

Proposal: Certified Reference Material Comparison to Verify CCLEAN Laboratory Performance

Prepared by Applied Marine Sciences for the CCLEAN Steering Committee · October 5, 2026

Purpose and Request

CCLEAN requests Steering Committee approval of **\$8,538** to test the accuracy of its analytical laboratories with **two** reference mixtures of current-use pesticides at known concentrations from Labmix24 (Hamminkeln, Germany). The mixtures will be sent as blind duplicates to **Physis** and **Weck** to test whether each can accurately detect and quantify neonicotinoids, pyrethroids, and fipronil at concentrations relevant to aquatic toxicity. The results will inform which laboratory and method CCLEAN uses going forward, and support interpretation of the historical neonicotinoid data.

Background

CCLEAN has measured neonicotinoids since Program Year 2018 using Physis Environmental Laboratories (EPA 625.1-MRM, GC/MS/MS). State Water Board QA staff have raised concerns that GC heating degrades these compounds, and Central Coast Regional Water Quality Control Board staff have said they will not accept GC/MS neonicotinoid data for regulatory purposes.

In the 2026 wet season, CCLEAN sent field duplicates of four POTW effluents, three rivers, and two ocean sites to both Physis and Weck Laboratories (LC/MS/MS). The lab comparison showed that CCLEAN's two candidate laboratories disagree sharply on neonicotinoid detections, and neither result can yet be verified against a known concentration.

Key findings:

- **Detections:** Physis reported one detection (Thiamethoxam, Effluent D). Weck detected two to five neonicotinoids in every effluent and in the Pajaro and Salinas rivers. Neither lab detected neonicotinoids in the San Lorenzo River or Monterey Bay.
- **Paradox in limits:** Physis reports lower MDLs (0.2–1 ng/L) than Weck (1.5–15 ng/L), yet detected far less. Thermal degradation is a plausible but unproven explanation.
- **Precision:** Mean replicate agreement was 107% at Weck versus 121% at Physis.

These results favor LC/MS/MS, but agreement between labs is not the same as accuracy. Only samples of known concentration can show which laboratory is correct, and by how much.

Objectives

1. Measure each laboratory's accuracy (percent recovery) for nine current-use pesticides at two known concentrations.
2. Determine whether past non-detections reflect true absence or insufficient method sensitivity, including compounds neither lab detected in ambient samples (e.g., Thiacloprid).
3. Test whether accuracy holds at concentrations near the UC Davis toxicity thresholds for local aquatic invertebrates.
4. Provide information for selecting CCLEAN's neonicotinoid method and for qualifying historical Physis data.

Approach

Labmix24 will prepare a high-concentration mixture and dilute it 25-fold to make a corresponding low mixture (Table 1). Bids were requested from three U.S. suppliers and Labmix24; only Labmix24 responded. Each mixture comes as four sealed 5-mL glass ampoules with an 18-month shelf life.

Table 1. Compounds and concentrations in the “High” and “Low” test mixtures (ng/L), with UC Davis toxicity thresholds and the highest 2026 CCLEAN detection for context.

Compound	CAS No.	Max. 2026 CCLEAN detection	UCD toxicity threshold	High (ng/L)	Low (ng/L)
Imidacloprid	138261-41-3	10,000	16	1,000	40
Thiamethoxam	153719-23-5	ND	0.6	300	12
Clothianidin	210880-92-5	36.4	130	200	8
Dinotefuran	16252-70-0	25	—	30	1.2
Acetamiprid	135410-20-7	6.33	—	10	0.4
Thiacloprid	111988-49-9	2.6	—	3	0.12
Permethrin (cis-/trans-)	52645-53-1	9.18	2	10	0.4
Bifenthrin	82657-04-3	ND	0.6	6	0.24
Fipronil	120068-37-3	1.66	2.2	2	0.08

— = no threshold available or not analyzed in the 2026 comparison; ND = not detected.

Study design

- **Blind submission:** Ampoules will be relabeled with coded sample IDs before submission, so labs cannot identify the level or compounds.
- **Laboratories:** Physis (GC/MS/MS) and Weck (LC/MS/MS) will each receive two ampoules per level, for four samples per lab.
- **Evaluation:** Percent recovery against the known concentration, relative percent difference between replicates, and false-negative rate at each level, judged against CCLEAN QAPP acceptance limits.
- **Diagnosis:** Comparing lab blank to spiked results to separate matrix effects from thermal degradation or calibration error as the cause of the inter-lab disagreement.

The low mixture falls below Weck’s reporting limits (10–50 ng/L) for most compounds, so it will test the detection limits each lab reports.

Budget, Schedule, and Deliverables

The study costs **\$8,538**: \$1,598 for the reference mixtures (Labmix24 Offer 15825, €661 per mixture) and \$6,940 for laboratory analysis. Analysis costs assume four samples per lab (2 ampoules × 2 levels).

Table 2. Proposed budget for CCLEAN Certified Reference Material laboratory comparison study.

Item	Unit cost (USD)	Qty	Cost (USD)
Labmix24 mixtures (3 × €661)	740	2	1,480
Currency handling and delivery (€105)	—	—	118
Reference materials subtotal			1,598
Physis – neonicotinoids	305	4	1,220
Physis – fipronil	305	4	1,220
Physis – pyrethroids	325	4	1,300
Weck – neonicotinoids	425	4	1,700
Weck – fipronil and pyrethroids	375	4	1,500
Laboratory analysis subtotal			6,940
Total			8,538

Euro costs converted at \$1.12 per euro; the final amount will follow the exchange rate on the payment date.

- **Schedule:** Order placed on approval; samples submitted blind to both labs on receipt.
- **Deliverable:** A short technical memo reporting recovery, precision, and detection by lab, compound, and concentration, with a recommendation on method selection.

Request

The Steering Committee is asked to:

1. Approve \$8,538: \$1,598 (€1,427) for two Labmix24 reference mixtures and \$8,538 for analysis by Physis and Weck.