Designation: D 5475 – 93 (Reapproved 2002)

An American National Standard

Standard Test Method for Nitrogen- and Phosphorus-Containing Pesticides in Water by Gas Chromatography with a Nitrogen-Phosphorus Detector[1]

This standard is issued under the fixed designation D 5475; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This is a gas chromatographic (GC) test method applicable to the determination of certain nitrogen- and phosphorus-containing pesticides in ground water and finished drinking water.[2] The analytes listed in Table 1 have been validated using this test method.

1.2 This test method has been validated on reagent water and finished drinking water by 10 volunteer laboratories. Summary statistics were calculated for mean recovery, overall method precision and bias and single analyst precision using a computer program, Interlaboratory Method Validation Study (IMVS).[2]

1.3 Collaborative study showed the test method to be acceptable for all analytes tested except merphos, which decomposed in the GC injection port.

1.4 This test method is restricted to use by or under the supervision of analysts experienced in the use of GC and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this test method using the procedure described in 12.3.

1.5 Analytes that are not separated chromatographically, that is, analytes which have very similar retention times, cannot be individually identified and measured in the same calibration mixture or water sample unless an alternative technique for identification and quantitation exist (13.5).

1.6 When this test method is used to analyze unfamiliar samples for any or all of the analytes above, analyte identifications should be confirmed by at least one additional qualitative technique.

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1129 Terminology Relating to Water[3]

D 1192 Specification for Equipment for Sampling Water and Steam in Closed Conduits[3]

D 1193 Specification of Reagent Water[3]

D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D19 on Water[3]

D 3370 Practices for Sampling Water from Closed Conduits[3]

D 3694 Practices for Preparation of Sample Containers and for Preservation of Organic Constituents[4]

2.2 *U.S. EPA Method:*

EPA Method 507, Revision 2.0 Determination of Nitrogen- and Phosphorus-Containing Pesticides in Water by Gas Chromatography with a Nitrogen-Phosphorus Detector[5]

3. Terminology

3.1 *Definitions*—For definitions of water terms used in this practice, refer to Terminology D 1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *internal standard*—a pure analyte(s) added to a solution in known amount(s) and used to measure the relative responses of other test method analytes and surrogates that are components of the same solution.

3.2.1.1 *Discussion*—The internal standard must be an analyte that is not a sample component.

3.2.2 *surrogate analyte*—a pure analyte(s), which is extremely unlikely to be found in any sample, and which is added to a sample aliquot in known amount(s) before extraction and is measured with the same procedures used to measure other sample components.

3.2.2.1 *Discussion*—The purpose of a surrogate analyte is to monitor test method performance with each sample.

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[1] This test method is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.06 on Methods for Analysis for Organic Substances in Water.

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[2] Edgell, K. W., Jenkins, E. L., Lopez-Avila, V., and Longbottom, J., "Capillary Column Gas Chromatography with Nitrogen-Phosphorus Detection for Determination of Nitrogen- and Phosphorus-Containing Pesticides in Finished Drinking Waters: Collaborative Study," Journal of Association of Official Analytical Chemists, Vol 74, 1991, pp. 295–309.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Annual Book of ASTM Standards*, Vol 11.02.

[5] Available as part of publication PB91–231480 from National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161.

TABLE 1 Chemical Service Registry Numbers, Retention Times, and Estimated Method Detection Limits for Forty-Five Pesticides

Analyte	CAS No.	Retention Time, min		Estimated MDL, µg/L
		Primary [A]	Confirmation [A]	
Alachlor	15972-60-8	35.96	34.1	0.38
Ametryn	834-12-8	36.0	34.52	2.0
Atraton	111-44-4	31.26	29.97	0.6
Atrazine	1912-24-9	31.77	31.23	0.13
Bromacil	314-40-9	37.22	40.0	2.5
Butachlor	23184-66-9	41.45	39.0	0.38
Butylate	2008-41-5	22.47	18.47	0.15
Carboxin	5234-68-5	42.77	42.05	0.6
Chlorpropham	101-21-3	29.09	... [B]	0.5
Cycloate	1134-23-2	28.58	29.67	0.25
Diazinon	333-41-5	33.23	... [B]	0.25
Dichlorvox	62-73-7	16.54	15.35	2.5
Diphenamid	957-51-7	38.87	37.97	0.6
Disulfoton	298-04-4	33.42	30.9	0.3
Disulfoton sulfone	2497-06-5	41.31	42.42	3.8
Disulfoton sulfoxide [C]	2497-07-6	19.08	... [B]	0.38
EPTC	563-12-2	20.07	16.57	0.25
Ethoprop	13194-48-4	28.58	26.42	0.19
Fenamiphos	22224-92-6	41.78	41.0	1.0
Fenarimol	60168-88-9	51.32	50.02	0.38
Fluridone	59756-60-4	56.68	59.07	3.8
Hexazinone	51235-04-2	46.58	47.8	0.76
Merphos [D]	150-50-5	42.35	39.28	0.25
Methyl paraoxon	950-35-6	35.58	34.1	2.5
Metolachlor	51218-45-2	37.74	35.7	0.75
Metribuzin	21087-64-9	35.2	34.73	0.15
Mevinphos	7786-34-7	22.51	21.92	5.0
MGK-264 [E]	113-48-4	38.73	36.73	0.5
Molinate	2212-67-1	25.66	22.47	0.15
Napropamide	15299-99-7	41.83	... [B]	0.25
Norflurazon	27314-13-2	45.92	47.58	0.5
Pebulate	1114-71-2	23.41	19.73	0.13
Prometon [C]	1610-18-0	31.58	30.0	0.3
Pronamide [C]	23950-58-5	32.76	32.63	0.76
Propazine	139-40-2	32.01	31.13	0.13
Simazine	122-34-9	31.49	31.32	0.075
Simetryn	1014-70-6	35.72	34.55	0.25
Stirofos	22248-79-9	41.27	39.65	0.76
Tebuthiuron	34014-18-1	25.15	42.77	1.3
Terbacil	5902-51-2	33.79	... [B]	4.5
Terbufos [C]	13071-79-9	32.57	... [B]	0.5
Terbutryn	886-50-0	36.80	34.8	0.25
Triademefon	43121-43-3	38.12	37.0	0.65
Tricyclazole	41814-78-2	42.25	44.33	1.0
Vernolate	1929-77-7	22.94	19.25	0.13

[A] See 7.13.2 and 7.13.3 for column description and operating conditions.
[B] Data not available.
[C] Compound shows instability in aqueous solutions.
[D] Merphos is converted to S,S,S-tributylphosphorotrithioate (DEF) in the hot GC injection port; DEF is actually detected using the mothod conditions.
[E] MGK-264 gives 2 peaks; peak identified in this table was used for quantification.

3.2.3 *laboratory duplicates (LD1 and LD2)*—two sample aliquots taken in the analytical laboratory and analyzed separately with identical procedures.

3.2.3.1 *Discussion*—Analyses of LD1 and LD2 give a measure of the precision with laboratory procedures, but not with sample collection, preservation, or storage procedures.

3.2.4 *field duplicates (FD1 and FD2)*—two separate samples collected at the same time and placed under identical circumstances and treated exactly the same throughout field and laboratory procedures.

3.2.4.1 *Discussion*—Analyses of FD1 and FD2 give a measure of the precision associated with sample collection, preservation, and storage, as well as with laboratory procedures.

3.2.5 *laboratory reagent blank (LRB)*—an aliquot of water that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples.

3.2.5.1 *Discussion*—The LRB is used to determine if test method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

3.2.6 *field reagent blank (FRB)*—water transferred in a bottle from the laboratory and poured at the field site into a sample container in the field and treated as a sample in all respects, including exposure to sampling site conditions, storage, preservation, and all analytical procedures.

3.2.6.1 *Discussion*—The purpose of the FRB is to determine if test method analytes or other interferences are present in the field environment.

3.2.7 *laboratory performance check solution (LPC)*—a solution of method analytes, surrogate compounds, and internal standards used to evaluate the performance of the instrument system with respect to a defined set of test method criteria.

3.2.8 *laboratory fortified blank (LFB)*—an aliquot of water to which known quantities of the test method analytes are added in the laboratory.

3.2.8.1 *Discussion*—The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements at the required method detection limit.

3.2.9 *laboratory fortified sample matrix (LFM)*—an aliquot of an environmental sample to which known quantities of the test method analytes are added in the laboratory.

3.2.9.1 *Discussion*—The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.

3.2.10 *calibration standard (CAL)*—a solution prepared from the primary dilution standard solution and stock standard solutions of the internal standards and surrogate analytes.

3.2.10.1 *Discussion*—The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

3.2.11 *quality control sample (QCS)*—a sample matrix containing test method analytes or a solution of test method analytes in a water-miscible solvent which is used to fortify water or environmental samples.

3.2.11.1 *Discussion*—The QCS is obtained from a source external to the laboratory, and is used to check laboratory performance with externally prepared test materials.

4. Summary of Test Method

4.1 A measured volume of sample of approximately 1 L is extracted with methylene chloride by shaking in a separatory funnel or mechanical tumbling in a bottle. The methylene chloride extract is isolated, water is removed, and concentrated to a volume of 5 mL during a solvent exchange to methyl tert-butyl ether (MTBE). Chromatographic conditions are described that permit the separation and measurement of the analytes in the extract by capillary column GC with a nitrogen-phosphorus detector (NPD).

4.2 This test method is based largely on USEPA Method 507.

5. Significance and Use

5.1 Nitrogen- and phosphorus-containing compounds are widely used in agriculture as pre-emergent agents to increase crop yields. Runoff from farmlands into lakes and streams as well as accidental discharge from irrigation systems into groundwater introduces these compounds into the environment. This discharge from agricultural areas along with possible health implications dictates a need to monitor the presence of these compounds.

6. Interferences

6.1 Test method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baselines in gas chromatograms. All reagents and apparatus must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in 12.2.

6.1.1 Glassware must be scrupulously cleaned as described in Practice D 3694. Clean all glassware as soon as possible after use by thoroughly rinsing with the last solvent used in it. Follow by washing with hot water and detergent and thorough rinsing with tap and reagent water. Drain dry, and heat in an oven or muffle furnace at 400°C for 1 h. Do not heat volumetric ware. Thermally stable materials might not be eliminated by this treatment. Thorough rinsing with acetone may be substituted for the heating. After drying and cooling, seal and store glassware in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

6.1.2 The use of high-purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required. (**Warning—** When a solvent is purified, stabilizers added by the manufacturer may be removed thus potentially making the solvent hazardous. Also, when a solvent is purified, preservatives added by the manufacturer are removed thus potentially reducing the shelf life.)

6.2 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. Between-sample rinsing of the sample syringe and associated equipment with MTBE can minimize sample cross contamination. After analysis of a sample containing high concentrations of analytes, one or more injections of MTBE should be made to ensure that accurate values are obtained for the next sample.

6.3 Matrix interferences may be caused by contaminants that are coextracted from the sample. Also, note that all the analytes listed in the scope and application section are not resolved from each other on any one column, that is, one analyte of interest may be an interferent for another analyte of interest. The extent of matrix interferences will vary considerably from source to source, depending upon the water sampled. Further processing of sample extracts may be necessary. Positive identifications should be confirmed (see 13.5).

6.4 It is important that samples and working standards be contained in the same solvent. The solvent for working standards must be the same as the final solvent used in sample preparation. If this is not the case, chromatographic comparability of standards to sample may be affected.

7. Apparatus and Equipment

7.1 *Sample Bottles*—Borosilicate, 1-L volume with graduations, fitted with screw caps lined with TFE-fluorocarbon. Protect samples from light. The container must be washed and

dried as described in 6.1.1 before use to minimize contamination. Cap liners are cut to fit from sheets and extracted with methanol overnight prior to use.

7.2 *Separatory Funnel*, 2000-mL, with TFE-fluorocarbon stopcock, ground glass, or TFE-fluorocarbon stopper.

7.3 *Tumbler Bottle*, 1.7-L, with TFE-fluorocarbon-lined screw cap. Cap liners are cut to fit from sheets and extracted with methanol overnight prior to use.

7.4 *Concentrator Tube*, Kuderna-Danish (K-D), 10 or 25-mL, graduated. Calibration must be checked at the volumes employed in the test. Ground glass stoppers are used to prevent evaporation of extracts.

7.5 *Evaporation Flask*, K-D, 500-mL. Attach to concentrator tube with springs.

7.6 *Snyder Column*, K-D, three-ball macro.

7.7 *Snyder Column*, K-D, two-ball micro.

7.8 *Vials*, glass, 5 to 10-mL capacity with TFE-fluorocarbon-lined screw cap.

7.9 *Separatory Funnel Shaker*, (optional), capable of holding 2-L separatory funnels and shaking them with rocking motion to achieve thorough mixing of separatory funnel contents.

7.10 *Tumbler*, capable of holding tumbler bottles and tumbling them end-over-end at 30 turns/min.

7.11 *Boiling Stones*, carborundum, No. 12 granules. Heat at 400°C for 30 min prior to use. Cool and store in desiccator.

7.12 *Water Bath*, heated, capable of temperature control (±2°C). The bath should be used in a well-ventilated hood.

7.13 *Gas Chromatograph*, analytical system complete with temperature programmable GC suitable for use with capillary columns and all required accessories including syringes, analytical columns, gases, detector, and stripchart recorder. A data system is recommended for measuring peak areas. Table 2 lists retention times observed for most of the method analytes using the columns and analytical conditions described below.

7.13.1 *Column 1 (Primary Column)*, 30 m long by 0.25-mm inside diameter (ID) by 0.25-µm film thickness, 95 % cross-bonded dimethyl-5 % diphenyl polysiloxane fused silica column. Helium carrier gas flow is established at 30- cm/s linear velocity and oven temperature is programmed from 60 to 300°C at 4°C/min. Data presented in this test method were obtained using this column. The injection volume was 2 µL in splitless mode with a 45-s delay. The injector temperature was 250°C and the detector temperature was 300°C. Alternative columns may be used in accordance with the provisions described in 12.4

7.13.2 *Column (Confirmation Column)*, 30 m long by 0.25-mm ID by 0.25-µm film thickness cross-bonded 14 % cyanopropylphenyl-86 % methyl polysiloxane fused silica column. Helium carrier gas flow is established at 30-cm/s linear velocity and oven temperature is programmed from 60 to 300°C at 4°C/min.

7.13.3 *Detector, Nitrogen-Phosphorus (NPD)*—An NPD was used to generate the validation data presented in this test method. Alternative detectors may be used in accordance with the provisions described in 12.4

TABLE 2 Acceptance Limits (as Percent of Mean Recovery) for Analysis of Laboratory Quality Control Sample

Analyte	Concentration Level [A]	Mean Recovery [B]	Overall Std Dev	Acceptance Limits [C]
Alachlor	5.00	4.63	0.78	49.5–150
Ametryn	2.00	1.88	0.28	55.3–145
Atraton	5.00	4.72	0.87	44.9–155
Atrazine	2.00	1.86	0.27	55.9–144
Bromacil	10.0	9.55	1.60	49.7–151
Butachlor	10.0	9.41	1.46	53.2–147
Butylate	5.00	3.81	0.89	29.9–170
Carboxin	10.0	9.37	2.00	35.0–164
Chlorpropham	10.0	9.76	1.78	45.3–155
Cycloate	5.00	4.29	0.84	41.2–159
Diazinon	2.00	1.78	0.34	42.7–157
Dichlorvos	5.00	4.84	0.76	53.1–147
Diphenamid	5.00	4.72	0.75	52.3–148
Disulfoton	2.00	1.73	0.33	42.9–157
Disulfoton sulfone	10.0	10.4	2.26	34.8–165
Disulfoton sulfoxide	10.0	9.51	1.86	41.3–159
EPTC	2.00	1.72	0.32	44.2–156
Ethoprop	2.00	1.84	0.37	39.7–160
Fenamiphos	20.0	18.0	3.50	41.7–158
Fenarimol	5.00	4.86	1.09	32.7–167
Fluridone	10.0	10.2	2.47	27.4–172
Hexazinone	5.00	4.96	1.06	36.1–164
Merphos	... [D]	...	...	...
Methyl paraoxon	10.0	10.0	1.73	48.1–152
Metolachlor	10.0	9.26	1.22	60.5–139
Metribuzin	2.00	1.94	0.38	41.2–159
Mevinphos	10.0	9.70	1.28	60.4–139
MGK-264	10.0	9.23	1.66	46.0–154
Molinate	2.00	1.88	0.34	45.7–154
Napropamide	5.00	4.37	0.69	52.6–147
Norflurazon	5.00	4.80	1.19	25.4–174
Pebulate	2.00	1.78	0.39	34.3–166
Prometon	2.00	1.92	0.33	48.4–152
Pronamide	10.0	9.72	1.47	54.5–145
Propazine	2.00	1.86	0.32	48.4–152
Simazine	2.00	1.90	0.28	55.8–144
Simetryn	2.00	1.93	0.34	47.2–153
Stirofos	20.0	18.6	3.87	37.5–162
Tebuthiuron	10.0	9.72	1.79	44.6–155
Terbacil	50.0	49.8	9.35	43.8–156
Terbufos	10.0	8.30	1.50	45.8–154
Terbutryn	2.00	1.90	0.24	52.1–138
Triademefon	2.00	1.96	0.40	38.8–161
Tricyclazole	20.0	18.1	2.95	51.1–149
Vernolate	2.00	1.62	0.41	24.1–176

[A] Concentration level is 10 to15 times the estimated MDL, µg/L.
[B] Calculated from the regression equations for mean recovery and overall standard deviation obtained in collaborative study of the method for reagent water matrix.
[C] Acceptance limits are defined as the mean recovery ±3 standard deviations.
[D] Merphos breakdown to DEF was incomplete and resulted in poor recovery and precision.

8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society

where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.[6] For trace analysis using organic solvents for liquid-liquid extraction, solvents specified as distilled-in-glass, nano-grade, or pesticide-grade frequently have lower levels of interfering impurities. In all cases, sufficient reagent blanks must be processed with the samples to ensure that all compounds of interest are not present as blanks due to reagents or glassware.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193, Type II, and shown to contain no interfering contaminants at concentrations sufficient to interfere with the analysis.

8.3 *Acetone*, Distilled-in-glass quality or equivalent.

8.4 *Mercuric Chloride*, for use as a bactericide. If any other bactericide can be shown to work as well as mercuric chloride, it may be used.

8.5 *Methylene Chloride*, Distilled-in-glass quality or equivalent.

8.6 *Methyl Tert-Butyl Ether (MTBE)*, Distilled-in-glass quality or equivalent.

8.7 *Phosphate Buffer, pH 7*—Prepare by mixing 29.6 mL 0.1 N HCl and 50 mL 0.1 M dipotassium phosphate.

8.8 *Sodium Chloride (NaCl), crystal*—Heat in a shallow tray at 450°C for a minimum of 4 h to remove interfering organic substances.

8.9 *Sodium Sulfate*, granular, anhydrous. Heat in a shallow tray at 450°C for a minimum of 4 h to remove interfering organic substances.

8.10 *Sodium Thiosulfate*, granular, anhydrous.

8.11 *1,3-dimethyl-2-nitrobenzene*, 98 % purity, for use as surrogate standard.

8.12 *Standard Solution, Stock* (1.00 µg/µL). Stock standard solutions may be purchased as certified solutions or prepared from pure standard materials using the following procedure:

8.12.1 Prepare stock standard solutions by accurately weighing 0.0100 g of pure material. Dissolve the material in MTBE and dilute to volume in a 10-mL volumetric flask. The stock solution for simazine should be prepared in methanol. Larger volumes may be prepared at the convenience of the analyst. If compound purity is certified at 96 % or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

8.12.2 Transfer the stock standard solutions into TFE-fluorocarbon sealed screw-cap amber vials. Store at room temperature and protect from light.

8.12.3 Stock standard solutions should be replaced after 2 months or sooner if comparison with laboratory fortified blanks or QC samples indicate a problem.

8.13 *Internal Standard Solution*—Prepare an internal standard solution by accurately weighing 0.0500 g of pure triphenylphosphate (TPP). Dissolve the TPP in MTBE and dilute to volume in a 100-mL volumetric flask. Transfer the internal standard solution to a TFE-fluorocarbon sealed screw-cap bottle and store at room temperature. Addition of 50 µL of the internal standard solution to 5 mL of sample extract results in a final TPP concentration of 5.0µ g/mL. Solution should be replaced when ongoing quality control (QC) (Section 12) indicates a problem. Note that TPP has been shown to be an effective internal standard for the test method analytes, [2] but other compounds may be used if the QC requirements in Section 12 are met.

8.14 *Surrogate Standard Solution*—Prepare a surrogate standard solution by accurately weighing 0.0250 g of pure 1,3-dimethyl-2-nitrobenzene. Dissolve the 1,3-dimethyl-2-nitrobenzene in MTBE and dilute to volume in a 100-mL volumetric flask. Transfer the surrogate standard solution to a TFE-fluorocarbon sealed screw-cap bottle and store at room temperature. Addition of 50 µL of the surrogate standard solution to a 1-L sample prior to extraction results in a surrogate standard concentration in the sample of 5 µg/L and, assuming quantitative recovery of 1,3-dimethyl-2-nitrobenzene, a surrogate standard concentration in the final extract of 12.5 µg/mL. Solution should be replaced when ongoing QC (Section 12) indicates a problem. The 1,3-dimethyl-2-nitrobenzene has been shown to be an effective surrogate standard for the test method analytes, [2] but other compounds may be used if the QC requirements in Section 12 are met.

8.15 *Laboratory Performance Check Solution*—Prepare the laboratory performance check solution by adding 5 µL of the vernolate stock solution, 0.5 mL of the bromacil stock solution, 30 µL of the prometon stock solution, 15 µL of the atrazine stock solution, 1.0 mL of the surrogate solution, and 500 µL of the internal standard solution to a 100-mL volumetric flask. Dilute to volume with MTBE and thoroughly mix the solution. Transfer to a TFE-fluorocarbon-sealed screw-cap bottle and store at room temperature. Solution should be replaced when ongoing QC (Section 12) indicates a problem.

9. Sample Collection, Preservation, and Handling

9.1 Collect the samples in accordance with Practices D 3370, D 3694, and Specification D 1192.

9.2 *Instructions Specific to This Test Method*—Grab samples must be collected in glass containers. Conventional sampling Practices D 3370 should be followed; however, the bottle must not be prerinsed with sample before collection.

10. Sample Preservation and Storage

10.1 Add mercuric chloride to the sample bottle in amounts to produce a concentration of 10 mg/L. Add 1 mL of a solution containing 10 mg/mL of mercuric chloride in water to the sample bottle at the sampling site or in the laboratory before shipping to the sampling site. (**Warning—**A major disadvantage of mercuric chloride is that it is a highly toxic chemical;

[6] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

mercuric chloride must be handled with caution, and samples containing mercuric chloride must be disposed of properly.)

10.2 If residual chlorine is present, add 80 mg of sodium thiosulfate per litre of sample to the sample bottle prior to collecting the sample.

10.3 After the sample is collected in a bottle containing preservative(s), seal the bottle and shake vigorously for 1 min.

10.4 Ice or refrigerate the samples at 4°C away from light from the time of collection until extraction. Preservation study results indicated that most test method analytes present in samples were stable for 14 days when stored under these conditions. [2] The analytes disulfoton sulfoxide, diazinon, pronamide, and terbufos exhibited significant aqueous instability, and samples to be analyzed for these compounds must be extracted immediately. The analytes carboxin, EPTC, fluridone, metolachlor, napropamide, tebuthiuron, and terbacil exhibited recoveries of less than 60 % after 14 days. Analyte stability may be affected by the matrix; therefore, the analyst should verify that the preservation technique is applicable to the samples under study.

10.5 *Extract Storage*—Store extracts at 4°C away from the light. Preservation study results indicate that most analytes are stable for 28 days; however, a 14 day maximum extract storage time is recommended. The analyst should verify appropriate extract holding times applicable to the samples under study.

11. Calibration

11.1 Establish GC operating parameters equivalent to those indicated in 7.13. Consult the manufacturer's operation manual for the GC system, if necessary. Calibrate the GC system using either the internal standard technique (11.2) or the external standard technique (11.3). Be aware that NPDs may exhibit instability (that is, fail to hold calibration curves over time). The analyst may, when analyzing samples for target analytes that are rarely found, prefer to analyze on a daily basis a low level (for example, 5 to 10 times detection limit), sample (containing all analytes of interest) and require some minimum sensitivity (for example, ½ full-scale deflection) to show that if the analyte were present it would be detected. The analyst may then quantitate using single-point calibration (11.2.5 or 11.3.4).

11.1.1 Calibration standard solutions must be prepared such that no unresolved analysts are mixed together.

11.2 *Internal Standard Calibration Procedure*—To use this approach, the analyst must select one or more internal standards compatible in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Triphenylphosphate has been identified as a suitable internal standard.

11.2.1 Prepare calibration standards at a minimum of three (recommend five) concentration levels for each analyte of interest by adding volumes of one or more stock standards to a volumetric flask. If Merphos is to be determined, calibrate with DEF (S,S,S-tributylphosphorus-trithioate). To each calibration standard, add a known constant amount of one or more of the internal standards and dilute to volume with MTBE. The standards should bracket the analyte concentrations expected in the sample extracts, or should define the working range of the detector.

11.2.2 Analyze each calibration standard according to the procedure (see 13.4). Tabulate response (peak height or area) against concentration for each compound and internal standard. Calculate the response factor (RF) for each analyte and surrogate using Eq 1.

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)} \quad (1)$$

where:

A_s = response for the analyte to be measured,
A_{is} = response for the internal standard,
C_{is} = concentration of the internal standard, µg/L, and
C_s = concentration of the analyte to be measured, µg/L.

11.2.3 If the RF value over the working range is constant (20 % RSD or less) use the average RF for calculations. Alternatively, use the results to plot a calibration curve of response ratios (A_s/A_{is}) versus C_s.

11.2.4 Verify the working calibration curve or RF on each working shift by the measurement of one or more calibration standards. If the response for any analyte of interest varies from the predicted response by more than ± 20 %, repeat the test using a fresh calibration standard. If the repetition also fails, generate a new calibration curve for that analyte using freshly prepared standards.

11.2.5 Single-point calibration is a viable alternative to a calibration curve. Prepare single point standards from the secondary dilution standards in MTBE. Prepare the single point standards at a concentration that produces a response that deviates from the sample extract response by no more than 20 %.

11.2.6 Verify calibration standards periodically, at least quarterly, by analyzing a standard prepared from reference material obtained from an independent source. Results from these analyses must be within the limits used to routinely check calibration.

11.3 *External Standard Calibration Procedure*:

11.3.1 Prepare calibration standards at a minimum of three (recommend five) concentration levels for each analyte of interest and surrogate compound by adding volumes of one or more stock standards and 250 µL methanol to a volumetric flask. If Merphos is to be determined, calibrate with DEF (S,S,S-tributylphosphorus-trithioate). Dilute to volume with MTBE. The standards should bracket the analyte concentrations expected in the sample extracts, or should define the working range of the detector.

11.3.2 Starting with the standard of lowest concentration, analyze each calibration standard in accordance with 13.4 and tabulate response (peak height or area) versus the concentration curve for each compound. Alternatively, if the ratio of response to concentration (calibration factor) is a constant over the working range (20 % RSD or less), assume linearity through the origin and use the average ratio or calibration factor in place of a calibration curve.

11.3.3 Verify the working calibration curve or calibration factor on each working day by the measurement of a minimum of two calibration check standards, one at the beginning and one at the end of the analysis day. These check standards should be at two different concentration levels to verify the calibration curve. For extended periods of analysis (greater

than 8 h), it is strongly recommended that check standards be interspersed with samples at regular intervals during the course of the analyses. If the response varies by more than ± 20 %, repeat the test using a fresh calibration standard. if the results still do not agree, generate a new calibration curve.

11.3.4 Single-point calibration is a viable alternative to a calibration curve. Prepare single point standards from the secondary dilution standards in MTBE. Prepare the single point standards at a concentration that produces a response that deviates from the sample extract response by no more than 20 %.

11.3.5 Verify calibration standards periodically, at least quarterly, by analyzing a standard prepared from reference material obtained from an independent source. Results from these analyses must be within the limits used to routinely check calibration.

12. Quality Control

12.1 Recommended quality control (QC) requirements are initial demonstration of laboratory capability, determination of surrogate compound recoveries in each sample and blank, monitoring internal standard peak area or height in each sample and blank (when internal standard calibration procedures are being employed), analysis of laboratory reagent blanks, laboratory fortified samples, laboratory fortified blanks, and QC samples.

12.2 *Laboratory Reagent Blanks (LRB)*—Before processing any samples, the analyst must demonstrate that all glassware and reagent interferences are under control. Each time a set of samples is extracted or reagents are changed, an LRB must be analyzed. If within the retention time window of any analyte the LRB produces a peak that would prevent the determination of that analyte, determine the source of contamination and eliminate the interference before processing samples.

12.3 *Initial Demonstration of Capability*:

12.3.1 Demonstrate initial method performance by extracting four 1-L samples of laboratory fortified blank (LFB) water at the concentration levels indicated in Table 2. Analyze each aliquot in accordance with the procedures beginning in Section 13.

12.3.2 For acceptable performance, each analyte mean recovery should be within the acceptance limits in Table 2, and their relative standard deviation should be <20 %.

12.3.3 The initial demonstration of capability is used primarily to preclude a laboratory from analyzing unknown samples by means of a new, unfamiliar test method prior to obtaining some experience with it. It is expected that as laboratory personnel gain experience with this test method the quality of data will improve beyond those required here.

12.4 The analyst is permitted to modify GC columns, GC conditions, detectors, continuous extraction techniques, concentration techniques (that is, evaporation techniques), internal standard or surrogate compounds. Each time such test method modifications are made, the analyst must repeat the procedures in 12.3.

12.5 *Assessing Surrogate Recovery*:

12.5.1 When surrogate recovery from a sample or method blank is < 70 % or > 130 %, check the following: calculations to locate possible errors; spiking solutions for degradation; contamination; and instrument performance. If those steps do not reveal the cause of the problem, reanalyze the extract.

12.5.2 If sample extract reanalysis meets the surrogate recovery criterion, report only data for the analyzed extract. If sample extract continues to fail the recovery criterion, report all data for that sample as suspect.

12.6 *Assessing the Internal Standard (IS)*:

12.6.1 When using the internal standard calibration procedure, the analyst is expected to monitor the IS response (peak area or peak height) of all samples during each analysis day. The IS response for any sample chromatogram should not deviate from the daily calibration check standard's IS response by more than 30 %.

12.6.2 If >30 % deviation occurs with an individual extract, optimize instrument performance and inject a second aliquot of that extract.

12.6.2.1 If the reinjected aliquot produces an acceptable internal standard response, report results for that aliquot.

12.6.2.2 If a deviation of greater than 30 % is obtained for the reinjected extract, repeat analysis of the samples beginning with Section 13, provided the sample is still available. Otherwise, report results obtained from the reinjected extract, but annotate as suspect.

12.6.3 If consecutive samples fail the IS response acceptance criterion, immediately analyze a calibration check standard.

12.6.3.1 If the check standard provides a response factor (RF) within 20 % of the predicted value, follow procedures itemized in 12.6.2 for each sample failing the IS response criterion.

12.6.3.2 If the check standard provides a response factor that deviates more than 20 % of the predicted value, recalibrate as specified in Section 11.

12.7 *Assessing Laboratory Performance—Laboratory Fortified Blank*:

12.7.1 The laboratory must analyze at least one laboratory fortified blank (LFB) sample with every 20 samples or one per sample set (all samples extracted within a 24-h period), whichever is greater. Calculate accuracy as percent recovery (X). If the recovery of any analyte falls outside the control limits (Table 2), that analyte is judged out of control, and identify the source of the problem and resolve before continuing analyses.

12.7.2 Until sufficient data become available from within the analyst's own laboratory, usually a minimum of results from 20 to 30 analyses, the laboratory should assess laboratory performance against the control limits in 12.3.2 that are derived from the data in Table 2. When sufficient internal performance data becomes available, develop control limits from the mean percent recovery (X) and standard deviation (S) of the percent recovery. Use these data to establish upper and lower control limits as follows:

$$\text{Upper Control Limit} = X + 3S$$
$$\text{Lower Control Limit} = X - 3S$$

After each five to ten new recovery measurements, calculate new control limits using only the most recent 20 to 30 data points. These calculated control limits should never exceed those established in 12.3.2.

12.7.3 It is recommended that the laboratory periodically determine and document its detection limit capabilities for the analytes of interest.

12.7.4 At least quarterly, analyze a QC sample from an outside source.

12.7.5 Laboratories are encouraged to participate in external performance evaluation studies such as the laboratory certification programs offered by many states or the studies conducted by the USEPA. Performance evaluation studies serve as independent checks on the analyst's performance.

12.8 *Assessing Analyte Recovery—Laboratory Fortified Sample Matrix*:

12.8.1 The laboratory must add a known concentration to a minimum of 10 % of the routine samples or one sample concentration per set, whichever is greater. The concentration should not be less than the background concentration of the sample selected for fortification. Ideally, the concentration should be the same as that used for the laboratory fortified blank (12.7). Over time, samples from all routine sample sources should be fortified.

12.8.2 Calculate the percent recovery, *R*, of the concentration for each analyte using Eq 2.

$$R = \frac{C_{LFM} - C_{MS}}{C_A} \times 100 \quad (2)$$

where:

C_{LFM} = concentration measured in laboratory fortified matrix sample, µg/L,

C_{MS} = concentration measured in unfortified sample, µg/L, and

C_A = concentration added to LFM, µg/L.

Compare these values to the control limits established in 12.3.2.

12.8.3 If the recovery of any such analyte falls outside the designated range, and the laboratory performance for that analyte is shown to be in control (12.7), the recovery problem encountered with the fortified sample is judged to be matrix related, not system related. The results for that analyte in the unfortified sample is labeled *suspect/matrix* to inform that data user that the results are suspect due to matrix effects.

12.9 *Assessing Instrument System—Laboratory Performance Check Sample*—Monitor instrument performance on a daily basis by analysis of the LPC sample. The LPC sample contains compounds designed to indicate appropriate instrument sensitivity, column performance (primary column), and chromatographic performance. The LPC sample components and performance criteria are listed in Table 3. Inability to demonstrate acceptable instrument performance indicates the need for reevaluation of the instrument system.

12.10 The laboratory may adopt additional quality control practices for use with this test method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. For example, field or laboratory duplicates may be analyzed to assess the precision of the environmental measurements or field reagent blanks may be used to assess contamination of samples under site conditions, transportation, and storage.

TABLE 3 Laboratory Performance Check Solution

Test	Analyte	Conc. µg/mL	Requirements
Sensitivity	Vernolate	0.05	Detection of analyte; S/N > 3
Chromatographic performance	Bromacil	5	0.80 < PGF < 1.20[A]
Column performance	Prometon, Atrazine	0.3, 0.15	Resolution > 0.7[B]

[A] PGF—peak Gaussian factor. Calculated using the equation:

$$PGF = \frac{0.83 \times W(1/2)}{W(1/10)}$$

where W (½) is the peak width at half height and W (1/10) is the peak width at tenth height.

[B] Resolution between the two peaks as defined by the equation:

$$R = \frac{t}{W}$$

where t is the difference in elution times between the two peaks and W is the average peak width, at the baseline, of the two peaks.

13. Procedure

13.1 *Extraction (Manual Test Method)*:

13.1.1 Mark the water meniscus on the side of the sample bottle containing about 1 L of sample for later determination of sample volume (13.1.6). Add preservative to blanks and QC check standards. Fortify the sample with 50 µL of the surrogate standard solution. Pour the entire sample into a 2-L separatory funnel.

13.1.2 Adjust the sample to pH 7 by adding 50 mL of phosphate buffer.

13.1.3 Add 100 g NaCl to the sample, and shake to dissolve salt.

13.1.4 Add 60 mL methylene chloride to the sample bottle, and shake 30 s to rinse the inner walls. Transfer the solvent to the separatory funnel and extract the sample by vigorously shaking the funnel for 2 min with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one third the volume of the solvent layer, employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the methylene chloride extract in a 500-mL Erlenmeyer flask.

13.1.5 Add a second 60-mL volume of methylene chloride to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

13.1.6 Determine the original sample volume by refilling the sample bottle to the mark and transferring the water to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

13.2 *Extraction (Automated Test Method)*—Data presented in this test method were generated using the automated extraction procedure with the mechanical tumbler.

13.2.1 Mark the water meniscus on the side of the sample bottle for later determination of sample volume (13.1.6). Add preservative to blanks and QC check standards. Fortify the sample with 50 µL of the surrogate standard solution. If the mechanical separatory funnel shaker is used, pour the entire

sample into a 2-L separatory funnel. If the mechanical tumbler is used, pour the entire sample into a tumbler bottle.

13.2.2 Adjust the sample to pH 7 by adding 50 mL of phosphate buffer.

13.2.3 Add 100 g NaCl to the sample, and shake to dissolve salt.

13.2.4 Add 300 mL methylene chloride to the sample bottle, and shake 30 s to rinse the inner walls. Transfer the solvent to the sample contained in the separatory funnel or tumbler bottle, and shake for 10 s, venting periodically. Repeat shaking and venting until pressure release is not observed. Place the sample container in appropriate mechanical mixing device (separatory funnel shaker or tumbler). Shake or tumble the sample for 1 h. Complete mixing of the organic and aqueous phases should be observed within about 2 min after starting the mixing device.

13.2.5 Remove the sample container from the mixing device. If the tumbler is used, pour contents of tumbler bottle into a 2-L separatory funnel. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one third the volume of the solvent layer, employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration through glass wool, centrifugation, or other physical methods. Collect the methylene chloride extract in a 500-mL Erlenmeyer flask.

13.2.6 Determine the original sample volume by refilling the sample bottle to the mark and transferring the water to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

13.3 *Extract Concentration*:

13.3.1 Assemble a K-D concentrator by attaching a 25-mL concentrator tube to a 500-mL evaporative flask. Other concentration devices or techniques may be used in place of the K-D if the requirements of 12.3 are met.

13.3.2 Dry the extract by pouring it through a solvent-rinsed drying column containing about 10 cm of anhydrous sodium sulfate. Collect the extract in the K-D concentrator, and rinse the column with 20 to 30 mL methylene chloride. Alternatively, add about 5 g anhydrous sodium sulfate to the extract in the Erlenmeyer flask; swirl flask to dry extract and allow to sit for 15 min. Decant the methylene chloride extract into the K-D concentrator. Rinse the remaining sodium sulfate with two 25-mL portions of methylene chloride and decant the rinses into the K-D concentrator.

13.3.3 Add to 1 to 2 clean boiling stones to the evaporative flask and attach a macro-Snyder column. Prewet the Snyder column by adding about 1 mL methylene chloride to the top. Place the K-D apparatus on a hot water bath, 60 to 70°C, so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 min. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 2 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 min.

13.3.4 Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of MTBE. Add 5 to 10 mL of MTBE and a fresh boiling stone. Attach a micro-Snyder column to the concentrator tube and prewet the column by adding about 0.5 mL of MTBE to the top. Place the micro K-D apparatus on the water bath so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete concentration in 5 to 10 min. When the apparent volume of liquid reaches 2 mL, remove the micro K-D from the bath and allow it to drain and cool. Add 5 to 10 mL MTBE to the micro K-D and reconcentrate to 2 mL. Remove the micro K-D from the bath and allow it to drain and cool. Remove the micro-Snyder column, and rinse the walls of the concentrator tube while adjusting the volume to 5.0 mL with MTBE.

NOTE 1—**Caution:** If methylene chloride is not completely removed from the final extract, it may cause detector problems.

13.3.5 Transfer extract to an appropriate-sized TFE-fluorocarbon sealed screw-cap vial and store, refrigerated at 4°C, until analysis by GC-NPD.

13.4 *Gas Chromatography*:

13.4.1 Paragraph 7.13 summarizes the recommended operating conditions for the gas chromatograph. Included in Table 2 are retention times observed using this test method. Other GC columns, chromatographic conditions, or detectors may be used if the requirements of 12.3 are met.

13.4.2 Calibrate the system daily as described in Section 11. The standards and extracts must be in MTBE.

13.4.3 If the internal standard calibration procedure is used, add 50µ L of the internal standard solution to the sample extract, and shake to distribute the internal standard.

13.4.4 Inject 2 µL of the sample extract. Record the resulting peak size in area units.

13.4.5 If the response for the peak exceeds the working range of the system, dilute the extract and reanalyze.

13.5 *Identification of Analytes*:

13.5.1 Identify a sample component by comparison of its retention time to the retention time of a reference chromatogram. If the retention time of an unknown compound corresponds, within limits, to the retention time of a standard compound, then identification is considered positive.

13.5.2 Base the width of the retention time window used to make identifications upon measurements of actual retention time variations of standards over the course of a day. Use three times the standard deviation of a retention time to calculate a suggested window size for a compound. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

13.5.3 Identification requires expert judgement when sample components are not resolved chromatographically. When peaks obviously represent more than one sample component (that is, broadened peak with shoulder(s) or valley between two or more maxima), or any time doubt exists over the identification of a peak on a chromatogram, employ appropriate alternative techniques to help confirm peak identification. For example, more positive identification may be made by the use of an alternative detector that operates on a chemical/physical principle different from that originally used,

for example, mass spectrometry, or the use of a second chromatography column. A suggested alternative column is described in 7.8.

14. Calculation

14.1 Calculate analyte concentrations in the sample from the response for the analyte using the calibration procedure described in Section 11.

14.2 If the internal standard calibration procedure is used, calculate the concentration (C) in the sample using the response factor (RF) determined in 11.2 and Eq 3 or determine sample concentration from the calibration curve.

$$C, \mu g/L = \frac{(A_s)(I_s)}{(A_{is})(RF)(V_o)} \quad (3)$$

where:

A_s = response for the analyte to be measured,
A_{is} = response for the internal standard,
I_s = amount of internal standard added to each extract, µg, and
V_o = volume of water extracted, L.

14.3 If the external standard calibration procedure is used, calculate the amount of material injected from the peak response using the calibration curve or calibration factor determined in 11.3. The concentration (C) in the sample can be calculated from Eq 4.

$$C, \mu g/L = \frac{(A)(V_t)}{(V_i)(V_s)} \quad (4)$$

where:

A = amount of material injected, ng,
V_i = volume of extract injected, µL,
V_t = volume of total extract, µL, and
V_s = volume of water extracted, µ L.

15. Precision and Bias [7]

15.1 The collaborative studies for performance evaluation of this test method were conducted in accordance with Practice D 2777.

15.1.1 Ten laboratories participated in the study. Two samples, different in concentration of the 45 analytes, were analyzed as Youden pairs at each of 3 levels to provide data for estimating single-analyst precision. Precision and bias data is shown in Table 4. Analyses of the spiked reagent water evaluated the proficiency of the test method on a sample free of interferences. Analyses of the spiked finished drinking waters were intended to demonstrate the suitability of the test method on a regulated matrix and to compare the results with those of reagent water as shown in Table 5.

16. Keywords

16.1 drinking water; gas chromatography; nitrogen- and phosphorous-containing pesticides

[7] Supporting data for the precision and bias statements are available from ASTM Headquarters. Request RR:D 19–1152.

TABLE 4 Collaborative Study Data Sets

Analyte	Reagent Water					Finished Drinking Water			
	C [A]	X [B]	s_R [C]	s_r [D]	Regr. Equations	X	s_R	s_r	Regr. Equations
Alachlor	1.50	1.48	0.40	0.46	X = 0.912C + 0.072	1.66	0.61	0.28	X = 0.902C + 0.274
	2.24	2.05	0.30		s_R = 0.138X + 0.142	2.25	0.59		s_R = 0.155X + 0.308
	5.98	5.33	1.09	0.70	s_r = 0.075X + 0.325	5.45	1.14	0.94	s_r = 0.139X + 0.013
	7.48	6.77	1.21			6.94	0.92		
	12.00	11.66	1.39	1.41		11.54	2.50	1.66	
	15.00	13.75	2.04			13.99	2.98		
Ametryn	0.60	0.60	0.13	0.09	X = 0.911C + 0.063	0.60	0.12	0.12	X = 0.863C + 0.071
	0.90	0.90	0.14		s_R = 0.130X + 0.040	0.82	0.14		s_R = 0.181X + 0.000
	2.40	2.18	0.13	0.47	s_r = 0.194X − 0.051	2.17	0.49	0.20	s_r = 0.073X + 0.063
	3.00	2.96	0.63			2.59	0.23		
	4.80	4.30	0.39	0.86		4.34	1.30	0.48	
	6.00	5.50	1.13			5.23	0.58		
Atraton	1.00	1.05	0.21	0.14	X = 0.922C + 0.107	1.01	0.17	0.12	X = 0.887C + 0.114
	1.50	1.46	0.26		s_R = 0.185X − 0.006	1.43	0.23		s_R = 0.127X + 0.038
	4.00	3.57	0.26	0.83	s_r = 0.216X − 0.130	3.59	0.56	0.33	s_r = 0.086X + 0.011
	5.00	4.92	1.19			4.47	0.19		
	8.00	7.35	0.94	1.64		7.35	1.51	0.77	
	10.00	9.66	2.69			9.15	1.13		
Atrazine	0.30	0.31	0.02	0.09	X = 0.911C + 0.036	0.42	0.17	0.04	X = 0.932C + 0.117
	0.45	0.45	0.14		s_R = 0.143X + 0.008	0.49	0.08		s_R = 0.112X + 0.091
	1.20	1.06	0.14	0.16	s_r = 0.089X + 0.057	1.21	0.39	0.22	s_r = 0.12
	1.50	1.40	0.18			1.52	0.25		
	2.40	2.31	0.30	0.30		2.35	0.21	0.10	
	3.00	2.77	0.39			3.08	0.37		
Bromacil	3.01	3.16	0.74	0.82	X = 0.885C + 0.702	3.18	1.04	0.41	X = 0.888C + 0.512
	4.51	5.18	1.09		s_R = 0.131X + 0.348	4.55	1.03		s_R = 0.136X + 0.558
	12.00	10.93	1.21	2.94	s_r = 0.149X + 0.241	11.14	2.77	1.30	s_r = 0.096X + 0.047
	15.00	14.66	3.76			13.70	1.64		
	24.10	21.42	2.79	2.60		21.37	4.46	2.32	
	30.10	26.50	2.82			28.10	3.37		
Butachlor	2.00	1.77	0.44	0.34	X = 0.950C − 0.088	2.07	0.43	0.33	X = 0.907C + 0.232
	3.00	2.88	0.67		s_R = 0.132X + 0.223	2.88	0.42		s_R = 0.166X + 0.044
	8.00	7.39	1.33	0.88	s_r = 0.126X + 0.034	7.66	1.31	1.17	s_r = 0.121X + 0.034
	10.00	9.38	1.04			9.37	2.07		

TABLE 4 *Continued*

Analyte	Reagent Water					Finished Drinking Water			
	C [A]	X [B]	s_R [C]	s_r [D]	Regr. Equations	X	s_R	s_r	Regr. Equations
	16.00	15.51	2.33	2.52		13.85	1.64	1.85	
	20.00	18.45	2.88			19.19	3.55		
Butylate	0.80	0.57	0.24	0.11	X = 0.769C − 0.034	0.60	0.10	0.12	X = 0.759C + 0.020
	1.19	0.90	0.28		s_R = 0.204X + 0.115	0.98	0.10		s_R = 0.182X − 0.026
	3.18	2.51	0.38	0.68	s_r = 0.203X − 0.028	2.46	0.59	0.60	s_r = 0.152X + 0.008
	3.98	2.83	1.04			3.02	0.45		
	6.37	5.12	1.01	0.80		4.92	0.62	0.54	
	7.96	5.88	1.39			5.82	1.25		
Carboxin	3.00	3.36	0.82	0.70	X = 0.870C + 0.674	2.67	1.26	0.48	X = 0.664C + 0.602
	4.51	4.44	0.93		s_R = 0.212X + 0.017	3.45	1.54		s_R = 0.386X + 0.287
	12.00	10.30	0.85	3.23	s_r = 0.242X − 0.223	8.01	4.60	1.24	s_r = 0.173X − 0.072
	15.00	14.95	4.36			11.00	4.48		
	24.00	20.80	3.27	5.02		15.21	6.65	3.75	
	30.00	27.54	8.19			22.83	6.00		
Chlorpropham	2.00	2.22	0.43	0.27	X = 0.944C + 0.322	1.85	0.89	0.72	X = 0.940C + 0.049
	2.99	3.13	0.71		s_R = 0.179X + 0.034	3.07	0.61		s_R = 0.144X + 0.510
	7.98	7.48	0.29	1.79	s_r = 0.211X − 0.287	7.34	2.05	0.72	s_r = 0.034X + 0.621
	9.98	10.40	2.59			9.19	1.02		
	16.00	15.08	2.26	3.03		15.12	3.45	1.53	
	20.00	19.29	4.89			19.18	3.72		
Cycloate	0.79	0.75	0.20	0.04	X = 0.549C + 0.046	0.65	0.25	0.10	X = 0.859C − 0.018
	1.19	0.99	0.19		s_R = 0.185X + 0.044	1.04	0.41		s_R = 0.096X + 0.216
	3.17	2.58	0.44	0.60	s_r = 0.202X − 0.126	2.62	0.47	0.23	s_r = 0.071X + 0.039
	3.96	3.58	0.91			3.37	0.47		
	6.34	5.28	0.94	0.95		5.49	0.61	0.51	
	7.92	7.14	1.37			6.85	0.90		
Diazinon	0.51	0.36	0.12	0.07	X = 0.949C − 0.120	0.43	0.14	0.08	X = 0.906C − 0.034
	0.77	0.60	0.14		s_R = 0.160X + 0.058	0.65	0.14		s_R = 0.235X + 0.025
	2.03	1.78	0.34	0.16	s_r = 0.106X + 0.021	1.85	0.47	0.43	s_r = 0.183X − 0.013
	2.54	2.40	0.42			2.18	0.48		
	4.06	3.70	0.57	0.60		3.74	0.84	0.62	
	5.08	4.61	0.97			4.55	1.36		
Dichlorvos	1.00	1.00	0.09	0.10	X = 0.963C + 0.030	1.02	0.09	0.11	X = 0.883C + 0.132
	1.50	1.45	0.10		s_R = 0.181X − 0.119	1.45	0.18		s_R = 0.156X − 0.064
	4.00	3.85	0.30	0.76	s_r = 0.175X − 0.106	3.61	0.53	0.22	s_r = 0.076X + 0.008
	5.00	4.99	1.08			4.66	0.36		
	8.00	7.68	1.18	1.25		7.09	1.11	0.79	
	10.00	9.58	1.88			9.05	1.68		
Diphenamid	1.00	1.04	0.27	0.17	X = 0.916C + 0.138	1.07	0.49	0.32	X = 1.049C + 0.069
	1.49	1.52	0.18		s_R = 0.144X + 0.071	1.75	0.71		s_R = 0.159X + 0.351
	3.98	3.68	0.24	0.96	s_r = 0.204X − 0.080	4.05	0.77	0.82	s_r = 0.103X + 0.184
	4.98	5.20	1.35			5.59	1.58		
	7.97	7.37	1.29	1.33		8.24	1.64	0.92	
	9.96	8.62	1.20			10.33	1.71		
Disulfoton	0.50	0.44	0.09	0.06	X = 0.870C − 0.014	0.39	0.13	0.07	X = 0.814C − 0.042
	0.76	0.61	0.11		s_R = 0.192X − 0.004	0.51	0.19		s_R = 0.311X + 0.022
	2.02	1.65	0.24	0.36	s_r = 0.190X − 0.044	1.57	0.65	0.18	s_r = 0.134X + 0.010
	2.52	2.30	0.48			2.04	0.75		
	4.03	3.41	0.52	0.67		3.07	1.09	0.63	
	5.04	4.52	1.15			4.41	0.73		
Disulfoton sulfone	1.50	1.54	0.16	0.20	X = 1.046C − 0.086	1.37	0.61	0.28	X = 0.908C + 0.008
	2.25	2.14	0.19		s_R = 0.243X − 0.272	2.05	0.51		s_R = 0.181X + 0.292
	6.00	5.77	0.42	1.75	s_r = 0.263X − 0.283	5.38	1.19	0.59	s_r = 0.114X + 0.079
	7.50	8.34	2.47			7.18	1.48		
	12.00	11.79	1.97	3.14		10.37	2.63	1.78	
	15.00	16.55	5.08			13.82	2.95		
Disulfoton sulfoxide	3.00	2.98	0.31	0.31	X = 0.933C + 0.183	3.41	0.83	0.43	X = 0.946C + 0.556
	4.51	4.38	0.37		s_R = 0.252X − 0.541	4.80	0.84		s_R = 0.094X + 0.496
	12.00	11.27	1.50	3.61	s_r = 0.246X − 0.558	11.59	1.95	1.86	s_r = 0.096X + 0.063
	15.00	14.92	4.47			15.01	2.70		
	24.10	22.17	4.09	4.08		21.71	1.82	1.76	
	30.10	27.82	7.60			31.24	2.45		
EPTC	0.50	0.45	0.13	0.04	X = 0.852C + 0.017	0.51	0.12	0.07	X = 0.813C + 0.096
	0.76	0.65	0.11		s_R = 0.165X + 0.038	0.69	0.10		s_R = 0.106X + 0.052
	2.02	1.70	0.25	0.38	s_r = 0.190X − 0.064	1.70	0.24	0.14	s_r = 0.064X + 0.026
	2.52	2.26	0.53			2.19	0.20		
	4.03	3.50	0.56	0.55		3.37	0.47	0.30	
	5.04	4.20	0.83			4.23	0.61		
Ethoprop	0.50	0.47	0.10	0.09	X = 0.908C + 0.026	0.48	0.15	0.06	X = 0.930C + 0.007
	0.74	0.72	0.16		s_R = 0.195X + 0.012	0.68	0.14		s_R = 0.157X + 0.058
	1.98	1.72	0.42	0.19	s_r = 0.122X + 0.011	1.90	0.22	0.10	s_r = 0.097X + 0.000
	2.48	2.35	0.43			2.22	0.30		
	3.97	3.70	0.54	0.62		3.83	0.73	0.58	

TABLE 4 *Continued*

Analyte	Reagent Water					Finished Drinking Water			
	C [A]	X [B]	s_R [C]	s_r [D]	Regr. Equations	X	s_R	s_r	Regr. Equations
	4.96	4.50	1.01			4.58	1.22		
Fenamiphos	4.00	4.43	1.35	1.00	X = 0.855C + 0.865	3.38	0.86	0.70	X = 0.898C − 0.190
	6.01	5.74	1.14		s_R = 0.166X + 0.515	5.31	1.40		s_R = 0.239X + 0.068
	16.00	13.33	4.23	1.50	s_r = 0.106X + 0.421	14.23	3.97	2.83	s_r = 0.156X + 0.041
	20.00	18.26	2.65			16.80	2.95		
	32.00	30.52	4.38	4.85		27.87	6.98	4.53	
	40.00	34.85	6.68			37.97	9.85		
Fenarimol	1.00	1.17	0.34	0.29	X = 0.933C + 0.194	1.12	0.36	0.18	X = 0.902C + 0.213
	1.50	1.52	0.31		s_R = 0.218X + 0.030	1.59	0.54		s_R = 0.134X + 0.237
	4.00	3.53	0.40	1.18	s_r = 0.251X − 0.043	3.58	0.62	0.37	s_r = 0.120X + 0.007
	5.00	5.27	1.55			4.54	0.49		
	8.00	7.37	1.20	1.96		7.49	1.93	1.27	
	10.00	10.13	3.14			9.90	1.34		
Fluridone	3.00	3.35	1.20	0.70	X = 0.971C + 0.489	3.54	1.16	1.45	X = 0.928C + 0.899
	4.50	5.05	0.34		s_R = 0.245X − 0.029	5.53	2.79		s_R = 0.216X + 0.659
	12.00	11.15	1.64	3.07	s_r = 0.262X − 0.402	11.20	2.36	1.61	s_r = 0.123X + 0.829
	15.00	15.44	4.56			14.21	2.37		
	24.00	23.28	4.62	6.76		22.64	6.65	5.40	
	30.00	31.10	11.8			31.07	7.85		
Hexazinone	1.00	1.20	0.27	0.24	X = 0.943C + 0.250	1.13	0.35	0.13	X = 0.881C + 0.254
	1.51	1.67	0.33		s_R = 0.217X − 0.020	1.61	0.45		s_R = 0.132X + 0.206
	4.02	3.64	0.41	0.19	s_r = 0.242X − 0.091	3.65	0.73	0.45	s_r = 0.134X − 0.055
	5.02	5.27	1.53			4.62	0.50		
	8.03	7.43	1.13	1.74		7.29	1.73	1.16	
	10.00	10.47	2.97			9.46	1.16		
Merphos	1.66	0.76	0.27	0.30	X = 0.499C − 0.036	0.93	0.46	0.23	X = 0.556C − 0.044
	2.48	1.30	0.47		s_R = 0.366X − 0.008	1.21	0.48		s_R = 0.212X + 0.243
	6.62	3.08	1.14	0.44	s_r = 0.150X + 0.132	3.58	0.98	0.86	s_r = 0.176X + 0.044
	8.28	4.02	1.51			4.54	0.98		
	13.20	6.57	2.71	1.66		7.77	1.84	1.35	
	16.60	8.49	2.55			9.05	2.72		
Methyl paraoxon	1.99	2.20	0.47	0.61	X = 0.977C + 0.270	2.62	0.65	0.48	X = 1.019C + 0.582
	2.99	3.30	0.78		s_R = 0.155X + 0.175	3.65	0.60		s_R = 0.204X + 0.031
	7.97	7.35	1.35	0.88	s_r = 0.071X + 0.402	8.10	1.57	2.47	s_r = 0.196X − 0.089
	9.96	10.09	1.84			10.98	2.70		
	15.90	15.63	2.28	1.95		16.74	2.89	2.58	
	19.90	21.01	3.30			21.64	4.94		
Metolachlor	3.00	2.85	0.42	0.39	X = 0.919C + 0.070	3.46	0.84	0.37	X = 0.939C + 0.657
	4.51	4.17	0.60		s_R = 0.124X + 0.073	4.91	0.92		s_R = 0.063X + 0.609
	12.00	10.97	1.68	1.32	s_r = 0.118X − 0.035	12.27	1.14	1.17	s_r = 0.079X + 0.045
	15.00	13.84	1.60			14.29	1.73		
	24.00	22.71	2.33	3.11		22.04	1.63	1.97	
	30.00	27.28	3.94			30.22	3.14		
Metribuzin	0.60	0.66	0.15	0.07	X = 0.920C + 0.097	0.63	0.15	0.07	X = 0.896C + 0.096
	0.90	0.91	0.14		s_R = 0.203X − 0.010	0.92	0.16		s_R = 0.103X + 0.079
	2.40	2.16	0.19	0.54	s_r = 0.229X − 0.104	2.19	0.30	0.23	s_r = 0.107X − 0.015
	3.00	3.10	0.82			2.82	0.20		
	4.80	4.37	0.69	0.98		4.32	1.00	0.55	
	6.00	5.71	1.62			5.59	0.40		
Mevinphos	1.50	1.47	0.17	0.15	X = 0.973C − 0.027	1.51	0.42	0.32	X = 0.927C + 0.053
	2.25	2.06	0.23		s_R = 0.137X − 0.045	1.99	0.17		s_R = 0.095X + 0.183
	6.00	5.73	0.32	0.15	s_r = 0.171X − 0.142	5.39	1.04	0.40	s_r = 0.030X + 0.261
	7.50	7.61	1.67			7.20	0.80		
	12.00	11.59	0.70	1.82		10.84	2.07	0.72	
	15.00	14.45	2.68			14.88	0.25		
MGK-264	2.36	2.02	0.58	0.62	X = 0.937C − 0.144	2.31	0.79	0.44	X = 0.915C + 0.127
	3.55	3.36	1.21		s_R = 0.138X + 0.392	3.34	1.07		s_R = 0.137X + 0.532
	9.46	8.26	0.96	1.04	s_r = 0.134X + 0.241	8.83	2.14	1.59	s_r = 0.138X + 0.059
	11.80	10.59	1.70			10.68	2.15		
	18.90	18.41	2.49	3.77		17.04	1.70	2.47	
	23.60	22.01	4.47			22.67	3.79		
Molinate	0.50	0.51	0.11	0.06	X = 0.922C + 0.033	0.51	0.23	0.08	X = 0.839C + 0.086
	0.75	0.69	0.14		s_R = 0.172X + 0.018	0.71	0.21		s_R = 0.084X + 0.174
	2.00	1.82	0.16	0.39	s_r = 0.215X − 0.065	1.77	0.31	0.16	s_r = 0.056X + 0.044
	2.50	2.47	0.53			2.23	0.27		
	4.00	3.60	0.48	0.84		3.35	0.53	0.25	
	5.00	4.76	1.23			4.30	0.67		
Napropamide	0.80	0.76	0.20	0.20	X = 0.857C + 0.086	0.64	0.36	0.18	X = 0.868C − 0.075
	1.19	1.12	0.32		s_R = 0.130X + 0.119	0.90	0.47		s_R = 0.134X + 0.290
	3.18	2.69	0.32	0.62	s_r = 0.122X + 0.088	2.59	0.48	0.28	s_r = 0.090X + 0.109
	3.98	3.70	0.73			3.48	0.46		
	6.37	5.40	0.84	0.62		5.46	1.84	0.84	

TABLE 4 *Continued*

Analyte	Reagent Water					Finished Drinking Water			
	C [A]	X [B]	s_R [C]	s_r [D]	Regr. Equations	X	s_R	s_r	Regr. Equations
	7.96	6.93	0.96			6.97	0.94		
Norflurazon	1.00	1.35	0.50	0.34	X = 0.877C + 0.418	1.10	0.35	0.19	X = 0.867C + 0.226
	1.50	1.63	0.35		s_R = 0.227X + 0.103	1.54	0.47		s_R = 0.149X + 0.209
	4.00	3.64	0.63	1.32	s_r = 0.275X − 0.051	3.50	0.87	0.43	s_r = 0.109X + 0.040
	5.00	4.79	1.66			4.45	0.61		
	8.00	7.48	1.29	1.99		6.93	1.50	1.02	
	10.00	9.97	2.95			9.73	1.41		
Pebulate	0.49	0.51	0.16	0.08	X = 0.858C + 0.069	0.51	0.20	0.06	X = 0.790C + 0.114
	0.74	0.64	0.21		s_R = 0.166X + 0.092	0.68	0.20		s_R = 0.117X + 0.132
	1.97	1.84	0.45	0.49	s_r = 0.195X − 0.030	1.64	0.25	0.19	s_r = 0.077X + 0.018
	2.46	2.22	0.55			2.03	0.44		
	3.94	3.40	0.42	0.52		3.24	0.51	0.24	
	4.92	4.28	0.79			4.14	0.68		
Prometon	0.50	0.47	0.13	0.05	X = 0.969C − 0.014	0.42	0.14	0.17	X = 0.938C − 0.031
	0.75	0.72	0.09		s_R = 0.154X + 0.035	0.71	0.15		s_R = 0.118X + 0.085
	2.00	1.79	0.17	0.45	s_r = 0.215X − 0.072	1.85	0.35	0.17	s_r = 0.041X + 0.142
	2.50	2.59	0.58			2.27	0.12		
	4.00	3.73	0.44	0.78		3.80	0.96	0.41	
	5.00	4.92	1.24			4.54	0.50		
Pronamide	2.00	2.44	0.74	0.30	X = 0.913C + 0.586	1.81	0.47	0.38	X = 0.894C − 0.026
	3.00	3.24	0.44		s_R = 0.121X + 0.299	2.54	0.85		s_R = 0.115X + 0.352
	8.00	7.56	0.82	1.38	s_r = 0.190X − 0.248	7.09	1.04	0.68	s_r = 0.075X + 0.208
	10.00	10.46	2.07			8.99	0.92		
	16.00	15.43	1.25	3.06		14.00	3.01	1.64	
	20.00	18.16	3.88			18.45	1.75		
Propazine	0.30	0.29	0.08	0.04	X = 0.917C + 0.021	0.31	0.06	0.06	X = 0.891C + 0.043
	0.45	0.44	0.08		s_R = 0.159X + 0.024	0.44	0.08		s_R = 0.132X + 0.020
	1.20	1.10	0.12	0.23	s_r = 0.189X − 0.027	1.09	0.18	0.14	s_r = 0.092X + 0.021
	1.50	1.49	0.33			1.42	0.11		
	2.40	2.19	0.31	0.42		2.14	0.45	0.24	
	3.00	2.67	0.60			2.74	0.34		
Simazine	0.49	0.48	0.07	0.14	X = 0.928C + 0.041	0.55	0.15	0.07	X = 0.964C + 0.075
	0.74	0.77	0.22		s_R = 0.126X + 0.037	0.78	0.16		s_R = 0.078X + 0.102
	1.97	1.80	0.25	0.25	s_r = 0.089X + 0.081	1.98	0.22	0.13	s_r = 0.094X − 0.002
	2.46	2.29	0.26			2.39	0.25		
	3.94	3.78	0.44	0.48		3.88	0.32	0.53	
	4.92	4.60	0.62			4.92	0.73		
Simetryn	0.50	0.53	0.15	0.07	X = 0.925C + 0.076	0.56	0.10	0.07	X = 0.897C + 0.114
	0.74	0.77	0.12		s_R = 0.163X + 0.032	0.78	0.07		s_R = 0.015X + 0.024
	1.98	1.81	0.10	0.40	s_r = 0.212X − 0.063	1.86	0.34	0.18	s_r = 0.085X + 0.014
	2.48	2.53	0.59			2.33	0.22		
	3.97	3.61	0.45	0.82		3.58	0.78	0.39	
	4.96	4.74	1.32			4.74	0.14		
Stirofos	4.00	3.97	1.09	0.63	X = 0.914C + 0.286	4.07	1.39	0.69	X = 0.987C − 0.018
	6.01	5.82	1.41		s_R = 0.187X + 0.402	5.59	1.38		s_R = 0.163X + 0.633
	16.00	13.36	5.31	2.19	s_r = 0.187X − 0.317	15.91	2.80	2.66	s_r = 0.158X − 0.081
	20.00	18.97	1.61			19.33	3.92		
	32.00	32.13	4.57	6.94		31.80	5.76	5.72	
	40.00	36.09	8.22			40.40	8.23		
Tebuthiuron	1.99	2.18	0.64	0.27	X = 0.944C + 0.280	2.44	0.46	0.27	X = 0.881C + 0.647
	2.99	3.07	0.65		s_R = 0.163X + 0.207	3.19	0.41		s_R = 0.103X + 0.164
	7.97	7.22	0.47	0.76	s_r = 0.217X − 0.295	7.49	0.94	0.71	s_r = 0.081X + 0.043
	9.96	10.20	2.35			9.57	0.90		
	15.90	15.17	2.04	3.19		14.40	2.36	1.43	
	19.90	19.54	5.46			18.83	1.76		
Terbacil	10.00	9.66	3.84	4.39	X = 0.986C + 0.514	10.04	0.63	2.17	X = 0.939C + 0.977
	15.00	17.30	6.58		s_R = 0.124X + 3.176	16.00	2.37		s_R = 0.239X − 1.520
	40.00	38.44	11.2	4.84	s_r = 0.018X + 4.136	37.54	10.3	8.23	s_r = 0.117X + 0.799
	50.00	46.81	11.0			46.69	13.8		
	80.00	84.00	4.54	5.92		76.02	11.4	6.67	
	100.0	97.26	11.6			96.81	14.4		
Terbufos	3.00	2.10	0.47	0.28	X = 0.885C − 0.547	2.07	0.72	0.37	X = 0.885C − 0.445
	4.50	3.47	0.62		s_R = 0.169X + 0.099	3.90	0.52		s_R = 0.250X + 0.070
	12.00	9.85	2.24	0.82	s_r = 0.105X − 0.029	9.95	2.63	1.82	s_r = 0.166X − 0.118
	15.00	12.57	2.01			12.52	3.04		
	24.00	21.72	2.70	3.02		21.57	5.64	3.64	
	30.00	25.41	5.17			25.23	8.03		
Terbutryn	0.60	0.60	0.10	0.10	X = 0.934C + 0.028	0.60	0.21	0.10	X = 0.853C + 0.079
	0.91	0.87	0.15		s_R = 0.102X + 0.049	0.84	0.28		s_R = 0.260X + 0.055
	2.42	2.20	0.30	0.26	s_r = 0.109X + 0.018	2.11	0.52	0.43	s_r = 0.164X − 0.015
	3.02	2.81	0.28			2.63	0.71		
	4.83	4.75	0.49	0.64		4.30	1.16	0.67	
	6.04	5.70	0.69			5.23	1.65		

TABLE 4 *Continued*

Analyte	Reagent Water					Finished Drinking Water			
	C [A]	X [B]	s_R [C]	s_r [D]	Regr. Equations	X	s_R	s_r	Regr. Equations
Triademefon	0.50	0.57	0.12	0.11	X = 0.937C + 0.090	0.51	0.12	0.06	X = 0.881C + 0.078
	0.74	0.75	0.17		s_R = 0.204X − 0.001	0.75	0.13		s_R = 0.134X + 0.041
	1.98	1.82	0.19	0.43	s_r = 0.199X − 0.022	1.79	0.33	0.20	s_r = 0.102X − 0.007
	2.48	2.59	0.61			2.26	0.18		
	3.97	3.75	0.64	0.81		3.54	0.79	0.41	
	4.96	4.83	1.35			4.54	0.53		
Tricyclazole	5.00	5.72	0.47	0.91	X = 0.841C + 1.284	5.38	1.92	0.53	X = 0.979C + 0.290
	7.49	7.10	2.15		s_R = 0.160X + 0.054	7.29	2.22		s_R = 0.215X + 0.710
	20.00	17.26	0.92	3.84	s_r = 0.197X − 0.341	18.16	4.18	4.86	s_r = 0.228X − 0.876
	25.00	22.71	5.05			25.57	6.41		
	40.00	35.79	4.71	7.16		39.19	10.0	8.25	
	50.00	44.50	8.60			52.48	11.6		
Vernolate	0.50	0.43	0.14	0.07	X = 0.798C + 0.029	0.49	0.13	0.06	X = 0.795C + 0.080
	0.75	0.61	0.18		s_R = 0.229X + 0.040	0.64	0.12		s_R = 0.120X + 0.061
	2.00	1.66	0.20	0.40	s_r = 0.231X − 0.049	1.66	0.28	0.15	s_r = 0.072X + 0.018
	2.50	2.10	0.61			2.10	0.23		
	4.00	3.30	0.62	0.74		3.21	0.52	0.28	
	5.00	3.75	1.35			4.17	0.58		

[A]Spike concentration, µg/L.

[B]Mean recovery concentration, µg/L.

[C]Overall standard deviation, µg/L.

[D]Single analyst standard deviation, µg/L.

TABLE 5 Method Performance for Nitrogen- and Phosphorus-Containing Pesticides in Finished Drinking Water [A]

Pesticide	Concentration, µg/L [B]	Reagent Water				Finished Drinking Water			
		s_r	s_R	RSD_r, %	RSD_R,%	s_r	s_R	RSD_r, %	RSD_R, %
Alachlor	5.0	0.67	0.78	14.5	16.9	0.68	1.05	14.2	21.9
Ametryn	2.0	0.31	0.28	16.7	15.1	0.19	0.33	9.7	16.3
Atraton	5.0	0.89	0.87	17.8	17.3	0.40	0.62	8.8	13.6
Atrazine	2.0	0.22	0.27	11.8	14.5	0.18	0.31	9.2	15.8
Bromacil	10.0	1.66	1.60	17.4	16.7	0.95	1.84	10.1	19.5
Butachlor	10.0	1.22	1.46	13.0	15.5	1.16	1.59	12.5	17.1
Butylate	5.0	0.74	0.89	19.6	23.4	0.59	0.67	15.4	17.5
Carboxin	10.0	2.04	2.00	21.8	21.4	1.18	3.08	16.3	42.6
Chlorpropham	10.0	1.77	1.78	18.2	18.2	0.94	1.87	10.0	19.8
Cycloate	5.0	0.74	0.84	17.3	19.5	0.34	0.63	8.0	14.6
Diazinon	2.0	0.21	0.34	11.8	19.2	0.31	0.44	17.6	24.9
Dichlorvos	5.0	0.74	0.76	15.3	15.6	0.35	0.64	7.8	14.2
Diphenamid	5.0	0.88	0.75	18.7	15.9	0.73	1.20	13.8	22.5
Disulfoton	2.0	0.28	0.33	16.4	19.0	0.22	0.52	14.0	32.5
Disulfoton sulfone	10.0	2.45	2.26	23.6	21.7	1.12	1.94	12.3	21.3
Disulfoton sulfoxide	10.0	1.78	1.86	18.7	19.5	1.02	1.44	10.2	14.4
EPTC	2.0	0.26	0.32	15.3	18.7	0.14	0.23	7.9	13.6
Ethoprop	2.0	0.24	0.37	12.8	20.2	0.18	0.35	9.7	18.8
Fenamiphos	20.0	2.33	3.50	12.9	19.5	2.81	4.32	15.8	24.3
Fenarimol	5.0	1.18	1.09	24.2	22.4	0.57	0.87	12.1	18.4
Fluridone	10.0	2.27	2.47	22.2	24.2	2.08	2.86	20.4	28.1
Hexazinone	5.0	1.11	1.06	22.4	21.3	0.57	0.82	12.2	17.6
Merphos	10.0	0.88	1.80	17.7	36.4	1.01	1.41	18.4	25.6
Methyl paraoxon	10.0	1.11	1.73	11.1	17.2	2.02	2.23	18.8	20.7
Metolachlor	10.0	1.06	1.22	11.4	13.2	0.84	1.24	8.3	12.4
Metribuzin	2.0	0.34	0.38	17.5	19.8	0.19	0.27	9.9	14.5
Mevinophos	10.0	1.52	1.28	15.6	13.2	0.54	1.07	5.8	11.5
MGK-264	10.0	1.48	1.66	16.0	18.0	1.34	1.80	14.4	19.4
Molinate	2.0	0.34	0.34	18.0	18.2	0.14	0.32	8.1	18.3
Napropamide	5.0	0.62	0.69	14.2	15.7	0.49	0.86	11.6	20.2
Norflurazon	5.0	1.27	1.19	26.4	24.8	0.54	0.89	11.8	19.5
Pebulate	2.0	0.32	0.39	17.8	21.8	0.15	0.33	8.8	19.5
Prometon	2.0	0.34	0.33	17.8	17.2	0.22	0.30	11.8	16.4
Pronamide	10.0	1.60	1.47	16.4	15.2	0.88	1.38	9.8	15.4
Propazine	2.0	0.32	0.32	17.4	17.2	0.19	0.26	10.4	14.3
Simazine	2.0	0.25	0.28	13.2	14.6	0.19	0.26	9.3	12.9
Simetryn	2.0	0.34	0.34	17.9	18.0	0.18	0.24	9.2	12.8
Stirofos	20.0	3.15	3.87	17.0	20.9	3.04	3.85	15.4	19.5
Tebuthiuron	10.0	1.81	1.79	18.7	18.4	0.81	1.14	8.6	12.0
Terbacil	50.0	5.03	9.35	10.1	18.8	6.41	9.93	13.4	20.7
Terbufos	10.0	0.84	1.50	10.2	18.1	1.28	2.17	15.2	25.8
Terbutryn	2.0	0.22	0.24	11.8	12.8	0.28	0.52	15.6	29.1
Triademefon	2.0	0.37	0.40	18.8	20.3	0.18	0.29	9.8	15.6
Tricyclazole	20.0	3.22	2.95	17.8	16.3	3.65	4.98	18.4	25.1

Pesticide	Concentration, µg/L [B]	Reagent Water				Finished Drinking Water			
		s_r	s_R	RSD_r, %	RSD_R,%	s_r	s_R	RSD_r, %	RSD_R, %
Vernolate	2.0	0.33	0.41	20.1	25.4	0.14	0.26	8.3	15.7
Average				16.8	18.8			12.0	19.2
Standard Deviation				3.8	4.0			3.5	6.0

[A] s_r and s_R = standard deviations for repeatability and reproducibility, respectively. RSD_r and RSD_R = corresponding relative standard deviations.
[B] Concentration value is 10 to 15 times the estimated method detection limit (MDL).