme-toxaphene conjugate bound to the toxaphene antibody then catalyzes a colorless substrate to a colored product. The test is interpreted by comparing the color produced by a sample to the response produced by a reference reaction.

3.0 INTERFERENCES

3.1 Compounds that are chemically similar may cause a positive test (false positive) for toxaphene. The test kit used to develop this method was evaluated for interferences, and found to be relatively insensitive to other organochlorine pesticides (e.g., Lindane, DDT and DDE). The data for the lower limit of detection of these compounds are provided in Table 1. Consult the information provided by the manufacturer of the kit used for additional information regarding cross reactivity with other compounds.

3.2 Storage and use temperatures may modify the method performance. Follow the manufacturer's directions for storage and use.

4.0 APPARATUS AND MATERIALS

Immunoassay test kit: EnviroGard™ Toxaphene in Soil (Millipore, Inc.), 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.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

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

6.2 Soils samples may be contaminated, and should therefore be considered hazardous and handled accordingly.

7.0 PROCEDURE

Follow the manufacturer's instructions for the test kit being used.

8.0 QUALITY CONTROL

8.1 Follow the manufacturer's instructions for the test kit being used 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 Do not use test kits past their expiration date.

8.4 Do not use tubes or reagents designated for use with other test kits.

8.5 Use the test kits within their specified storage temperature and operating temperature limits.

8.6 Method 4040 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

Table 2 provides the results of single determinations in soil from New Mexico.

10.0 REFERENCES

1. EnviroGard™ Toxaphene Soil Test Kit Guide, Millipore, Inc.
2. Marsden, P.J., S-F Tsang, V. Frank, N. Chau, and M. Roby, "Comparison of the Millipore Immunoassay for Toxaphene with Soxhlet Extraction and Method 8081", Science Applications International Corporation, under contract to Millipore Inc., May 1994, unpublished.

TABLE 1

POSSIBLE SOIL INTERFERENCES (a)

Compound	Soil Equivalent Concentration $\mu\text{g}/\text{kg}$ (ppb) Required to Yield a Positive Result
Diesel fuel	45000
Endrin	6
Endosulfan I	6
Endosulfan II	6
Dieldrin	6
Heptachlor	6
Aldrin	20
Technical Chlordane	14
gamma-BHC (Lindane)	300
alpha-BHC	1000
delta-BHC	1000

The following compounds were found to yield a negative result for concentrations up to 200,000 $\mu\text{g}/\text{kg}$:

Gasoline	PCB (Aroclor 1248)
Pentachlorophenol	Trinitrotoluene
DDT	

(a) Millipore, Inc. product literature

TABLE 2

TOXAPHENE ($\mu\text{g/g}$) RESULTS FOR NEW MEXICO SOIL SAMPLES

Method 3540/8081 (Lab 1)	Method 4040 (Lab 2)	AGREEMENT
0.09 J	<0.5	Y
0.04 J	>0.5	FP
0.04 J	>0.5	FP
0.01 J	<0.5	Y
40	>0.5	Y
19.3	>0.5	Y
<0.50	<0.5	Y
<0.50	>0.5	FP
0.26 J	>0.5	FP
1.0	>0.5	Y
0.14 J	>0.5	FP
0.27 J	>0.5	FP
27.2	>0.5	Y
0.14 J	>0.5	FP
0.48 J	>0.5	Y
0.21 J	>0.5	FP
6.0	NA	-
6.0	NA	-
4.8	>0.5	Y
0.049 J	>0.5	FP
0.054 J	>0.5	FP
1.3	>0.5	Y
0.15 J	>0.5	Y
0.058 J	>0.5	FP
89.6	>0.5	Y
0.5	>0.5	Y

TABLE 2 (cont.)

Method 3540/8081 (Lab 1)	Method 4040 (Lab 2)	AGREEMENT
3.7	NA	-
3.6	NA	-
35.6	>0.5	Y
0.16 J	>0.5	FP
0.88	>0.5	Y
0.41 J	>0.5	FP
0.30 J	>0.5	FP
0.10 J	>0.5	FP
323	>0.5	Y

NA = not analyzed

J = an estimate value. This is used to indicate the result is less than the sample quantitation limit but greater than zero.

Y = Yes, FN = False Negative, FP = False Positive