

Electrode Testing & Verification Guide

Water Purification Monitoring Systems

SECTION 1: ELECTRODE-SPECIFIC INSPECTION PROCEDURES

A. Modified Glassy Carbon Electrodes (GCEs) and Glassy Carbon Electrodes

Detects: Lead, magnesium, manganese, mercury

Visual Inspection:

- Examine the electrode surface under magnification for roughness, scratches, or pitting
- Surface should appear dark and uniform without cracks or chips
- Check for organic residue buildup—glassy carbon can accumulate biofilm

Pre-Testing Preparation:

- Gently polish the electrode surface with fine sandpaper (600-1200 grit) or use polishing pads
- Rinse thoroughly with distilled water
- For voltammetric testing (most common for these electrodes), conduct a blank scan in the supporting electrolyte first
- Surface preparation is critical—poor surfaces give erratic readings

Testing Method:

- Use cyclic voltammetry or differential pulse voltammetry (DPV) for accurate detection
- Run multiple cycles to verify consistent peak heights at characteristic potentials
- Lead, mercury, and manganese each have distinctive peak potentials—verify these match expected values
- For magnesium detection, peaks may be less distinct; compare test results against known magnesium standards

Verification of Filtration:

- Test untreated water sample to establish baseline contaminant levels
- Test treated water after passing through filtration system
- Peak heights should be significantly reduced (typically 80-95% reduction indicates effective filtration)
- If peaks remain similar, filter may not be functioning or electrode needs replacement

B. Modified Glassy Carbon Electrodes with Nano-Additives (Carbon Nanomaterials, Graphene Composites)

Detects: Lead, magnesium, manganese, mercury

Visual Inspection:

- Surface appearance varies—may appear darker or have slight texture compared to bare glassy carbon
- Look for even coating distribution of nanomaterials
- Check for peeling, flaking, or uneven coverage which indicates poor electrode integrity

Pre-Testing Preparation:

- These electrodes are more sensitive but also more fragile than bare GCEs
- Use gentle rinsing only—avoid aggressive polishing that can remove the nanomaterial coating
- For initial conditioning, scan in electrolyte solution at low scan rates
- Allow equilibration time (2-3 minutes) before testing—nanomaterial-based electrodes need stabilization

Testing Method:

- Enhanced sensitivity means lower detection limits for contaminants
- Use DPV or stripping voltammetry for optimal results
- Smaller peak heights compared to bare GCE indicate the nanomaterial is working properly
- Test contaminated water to confirm appropriate peak response
- These electrodes give faster response times—allow 20-30 seconds for stabilization

Verification of Filtration:

- Pre-filter and post-filter water analysis shows clear differentiation due to enhanced sensitivity
 - Even trace contaminants remaining after filtration will show small but measurable peaks
 - Ideal for detecting if filtration is only partially working
 - If no peaks appear in treated water, filtration is highly effective
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C. Gold Electrodes and Screen-Printed Gold Electrodes (SPGEs)

Detects: Lead, magnesium, manganese, mercury

Visual Inspection:

- Gold electrodes should have a shiny, metallic appearance
- Check for oxidation (darkening) or surface degradation
- Screen-printed electrodes have a flat surface—verify uniform gold coating with no bare substrate visible
- Look for cracks in the printed layer

Pre-Testing Preparation:

- Gold has better stability than many other materials but can develop oxide layers
- Clean with electrochemical cleaning: perform cyclic voltammetry in 0.5M H₂SO₄ for 10-15 cycles
- Alternatively, use dilute hydrochloric acid rinse followed by distilled water
- Screen-printed electrodes need gentler handling—do not use abrasive cleaning

Testing Method:

- Gold electrodes excel at mercury detection due to strong amalgamation properties
- Use anodic stripping voltammetry (ASV) for sensitive measurement
- Gold is excellent for pre-concentration of heavy metals before measurement
- Record baseline current before and after heavy metal accumulation
- Multiple measurement cycles on same electrode are possible without significant degradation

Verification of Filtration:

- Mercury shows strong current peaks with gold electrodes—easy to see if mercury is present
 - For lead and manganese, peaks are also distinctive
 - Compare peak current (not just peak potential) between untreated and treated water
 - Persistent peaks after filtration indicate filter breakthrough or inadequate treatment
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D. Boron-Doped Diamond (BDD) Electrodes

Detects: Lead, manganese, mercury

Visual Inspection:

- Appearance is typically dark gray or black
- Surface should be smooth and uniform
- Check for any visible substrate exposure (usually metal underneath diamond coating)

Pre-Testing Preparation:

- BDD electrodes are very robust but require electrochemical conditioning
- Apply a controlled potential (cathodic or anodic depending on application) for 5-10 minutes
- This removes surface contaminants and activates the electrode
- BDD has excellent stability—minimal surface preparation needed

Testing Method:

- Extremely low background current allows detection of very low contaminant levels
- Use linear sweep voltammetry or ASV
- Excellent for manganese detection specifically
- BDD provides high signal-to-noise ratio—peaks are very clear

Verification of Filtration:

- Even residual traces of contaminants show as distinct peaks
 - Sensitive enough to detect if filtration is degrading before other methods would show it
 - Compare treated water measurements weekly to establish trends
 - Any increase in peak heights indicates filter exhaustion
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E. Copper-Based Electrodes

Detects: Lead, manganese, mercury

Visual Inspection:

- Should have characteristic reddish-copper color
- Check for corrosion (green oxidation layer), which indicates electrode degradation
- Verify no pitting or erosion patterns

Pre-Testing Preparation:

- Copper readily oxidizes—check condition frequently
- Clean with dilute acid (acetic or citric acid) if oxidation is visible
- Rinse thoroughly with distilled water
- May need re-polishing more frequently than other electrode types

Testing Method:

- Copper is particularly good for lead detection (forms copper-lead amalgams)
- Use stripping voltammetry techniques
- Copper itself can dissolve during testing—monitor for copper peaks in baseline
- If large copper peaks appear without sample, electrode is degrading

Verification of Filtration:

- Lead removal is easily verified—strong lead peak disappears when filtered
 - Mercury detection is also reliable with copper-based systems
 - Regular testing recommended due to potential electrode degradation
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F. Ion-Selective Electrodes (ISEs) and Potentiometric Electrodes

Types: Mg^{2+} Ion Selective Electrodes, PVC-matrix membrane ISEs, Ionophore electrodes, Cerium oxide-modified electrodes
Detects: Lead, magnesium, manganese, mercury (depending on type)

Visual Inspection - Electrode Body:

- Check for cracks in the body material (plastic or ceramic)
- Verify the membrane is intact (small circular portion at tip)
- No visible crystallization or cloudiness on membrane

Membrane Inspection (Critical for ISEs):

- The membrane is the most important component—it's where ion detection occurs
- Magnesium ISE membranes should be clear and uniform
- If cloudy or discolored, the membrane is failing and needs replacement
- For PVC-matrix electrodes, the matrix should be dark and intact

Internal Chamber Check (if accessible):

- ISEs have internal filling solution (internal electrolyte)
- Level should be stable—not below the fill line
- Solution should be clear (not cloudy)
- Refill if level drops significantly between tests

Pre-Testing Preparation for ISEs:

- Allow 15-30 minutes equilibration before first measurement
- Rinse electrode with distilled water (not electrolyte) between samples
- Use gentle agitation to avoid disturbing the membrane
- For magnesium ISE, store in magnesium chloride solution when not in use

Testing Method - Potentiometric Measurement:

- Use a high-impedance pH/ISE meter
- Calibration is essential: use standard solutions of known concentration
- For magnesium: typically calibrate with 10 ppm and 100 ppm Mg^{2+} solutions
- Measure potential (mV) in sample water
- Compare to calibration curve to determine ion concentration

For Ionophore-Based Electrodes:

- Ionophore selectivity is carefully designed for specific ions
- Test response time: stable reading should be achieved in 30-60 seconds
- If response is slow or unstable, ionophore may be degrading
- Temperature affects response—keep samples at controlled temperature

Verification of Filtration:

- Baseline measurement: test untreated water to establish initial ion concentration
- Post-filter measurement: test treated water
- Significant decrease (typically 50-90% reduction) indicates working filtration
- For magnesium hardness reduction, compare baseline to treated water

- If no change in ion concentrations, filtration system not functioning
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G. Screen-Printed Carbon Electrodes (SPCEs)

Detects: Lead, magnesium, manganese, mercury

Visual Inspection:

- Working electrode should be visible as a dark circular area
- Reference electrode (usually silver/silver chloride) visible as separate electrode area
- No visible cracks, scratches, or areas of missing ink/coating
- Surface should be matte (dull), not shiny

Pre-Testing Preparation:

- Screen-printed electrodes come pre-cleaned from manufacturer
- Light electrochemical conditioning recommended: run cyclic voltammetry 5-10 cycles
- No aggressive polishing—this removes the printed layer
- Can be used immediately after conditioning

Testing Method:

- Best suited for electrochemical deposition methods
- Deposit heavy metals onto electrode surface by applying negative potential
- Then scan positively to strip them off and detect them
- Multiple measurements possible on same electrode

Verification of Filtration:

- Disposable nature means you can use fresh electrode for each sample
 - Compare treated vs. untreated water using separate electrodes
 - No electrode memory effects between samples
 - Good for quality control where consistency is needed
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H. Bismuth Film Electrodes

Detects: Lead, mercury

Visual Inspection:

- Should have a dark metallic sheen from bismuth coating
- Check for even coating across substrate
- Look for any bare substrate exposure
- Surface should be smooth without pitting

Pre-Testing Preparation:

- Bismuth film is softer than some alternatives—handle gently
- Clean gently with distilled water and soft cloth
- If film is damaged, may need recoating or replacement
- Avoid touching the sensing surface with bare hands

Testing Method:

- Bismuth forms sensitive amalgam with lead and mercury
- Anodic stripping voltammetry is most effective
- Very low detection limits achievable
- Each use slightly consumes the film—eventually needs replacement (after ~100 measurements)

Verification of Filtration:

- Excellent sensitivity makes it ideal for detecting if filtration is working
- Even trace amounts of lead or mercury easily detected
- Loss of sensitivity (larger potentials needed to detect same concentration) indicates bismuth film is wearing

SECTION 2: FILTRATION VERIFICATION PROCEDURES

Complete Testing Protocol

Step 1: Baseline Establishment

- Collect untreated water directly from source

- Test with selected electrode method
- Record all values: peak heights/currents, potentials, ion concentrations
- Run measurements 3 times to establish average baseline
- Document date, time, and water source

Step 2: Post-Filter Testing

- Collect treated water after full passage through filtration system
- Use same electrode and method
- Allow adequate stabilization time (depends on electrode type)
- Run measurements 3 times
- Record all values

Step 3: Comparison Analysis

- Calculate percent reduction: $((\text{Baseline} - \text{Treated}) / \text{Baseline}) \times 100$
- Expected results vary by contaminant and filter type:
 - Lead: 85-99% reduction expected
 - Mercury: 80-98% reduction expected
 - Manganese: 70-95% reduction expected
 - Magnesium/Hardness: 60-85% reduction expected

Step 4: System Integrity Check

- Examine filter housing for leaks
- Check all connections are tight
- Verify water pressure is stable across filter
- Sudden drops in pressure indicate filter saturation

Step 5: Documentation

- Keep test log with date, electrode condition, untreated values, treated values, reduction percentage
- Track filter replacement dates
- Note any electrode maintenance or cleaning performed
- Identify trends over time

Interpreting Results

Excellent Filtration (>90% reduction):

- Electrode surface is clean and functioning properly
- Filter media is fresh and active
- Continue normal maintenance schedule

Good Filtration (70-90% reduction):

- Acceptable performance but monitor closely
- Filter may need replacement soon (within 1-2 months)
- Verify electrode is properly calibrated

Marginal Filtration (40-70% reduction):

- Filter needs replacement immediately
- Verify electrode with standards to confirm electrode is working
- Check for channeling or bypass in filter media

Poor/No Filtration (<40% reduction):

- Filter failure—replace immediately
- Verify electrode against known standards to rule out electrode problem
- Check system setup for proper water flow through filter media

SECTION 3: ELECTRODE MAINTENANCE SCHEDULE

Frequency	Task	Electrodes Affected
Before each use	Visual inspection	All
Before each use	Rinse with distilled water	All
Weekly	Calibration check	ISEs, SPCEs
Weekly	Deep cleaning	GCEs, Carbon-based
Monthly	Electrochemical conditioning	BDD, Gold, Copper
Monthly	Membrane inspection	ISEs
Quarterly	Sensitivity verification	All
Semi-annually	Full overhaul/replacement	Screen-printed types

Frequency	Task	Electrodes Affected
Annually	Complete replacement	ISE membranes, Gold films

SECTION 4: TROUBLESHOOTING

Problem: No signal/peak from electrode

- Check electrode connection to meter
- Verify meter is powered and functioning
- Test electrode with known standard solution
- Visual inspection for cracks or damage
- Electrode may need replacement

Problem: Inconsistent readings

- Ensure adequate equilibration time before measurement
- Check for air bubbles in sample cell
- Verify temperature stability ($\pm 2^{\circ}\text{C}$)
- Clean electrode surface
- Recalibrate or condition electrode

Problem: Slower response time than normal

- Surface contamination: clean appropriately for electrode type
- Internal solution (ISEs): check level and clarity
- Ionophore degradation: may need electrode replacement
- Temperature too low: allow samples to warm to room temperature

Problem: Same peak heights in treated and untreated water

- Filtration system not working: check water flow path
- Filter completely saturated: replace filter media
- Electrode not functioning: test with standards
- Bypass in filter: check system setup

Problem: Electrode becoming discolored rapidly

- Copper electrodes oxidizing: clean more frequently, store in dry conditions
- ISE membrane clouding: membrane degradation, replace electrode
- Coating (BDD, Bismuth, Gold) peeling: physical damage or manufacturing defect, replace