

STANDARD OPERATING PROCEDURE

Sediment Collection, Preparation, and Laboratory Analysis for shrimp farms and Inland Water Systems

Version	1.0	Date of Issue	05-12-2025
Purposes	<p>This Standard Operating Procedure (SOP) describes the standardized methodology for:</p> <ol style="list-style-type: none"> 1. Field collection of sediment samples, with emphasis on Van Veen grab sampling; 2. Sample handling, transport, and storage; 3. Sediment preparation steps, including thawing, homogenization, drying, grinding, sieving; 4. Laboratory analyses, including dry weight, ash content, pH, EC/salinity, granulometry, total carbon (C), total nitrogen (TN), total phosphorus (TP), and nutrient extractions. 		
Scope	<p>This SOP applies to:</p> <ul style="list-style-type: none"> • Shrimp aquaculture farms (intensive, super-intensive, rice–shrimp, mangrove–shrimp); • Ponds, canals, rivers, and estuarine zones associated with aquaculture; • Citizen-science programs requiring standardized sediment methods; • Research projects assessing nutrient cycling, carbon accumulation, pollutants, or GHG-related sediment processes. 		
Responsibilities	<ul style="list-style-type: none"> • Field Team: sediment collection, field documentation, sample splitting, labeling. • Laboratory Team: drying, sieving, grinding, chemical analyses, QA/QC. • Data Manager: ensuring traceability, file organization, metadata. 		
Prerequisites	<p>1. Field Equipment</p> <ul style="list-style-type: none"> • Van Veen grab sampler (5–12 L recommended for ponds/rivers) • Deployment rope or wire line with marked intervals • GPS device • Stainless steel spatulas or spoons 		

	<ul style="list-style-type: none"> • Plastic or stainless-steel trays for sub-sampling • Pre-labeled HDPE or glass containers • Cooler boxes with ice packs • Disposable nitrile gloves • Boat hook or pole (for positioning) • Cleaning water and brushes • Field notebook or digital log sheet <p>2. Laboratory Equipment (from user document)</p> <ul style="list-style-type: none"> • Inox bowls • Mortar and pestle • 2 mm sieve • Drying ovens: <ul style="list-style-type: none"> ○ 40°C (for pH/EC/TOC/ION samples) ○ 105°C (for dry-weight/ash content) • Analytical balance (± 0.001 g) • Desiccator with silica gel • Aluminium trays/dishes • Extraction solutions (1M KCl; DI water; CaCl_2 0.01 M) • Centrifuge and 0.45 μm PES filters • Spectrophotometer / cuvette tests (for nutrients) • ICP capability (TP, metals—external lab if needed)
<p>Related documents</p>	<p>ISO 5667-12:2017. <i>Water quality — Sampling — Part 12: Guidance on sampling of bottom sediments.</i></p> <p>US EPA (2001). <i>Methods for Collecting Sediment Samples.</i> EPA 818-F-01-002</p> <p>ISO 10390:2005. <i>Soil quality — Determination of pH.</i></p> <p>Mudroch, A., & Azcue, J. M. (1995). <i>Manual of Aquatic Sediment Sampling.</i> Lewis Publishers.</p> <p>Brils, J. (2008). <i>Sediment monitoring and the European Water Framework Directive.</i> <i>Annali dell'Istituto Superiore di Sanità</i>, 44(3), 218–223.</p>

	<p>ISO 14255:1998. <i>Soil quality — Determination of nitrate, nitrite, and ammonium in field-moist soils using 1 M KCl extraction.</i></p> <p>Wang, X. et al. (2011). <i>Soil carbon and nitrogen determination by dry combustion.</i> Communications in Soil Science and Plant Analysis, 42, 329–338</p>
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Procedure	
<p>Step 1: Sediment Collection (Van Veen Grab Method)</p>	<p>1. Site Selection</p> <ul style="list-style-type: none"> • Select representative areas avoiding submerged vegetation, exposed gravel bars, or areas with strong hydrodynamic disturbance unless required for study design. • For shrimp ponds, sample locations should include: <ul style="list-style-type: none"> ○ middle of pond; ○ near inlet; ○ near outlet; ○ optional: sludge accumulation “hotspots.” <p>2. Preparation Before Deployment</p> <ol style="list-style-type: none"> 1. Inspect grab for smooth hinge movement; ensure that: <ul style="list-style-type: none"> ○ jaws close properly, ○ trigger mechanism is functioning, ○ rope/wire is securely fastened. 2. Rinse grab with ambient water to remove contamination from previous sites. 3. Put on gloves and prepare containers before deployment. <p>3. Grab Deployment Procedure</p> <ol style="list-style-type: none"> 1. Lower the open grab vertically through the water column at a controlled speed (avoid disturbance that may prematurely close the grab). 2. Allow the grab to reach the bottom with enough momentum to penetrate 5–10 cm into the sediment. 3. Once the grab lands, allow 2–5 seconds for stabilization. 4. Pull the rope upward with a firm, continuous motion to close the jaws. 5. Retrieve the grab without rotating or shaking to avoid sample loss. <p>4. Acceptance Criteria for a “Good” Sediment Grab</p> <p>A valid grab must:</p>

	<ul style="list-style-type: none"> • Have full and even closure of both jaws; • Contain undisturbed surface sediment, including intact floc layer if present; • Have no visible washout or leakage; • Not be overfilled (i.e., jaws can close completely); • Have minimal stones, debris, or vegetation. <p>Reject the grab if:</p> <ul style="list-style-type: none"> • The surface appears scoured or incomplete; • Jaws did not close properly; • Large stones prevent proper collection; • Sediment is too compact and not adequately sampled. <p>Repeat sampling until an acceptable grab is obtained.</p>
<p>Step 2: Transport & Storage</p>	<ul style="list-style-type: none"> • Transport in cooling boxes; avoid freezing unless required (see below). • For analyses requiring fresh material (dry weight), avoid long holding times (>48h). • For samples to be frozen: freeze at -20°C in labeled bags.
<p>Step 3: Sediment Preparation in the Laboratory</p>	<p>1. Thawing (if previously frozen)</p> <ul style="list-style-type: none"> • Place sediment overnight in refrigerator at 4°C. • Do not thaw at room temperature to prevent microbial alteration. <p>2. Homogenization</p> <ol style="list-style-type: none"> 1. Transfer thawed sediment into an inox bowl. 2. Mix thoroughly with stainless-steel spoon until homogeneous. 3. Weigh the required fresh weight (FW) for each analysis (see Table 1). <p>3. Pre-drying at 40°C (for pH/EC/TOC/Nutrients)</p> <ol style="list-style-type: none"> 1. Transfer homogenized sediment onto aluminum trays. 2. Dry at $\leq 40^{\circ}\text{C}$ until constant weight. 3. Mix the sample periodically to ensure complete drying. 4. Grind with mortar and pestle. 5. Sieve ≤ 2 mm; store in airtight containers until analysis. <p>4. Oven-drying at 105°C (for dry weight/ash content)</p>

Step 4: Sample analysis

Note that for different parameters, minimum weights needed for analyses (based on 50% DW) – take some extra material for backup

Parameter	Required FW (g)	Required DW (g)	Notes
Dry weight / ash	10	—	Fresh sediment
Granulometry	3 × 20	—	Replicates
pH	80 FW → dried	10	Dried at 40°C
EC / Salinity	—	10	Dried at 40°C
Total N	—	2	Dried at 40°C
Total C	—	2 + extra	Dried at 40°C
Nutrients	50 FW → dried	10	Extract with CaCl ₂

1. Dry Weight Analysis (105°C)
Procedure

1. Dry empty aluminum tray at 105°C overnight.
2. Cool in desiccator and record tray weight (m_0).
3. Add ~5 g well-mixed fresh sediment; record weight (m_1).
4. Dry at 105°C overnight.
5. Cool in desiccator; record final weight (m_2).

Calculation

$$\text{Dry Weight \%} = \frac{m_2 - m_0}{m_1 - m_0} \times 100$$

where, m_2 is weight of the container + dry sediment (after 105°C drying); m_1 is weight of the container + *fresh (wet)* sediment; m_0 is weight of the empty dried container.

2. pH in 1M KCl (on 40°C dried sample)

- Extract 1:5 (v/v) sediment:KCl solution.
- Shake 1 h; settle at least 2 h.
- Measure pH at 20°C.

3. EC & Salinity (DI extract)

- Extract 1:5 (m/v) sediment:DI water.
- Shake 30 min.
- Centrifuge; filter (0.45 µm PES).
- Measure EC adjusted to 25°C.

4. Total C & Total N (TOC/TN analyzer)

- Ground sample (<2 mm).
- Submit 2 g (or according to instrument requirement).

	<ul style="list-style-type: none"> External laboratory optional <p>5. Total P</p> <ul style="list-style-type: none"> Acid digestion (H_2SO_4/H_2O_2 or aqua regia, depending on method). ICP-OES or ICP-MS quantification. <p>6. Nutrient Extractions (NH_4^+, NO_2^-, NO_3^-, PO_4^{3-})</p> <p>Based on ISO 14255.</p> <ol style="list-style-type: none"> Extract dried sediment in 0.01 M $CaCl_2$, 1:10 ratio. Shake 2 h. Centrifuge 10 min at 3000 g. Filter (0.45 μm PES). Analyze with cuvette tests or spectrophotometry. Include blanks and standards.
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Quality management	
QA / QC procedures	<p>Field QA/QC</p> <ul style="list-style-type: none"> Clean grab between sites to prevent cross-contamination. Reject disturbed or incomplete grabs. Use field duplicates at 10% of stations. Record sediment texture, color, odor. <p>Laboratory QA/QC</p> <ul style="list-style-type: none"> Include procedural blanks for $KCl/CaCl_2$ extractions. Use certified reference material for C and N. Conduct replicate analysis ($\geq 10\%$). Control balance drift daily. <p>Health & Safety</p> <ul style="list-style-type: none"> Wear gloves and protective eyewear. Handle acids (H_2SO_4, digestion acids) inside fume hood. Lift Van Veen grab carefully; risk of pinch or crush injuries

Version	Authors	Material changes from previous version	Release Date
1.0	Long Ho	Initial full SOP	05-12-2025

Bibliography

ISO 5667-12:2017. *Water quality — Sampling — Part 12: Guidance on sampling of bottom sediments.*

US EPA (2001). *Methods for Collecting Sediment Samples. EPA 818-F-01-002*

ISO 10390:2005. *Soil quality — Determination of pH.*

Mudroch, A., & Azcue, J. M. (1995). *Manual of Aquatic Sediment Sampling. Lewis Publishers.*

Brils, J. (2008). *Sediment monitoring and the European Water Framework Directive.* *Annali dell'Istituto Superiore di Sanità*, 44(3), 218–223.

ISO 14255:1998. *Soil quality — Determination of nitrate, nitrite, and ammonium in field-moist soils using 1 M KCl extraction.*

Wang, X. et al. (2011). *Soil carbon and nitrogen determination by dry combustion.* *Communications in Soil Science and Plant Analysis*, 42, 329–338