

Standard Test Method for N-Methyl-Carbamoyloximes and N-Methylcarbamates in Water by Direct Aqueous Injection HPLC with Post-Column Derivatization¹

This standard is issued under the fixed designation D 5315; 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 high-performance liquid chromatographic (HPLC) test method applicable to the determination of certain n-methylcarbamoyloximes and n-methylcarbamates in ground water and finished drinking water (**1**)² (also see EPA Method 531.1, Revision 3.0). The following compounds can be determined using this test method:

Analyte	Chemical Abstract Services Registry Number ^A
Aldicarb	116-06-3
Aldicarb sulfone	1646-88-4
Aldicarb sulfoxide	1646-87-3
Baygon	114-26-1
Carbaryl	63-25-2
Carbofuran	1563-66-2
3-Hydroxycarbofuran	16655-82-6
Methiocarb	2032-65-7
Methomyl	16752-77-5
Oxamyl	23135-22-0

^A Numbering system of Chemical Abstracts, Inc.

1.2 This test method has been validated in a collaborative round-robin study (**2**) and estimated detection limits (EDLs) have been determined for the analytes listed in 1.1 (Table 1). Observed detection limits may vary between ground waters, depending on the nature of interferences in the sample matrix and the specific instrumentation used.

1.3 This test method is restricted to use by, or under the supervision of, analysts experienced in both the use of liquid chromatography and the interpretation of liquid chromatograms. Each analyst should demonstrate an ability to generate acceptable results with this test method using the procedure described in 12.3.

1.4 When this test method is used to analyze unfamiliar samples for any or all of the analytes listed in 1.1, analyte identifications should be confirmed by at least one additional qualitative technique.

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 The boldface numbers in parentheses refer to the references at the end of this test method.

TABLE 1 Relative Retention Times for the Primary and Confirmation Columns and EDLs for the 10 Carbamate Pesticides

Analyte	Retention Time (minutes)		
	Primary ^A	Confirmation ^B	EDL ^C
Aldicarb	27.0	21.4	1.0
Aldicarb sulfone	15.2	12.2	2.0
Aldicarb sulfoxide	15.0	17.5	2.0
Baygon	29.6	23.4	1.0
Carbaryl	30.8	25.4	2.0
Carbofuran	29.3	24.4	1.5
3-Hydroxycarbofuran	23.3	19.0	2.0
Methiocarb	34.9	28.6	4.0
Methomyl	18.4	14.8	0.50
Oxamyl	17.4	14.6	2.0

^A Primary column—250 by 4.6 mm inside diameter Altex Ultrasphere ODS, 5 μm.

^B Confirmation column—250 by 4.6 mm inside diameter Supelco LC-1, 5 μm.

^C Estimated method detection limit in micrograms per litre.

1.5 The values stated in SI units are to be regarded as the standard.

1.6 *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.* For specific hazard statements, see Note 1 and Note 3. Additional guidance on laboratory safety is available and suitable references for the information are provided (**3-5**).

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 for Reagent Water³

D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water³

D 3370 Practices for Sampling Water from Closed Conduits³

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

