



Environmental Analysis Program

Pomona College
Biogeochemistry Laboratory

SOP-L309

Pesticide Analysis via Gas Chromatography

Standard Operating Procedure

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1. Purpose

This SOP defines the protocols for extraction verification, instrument parameters, operation, and safety requirements for analyzing trace pesticide residues using Gas Chromatography (GC) coupled with specialized detectors (ECD or MS) (McNair et al., 2019; United States Environmental Protection Agency, 2007).

2. Scope

- **Applies to:** Targeted pesticide residue analysis in environmental water, soil extracts, and plant tissue digests using GC-ECD or GC-MS
- **Does not apply to:** LC-amenable pesticides, non-targeted screening, or preparative-scale separations
- **Location:** Environmental analysis laboratories

3. Responsibilities

Role	Responsibility
Lab PI	Approve SOP, ensure training compliance
Trained Personnel	Follow this SOP, report deviations
New Students	Complete training before independent use

4. Definitions

- **Autosampler:** A robotic device that withdraws a precise volume from each GC vial and injects it into the inlet without manual intervention; reduces injection variability and allows unattended overnight sequences.
- **Capillary column:** A fused-silica column (typically 15–60 m × 0.18–0.32 mm ID) coated on its interior with a thin film of stationary phase; separates pesticides by their differential affinity for the stationary phase relative to the carrier gas (McNair et al., 2019).
- **Carrier gas:** An inert gas (helium, nitrogen, or hydrogen) that moves sample vapor through the column; flow rate and mode (constant pressure or constant flow) affect retention times and peak shape (McNair et al., 2019).
- **CCV (continuing calibration verification) standard:** A mid-range calibration standard analyzed every 10 injections to confirm that instrument response has not drifted; must recover within $\pm 15\%$ of the true value (United States Environmental Protection Agency, 2007).
- **Column bleed:** Gradual decomposition and volatilization of the GC stationary phase at high temperature; produces a rising baseline at the end of a temperature program; distinguished from analyte peaks by reproducible retention and broad peak shape.
- **ECD (electron capture detector):** A selective detector that measures current suppression caused by electron-capturing compounds (organochlorine pesticides, polychlorinated biphenyls); extremely sensitive (pg range) for halogenated compounds (United States Environmental Protection Agency, 2007).

- **GC-MS (gas chromatography-mass spectrometry):** Coupling of a GC with a mass spectrometer; provides compound identification by mass spectrum matching in addition to retention time; the confirmatory technique for pesticide analysis (United States Environmental Protection Agency, 2018).
- **Inlet liner:** A deactivated glass tube inside the split/splitless inlet; protects the column from non-volatile matrix residues; replaced daily or after every 50–100 injections to prevent sample carry-over and peak degradation.
- **MDL (method detection limit):** The minimum analyte concentration that can be detected with 99% confidence under the stated method conditions; determined from replicate analyses of low-level standards; listed per analyte in the method SOP.
- **RPD (relative percent difference):** $|A - B| / ((A + B) / 2) \times 100$; used to assess duplicate precision; acceptance criterion is RPD $\leq 20\%$ for environmental matrices (United States Environmental Protection Agency, 2007).
- **Septum:** A silicone or PTFE-faced silicone disc that seals the inlet and is penetrated by the autosampler needle; replaced daily or after 50–100 injections to prevent coring fragments from entering the liner.
- **Splitless injection mode:** An inlet mode in which the split vent is closed during injection to transfer nearly all of the sample onto the column; standard for trace pesticide analysis to maximize sensitivity (McNair et al., 2019).
- **Standby method:** A GC method programmed after sequence completion that drops oven, inlet, and detector temperatures while maintaining a reduced carrier gas flow; prevents column oxidation and extends stationary phase lifetime.

5. Safety and Hazards

Hazards

- **Chemical toxicity:** Pesticide standards, target analytes, and extraction solvents (hexane, dichloromethane, acetone) are highly toxic, carcinogenic, or neurotoxic. Inhalation of vapors or skin absorption poses a severe chronic health risk.
- **Thermal:** The GC injection port (200–250 °C) and column oven (50–320 °C) operate at extreme temperatures causing instant severe burns. Ensure the inlet is below 50 °C before opening it.
- **High-pressure gas cylinders:** Carrier gas cylinders (helium, nitrogen, hydrogen) pose crush and high-pressure hazards if dropped or if regulators fail. Hydrogen carrier gas presents an explosion risk if allowed to accumulate in the oven.

5.1. Required PPE

- ☐ Safety glasses or chemical splash goggles
- ☐ Laboratory coat (fully fastened)
- ☐ Nitrile or solvent-resistant gloves (Viton or Silver Shield for bulk chlorinated solvents)

5.2. Emergency Procedures

In case of chemical spill, gas leak, or equipment failure, refer to [SOP-L002 Laboratory Safety](#) or call campus safety. For compressed gas emergencies, evacuate the area and contact EH&S.

6. Equipment and Materials

6.1. Equipment

Item	Notes
Gas chromatograph	With autosampler, split/splitless capillary inlet, and detector (ECD or MS)
Fused-silica capillary column	e.g., 30 m $\times$ 0.25 mm ID, 0.25 μ m film, semi-volatile optimized (McNair et al., 2019)
Carrier gas cylinders	Helium or nitrogen (carrier); nitrogen (ECD makeup)
Inline gas traps	Moisture, oxygen, and hydrocarbon traps

6.2. Consumables

Item	Notes
GC vials (2 mL amber glass)	With PTFE-lined septa (McNair et al., 2019)
Replacement injection port liners	Deactivated split/splitless with quartz wool plug
Low-bleed septa	Replace daily or every 50–100 injections
Certified pesticide reference standards	Spanning expected concentration range (United States Environmental Protection Agency, 2007)
Extraction solvents	Hexane, dichloromethane, acetone (GC grade)

7. Procedure

7.1. Gas Cylinder and Inlet Verification

1. Verify carrier gas cylinders have sufficient pressure (> 500 psi).
2. Confirm secondary regulator delivery pressure meets manufacturer specifications (typically 60–80 psi).
3. Check moisture, oxygen, and hydrocarbon traps; replace if indicator dyes signal saturation.

7.2. Injection Port Maintenance (Pre-Run Check)

! Hot Inlet

Ensure the inlet temperature has dropped below 50 °C before opening the inlet assembly.

1. Replace the injection port septum daily or every 50–100 injections (McNair et al., 2019).
2. Inspect the glass liner; replace if charring, particulate matter, or matrix deposits are visible.

7.3. Sequence Setup and Sample Loading

1. Prepare calibration curves from certified pesticide reference materials spanning expected concentration intervals (e.g., 10–1000 µg/L) (United States Environmental Protection Agency, 2007).
2. Aliquot extracts into clean 2 mL amber GC vials; cap with PTFE-faced septa.
3. Program the injection sequence including:
 - Solvent blanks (hexane/acetone matrix check)
 - Multi-point calibration curve
 - CCV standards every 10 injections
 - Duplicate sample matrices

7.4. Initiating the GC Run

1. Load the analytical method and verify parameters (United States Environmental Protection Agency, 2007):
 - **Inlet:** Splitless mode; 250 °C
 - **Oven program:** 50 °C (1 min hold) → ramp 10 °C/min → 300 °C (5 min hold)
 - **Carrier gas:** Constant flow, 1.2 mL/min helium
2. Allow all zones to reach setpoint; confirm **Ready** status in software.
3. Initiate the sequence; monitor the first injection cycle to confirm the autosampler syringe articulates without bending the needle.

7.5. Post-Analysis and Standby Shutdown

1. Review chromatogram baselines for peak symmetry, column bleed spikes, or retention time shifts.

💡 Retention Time Drift

If retention times shift, trimming 10–20 cm from the front of the analytical column can restore performance. Document the trim in the instrument logbook (McNair et al., 2019).

2. Upon sequence completion, load the Standby method to drop oven and detector temperatures while maintaining a reduced carrier gas purge to prevent column phase oxidation.

8. Quality Control and Acceptance Criteria

- Initial calibration: $r^2 \geq 0.995$ (United States Environmental Protection Agency, 2007).
- CCV standards every 10 injections: recovery within $\pm 15\%$; halt and re-calibrate if outside this window (United States Environmental Protection Agency, 2007).
- Method blanks: no target analyte peaks above the MDL.
- Duplicate RPD: 20% (United States Environmental Protection Agency, 2007).

9. Waste Disposal

Waste Type	Container	Disposal Method
Spent solvents and rinses	Halogenated or non-halogenated jug (as appropriate)	EH&S pickup
Used septa, liners, vials	Solid chemical waste	Hazardous waste
Expired pesticide standards	Hazardous waste container	EH&S pickup

10. Maintenance

- **Daily:** Replace septum; inspect liner.
- **Weekly:** Check gas trap indicators; verify carrier gas pressure.
- **As needed:** Trim or replace the analytical column if baseline degradation or retention time shifts persist (McNair et al., 2019).
- **Annually:** Full instrument preventive maintenance per manufacturer schedule.

11. References

- United States Environmental Protection Agency (2007)
- United States Environmental Protection Agency (2018)
- McNair et al. (2019)

McNair, H. M., Miller, J. M., & Snow, N. H. (2019). *Basic gas chromatography* (3rd ed.). John Wiley; Sons. <https://doi.org/10.1002/9781119450795>

United States Environmental Protection Agency. (2007). *Method 8081B: Organochlorine pesticides by gas chromatography* [SW-846]. U.S. EPA, Office of Solid Waste. <https://www.epa.gov/hw-sw846/sw-846-test-method-8081b-organochlorine-pesticides-gas-chromatography>

United States Environmental Protection Agency. (2018). *Method 8270E: Semivolatile organic compounds by gas chromatography/mass spectrometry (GC/MS)* [SW-846]. U.S. EPA, Office of Resource Conservation; Recovery. <https://www.epa.gov/hw-sw846/sw-846-test-method-8270e-semivolatile-organic-compounds-gas-chromatographymass>

12. Related SOPs

SOP	Title	Relationship
L002	Laboratory Safety	Required prerequisite
L307	ELISA Pesticide Screening	Complementary screening method

13. Training Record

Training is documented in the lab training log. Before working independently, personnel must:

1. Read this SOP in full
2. Observe a trained operator perform the procedure
3. Perform the procedure under supervision
4. Be signed off by the PI or designated trainer