CARBAMATE AND UREA PESTICIDES

EPA Method 632

Table 1A. Summary of Holding Times and Preservation for Carbamate and Urea Pesticides by High Performance Liquid Chromatography

Analytical Parameter [a]	**Technical and Contract Holding Times**	**Preservation**
Carbamate and Urea Pesticides in Water	Technical for Extraction: 7 days from collection; Contract for Extraction: 5 days from receipt at laboratory Technical and Contract for Analysis: 40 days from extraction	Cool to 4EC ±2EC; Store in TFE-fluorocarbon-sealed bottles away from the light
Carbamate and Urea Pesticides in Soil	Technical for Extraction: 14 days from collection; Contract for Extraction: 10 days from receipt at laboratory Technical and Contract for Analysis: 40 days from extraction	Cool to 4EC ±2EC; Store away from the light

[a] Individual target compounds are listed in Table 1B.

Data Calculations and Reporting Units:

Calculate the sample results using calibration factors (CF) determined according to Sections 7.4.2 and 7.8.1 of SW-846 Method 8000A or Section 13.1.1 of EPA Method 632.

Report water sample results in concentration units of micrograms per liter (Fg/L). Report soil sample results on a dry-weight basis in micrograms per kilogram (Fg/kg).

For rounding results, adhere to the following rules:
a)If the number following those to be retained is less than 5, round down;
b)If the number following those to be retained is greater than 5, round up; or
c)If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

TABLE 1B. Target Compound List, CAS Numbers, and Contract Required Quantitation Limits for EPA Method 632

COMPOUND	CAS No.	CRQL Water µg/L	CRQL Soil µg/kg
Aminocarb	2032-59-9	2	70
Barban	101-27-9	10	330
Carbaryl	63-25-2	0.4	13
Carbofuran	1563-66-2	1	33
Chlorpropham	101-21-3	2	70
Diuron	330-54-1	0.5	20
Fenuron/ Fenuron-TCA*	101-42-8/ 4482-55-7	2	33
Fluometuron	2164-17-2	5	170
Linuron	330-55-2	10	330
Methiocarb	2032-65-7	10	330
Methomyl	16752-77-5	4	130
Mexacarbate	315-18-4	2	70
Monuron/ Monuron-TCA*	150-68-5/ 140-41-0	0.5	20
Neburon	555-37-3	2	33
Oxamyl	23135-22-0	4	130
Propham	122-42-9	10	330
Propoxur	114-26-1	10	330
Siduron	1982-49-6	4	130
Swep	1918-18-9	4	130

*Compounds cannot be separated; report total concentration.

Table 2. Summary of Calibration Procedures for Carbamate and Urea Pesticides by EPA Method 632

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (minimum blank + 3 points for each analyte) (ICAL) [a, b, c]	Initially; whenever required, due to non-compliant CCV	RSD for CFs #20%;	1. Terminate analysis 2. Re-calibrate and verify before sample analysis
Continuing Calibration Verification (CCV) at midpoint of ICAL	Beginning of each day, after every 10 samples, and the end of run	%D between CF of CCV and avg CFs from ICAL #15%	1. Re-calibrate and verify 2. Re-analyze samples back to last good CCV
Retention time evaluation of CCV standards	Each analysis CCV standards	±3 x the SD of the avg ICAL RT for each analyte	1. Re-calibrate and verify 2. Re-analyze samples back to last good CCV

[a] The ICAL low standard must be above but near the CRQL. The low ICAL standard must have a signal to noise ratio $5:1. If this requirement cannot be met, the laboratory must submit a MDL study as part of the data package.

[b] ICAL and continuing CAL standards must contain all target analytes listed in Table 1B.

[c] Report the retention time window for each analyte. Determine retention time windows as ±3 x the standard deviation of the average initial calibration retention time for each analyte.

Table 3. Summary of Internal Quality Control Procedures for Carbamate and Urea Pesticides by EPA Method 632

QC Element	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	One per Batch or SDG[a] (1 per 20 samples minimum)	< CRQL for each compound	1. Investigate source of contamination and document 2. All samples processed with a method blank that is out of control must be re-extracted and re-analyzed
Surrogate Spike	Every sample and method blank at 10 times CRQL	60-150% of expected value	1. Re-analyze all samples with non-compliant surrogate recoveries
Matrix Spike and Matrix Spike Duplicate (MS/MSD)	One MS/MSD set per batch or SDG (1 MS/MSD set per 20 samples minimum)	50-135% of expected value; #30 RPD between MS and MSD	1. Report in case narrative

[a] SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.

Dilute and re-analyze samples with concentrations exceeding the range of the calibration curve. Results for such re-analyses should fall within the mid-range of the calibration curve. Report results and submit documentation for both analyses.

Second column confirmation is required for all positive results. Confirmation must be performed on a column of a phase different from that used for quantitation. Confirmation analyses must meet all calibration criteria specified in Table 2 and blank acceptance criteria specified in Table 3.