



Standard Test Method for Nitrogen- and Phosphorus-Containing Pesticides in Water by Gas Chromatography with a Nitrogen-Phosphorus Detector¹

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 Water³

D 1192 Specification for Equipment for Sampling Water and Steam in Closed Conduits³

D 1193 Specification of Reagent Water³

D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D19 on Water³

D 3370 Practices for Sampling Water from Closed Conduits³

D 3694 Practices for Preparation of Sample Containers and for Preservation of Organic Constituents⁴

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 Detector⁵

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.

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.