

CHLORINATED HERBICIDES
 SW-846 Method 8151,
 Revision 0 (September 1994)

Table 1A. Summary of Holding Times and Preservation for Chlorinated Herbicides

Analytical Parameter ^a	Technical and Contract Holding Times	Preservation
Chlorinated Herbicides in Water	<u>Technical to Extraction</u> : 7 days from collection; <u>Contract to Extraction</u> : 5 days from receipt at laboratory <u>Technical and Contract to Analysis</u> : 40 days from extraction	Cool to 4EC ±2EC;
Chlorinated Herbicides in Soil	<u>Technical to Extraction</u> : 14 days from collection; <u>Contract to Extraction</u> : 10 days from receipt at laboratory <u>Technical and Contract to Analysis</u> : 40 days from extraction	Cool to 4EC ±2EC;

^a Target Compound List is provided in Table 1B

Data Calculations and Reporting Units:

Calculate the sample results using calibration factors determined according to Sections 7.4.2 and 7.8.1 of SW-846 Method 8000A.

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 Chlorinated Herbicides SW-846 Method 8151

<u>Compound</u>	<u>CAS Number</u>	<u>Water (µg/L)</u>	<u>Soil (µg/Kg)</u>
2,4-D	94-75-7	1.0	33
2,4-DB	94-82-6	1.0	33
2,4,5-T	93-76-5	1.0	33
2,4,5-TP (Silvex)	93-72-1	1.0	33
Dalapon	75-99-0	1.0	33
Dicamba	1918-00-9	1.0	33
Dichlorprop	120-36-5	1.0	33
Dinoseb	88-85-7	1.0	33
MCPP	93-65-2	500	17000
MCPA	94-74-6	500	17000

Table 2. Summary of Calibration Procedures for Chlorinated Herbicides by SW-846 Method 8151

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (minimum blank + 5 points for each analyte) (ICAL) ^{a, b, c}	Initially; whenever required, due to failure of CCV	RSD for CFs #20%; or, if using a linear calibration curve, a correlation coefficient (r) of ≥ 0.99 for each compound	1. Terminate analysis 2. Re-calibrate and verify before sample analysis
Continuing Calibration Verification (CCV) at midpoint of ICAL (Separate source from ICAL standards)	Beginning of each 12-hour time period, after every 10 samples and end of run	%D between calculated and nominal amount for each compound must be between $\pm 25.0\%$	1. Re-calibrate and verify 2. Re-analyze samples back to last good CCV
Retention time evaluation for CCV standards	Each analysis of 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 $\geq 5:1$. If this requirement cannot be met, the laboratory must submit a MDL study as part of the data package.

^b Report the retention time window for each analyte. Determine retention time windows as ± 3 x the standard deviation (SD) of the average initial calibration retention time for each analyte.

^c ICAL and continuing CAL standards must contain all surrogate compounds and target analytes listed in Table 1B.

Table 3. Summary of Internal Quality Control Procedures for Chlorinated Herbicides by SW-846 Method 8151

QC Element	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	One per Batch or SDG ^a (1 per 20 samples minimum) per analytical instrument	< 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 standard, sample and method blank at 10 times CRQL	<u>Water:</u> 75-125% of expected value <u>Soil:</u> 65-135% of expected value	1. Re-analyze all samples with non-compliant surrogate recoveries 2. If re-analysis does not solve the problem, re-extract and re-analyze
Matrix Spike and Matrix Spike Duplicate (MS/MSD)	One MS/MSD set per batch or SDG (1 MS/MSD set per 20 samples minimum) containing a minimum of 5 of the analytes chosen from Table 1B	40-160% of expected value for dinoseb and 65-135% of expected value for other target analytes; #30 RPD between MS and MSD	1. Report in Case Narrative
Laboratory Control Sample (LCS)	One LCS per batch or SDG	40-160% for dinoseb; 80-120% for other target analytes	1. Re-extract and re-analyze all samples processed with an out-of-control LCS

^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.