



Designation: D 5659 – 95

Standard Test Method for Chlorophenoxy Acid Herbicides in Waste Using HPLC¹

This standard is issued under the fixed designation D 5659; 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 test method covers the analysis of 2,4-dichlorophenoxyacetic acid (2,4-D), 2,4,5-trichlorophenoxyacetic acid (2,4,5-T), and 2,4,5-trichlorophenoxypropionic acid (silvex) in liquids and solids, using high performance liquid chromatography with an ultraviolet detector (HPLC/UV). This test method is applicable for a concentration range from approximately 50 to 1000 ppm. This range takes into consideration the sample preparation and dilutions outlined in Section 10. Lower detection levels can be obtained by using larger sample sizes, smaller total final volumes, or with the use of in-line or solid phase extraction, concentration, and/or cleanup.

1.2 The chlorophenoxy herbicides may be present as a variety of salts or esters, which are converted to, analyzed, and reported as their respective acids.

1.3 This test method is applicable to liquid and solid waste and waste extract matrices including aqueous, oil, spent solvent, soil, ash, leachates, etc.

1.4 This test method may be applicable to other phenoxy acid herbicides.

1.5 *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.* See Section 7 and 10.3.1 for specific precautionary statements.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

D 3478 Test Method for Chlorinated Phenoxy Acid Herbicides in Water³

2.2 EPA Documents:

Method 8150 Chlorinated Herbicides, Test Methods for Evaluating Solid Waste Physical/Chemical Methods SW-846 Third Edition⁴

¹ This test method is under the jurisdiction of ASTM Committee D-34 on Waste Management and is the direct responsibility of Subcommittee D34.02 on Physical and Chemical Characterization.

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² *Annual Book of ASTM Standards*, Vol 11.01.

³ Discontinued, 1992. See *Annual Book of ASTM Standards*, Vol 11.02.

⁴ Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

Method 8000A Gas Chromatography, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, 3rd Edition, Final Update 1⁴

Method 555 Determination of Chlorinated Acids in Water by High Performance Liquid Chromatography with a Photodiode Array Ultraviolet Detector EPA/600/R-92/129, Methods for the Determination of Organic Compounds in Drinking Water, Supplement No. 2⁴

3. Summary of Test Method

3.1 The chlorophenoxy acids and esters are hydrolyzed to their respective salts by heating and stirring the sample with aqueous alkali. The salts are then converted to their respective acids by the addition of HCl. The aqueous solutions of the free acids are then analyzed using High Performance Liquid Chromatography (HPLC) using ultraviolet detection.

4. Significance of Use

4.1 Phenoxy acid herbicides are used extensively for weed control. Esters and salts of 2,4-D, 2,4,5-T, and Silvex have been used for agricultural crop and lawn care.

5. Interferences

5.1 Organic liquids that have high percent levels of chlorinated organics and are denser than water may interfere with the extraction of Silvex. It is necessary to mix these samples with hexadecane, typically in a 1:1 ratio, before hydrolysis.

5.2 Phenols, especially chlorophenols interfere with the procedure, by coeluting with the analytes of interest.

5.3 Interferences may be encountered from other organic compounds that absorb UV at the specified wavelengths. Also, closely eluting compounds may complicate identification based solely on retention time. When these types of interferences are encountered, the analyst must rely on other sources of information for positive identification, such as the following:

5.3.1 Secondary confirmation wavelengths such as 227 nm or 235 nm.

5.3.2 Use of a confirmation column.

5.3.3 Use of a confirmatory chromatography program such as changing the mobile phase composition or gradient.