Lab Records Audit Details

Lab Records Audit

- **Selection of records** to evaluate *data usability* or assess overall lab quality system
  - Project, program, permit or calendar period basis
- **Inspection of Records** – Major Audit Topics
  - Linkage between all associated documentation
  - Sample receipt (intake) and preservation verification
  - Verification of holding times for sample processing and analysis
  - Essential sample preparation steps (e.g. filtration, pH adjustment, digestion)
  - Initial and continuing instrument calibration and verification
  - Quality control procedures & evaluation of QC sample results
  - Calculating, reporting and qualifying sample analysis results
  - Evaluating MDLs and PQLs for NNC data

Evaluation of Sample Receipt, Preservation Check & Holding Times

- Criteria for preservations and holding times established in DEP SOP tables (based on 40 CFR rules, where applicable)
  - Is **pH preservation and chilling** verified upon laboratory sample receipt?
    - Records are checked for indication of verification of sample pH and temperature
    - Lab preservation check is evaluated independently of field preservation check
  - **Holding times** met?
    - Field record is audited to verify collection date and time
    - Lab analytical record is used to verify sample prep and analysis date and time, as applicable

Example records for sample receipt, preservation and holding time check

Verification of essential sample prep procedures

- **TKN/TP** - Digestion
- **Color**
  - pH adjustment as needed (pH 4 – pH 10)
  - **True Color** - Filtration (0.45 µm pore size or smaller)
  - Dilution of samples above 100 CU
- **Chlorophyll**
  - **Filtration & extraction**
    - Date/time of filtration, frozen storage conditions for filters, date/time of extraction
  - **Acidification** for pheophytin correction
    - Acid normality (correct volume of 0.1N HCl added)
    - Read sample 90 seconds after acidification
- **Nitrate + Nitrite (NO_x)**
  - pH adjustment as needed (pH 5– pH 9)

Example sample prep records

Calibration Evaluation – Example Topics

- Minimum number of standards used
- Corr. Coeff. ≥ 0.995 where applicable
- "Second-source" additional standard to verify initial calibration
  - In-house calibration standard or external-source standard reference material (SRM) or QC Check Sample with a verified concentration
  - In-house calibration standards prepared from a different lot or vendor
- All verifications within specified acceptance range (method or DEP default criterion)
- Sample and QC results "bracketed" between acceptable calibration verifications
- Other method-specific protocols (e.g. titrant standardizations)

Calibration Evaluation – Example: ***EPA 353.2/SM 4500-NO_3^- F Nitrate-Nitrite (NOx)***

- Calibration curve: 3 standards & blank
- Correlation coefficient: ≥ 0.995
- Second-source standard recovery: ± 10% of assigned value
- Initial & continuing calibration verification standard recovery (ICV/CCV): ± 10% of assigned value

Calibration Evaluation – Example: ***SM 2120B Color (Platinum Cobalt)***

- Number of calibration standards: 9
- Calibration standard values (CU): 5, 10, 15, 20, 25, 30, 40, 50, 100
- Color comparators verified against platinum-cobalt standards

Calibration Evaluation – Example: ***EPA 310.1/SM 2320B Alkalinity***

- Sulfuric acid titrant standardized with sodium carbonate solution
  - Verify pre-formulated titrant normality per vendor specifications
  - Autotitrator programmed and calibrated per manufacturer instructions
    - pH calibration and/or verification with pH buffer solutions
    - Autotitrator check with sodium carbonate standards in lieu of manual titration and standardization (verification standards)





Example records for calibration evaluation

QC Procedures and Evaluation of QC Results

- Evaluation of QC **blanks**
  - Method blanks, other analytical blanks
  - Field-QC blanks (identity may not be known to lab)
- Evaluation of lab control **spikes** (LCS) and matrix spikes (MS)
  - Spike amounts evaluated relative to concentrations of audited samples
- Evaluation of LCS **duplicates**, MS duplicates and sample duplicates
- Evaluation of **other designated QC samples** (e.g. QC check samples)
- All QC samples "bracketed" or batched with sample results
  - Method-required frequencies, where specified

QC Evaluation – Example: ***EPA 351.2/SM 4500-N_{org} D Total Kjeldahl Nitrogen***

- **Blanks** – detections (≥ MDL) in method blanks or other blanks with blank concentrations > 10% of sample concentration values
- **Spikes** within 90% - 110% of expected values, per method criteria
- **Duplicates** ≤ 20% relative percent difference (RPD) or percent relative standard deviation (%RSD) or per method criteria for control limits established by lab
- **Other QC samples** within 10% of assigned values, per method

QC Evaluation – Example: ***SM 2120B Color (Platinum Cobalt)***

- **Replicate aliquots** of each sample analyzed
- **Duplicates** (including sample prep) at 10% frequency
- **QC Check Sample** (if used)

QC Evaluation – Example: ***SM 2320 B Alkalinity***

- **QC check sample** (if run)
  - Vendor acceptance criteria
- **LCS (LFB)** – (if run)
  - Second-source stock
  - In-house control limits (default 80% - 120% R)
- **Sample duplicates** – 5% frequency
  - In-house control limits (default ≤ 20% RPD)

QC Evaluation – Example: ***EPA 446.0/SM 10200H Chlorophyll a***

- **Method Blank**
  - Includes blank filter
  - Should produce no detection for chlorophyll *a*
  - Qualify sample results if blank >10% of sample values
- **QC Check Sample**
  - Vendor-supplied pigment (assayed for Chlorophyll *a)*
  - Analyzed concentration in range of sample extracts
  - Acidified for pheophytin correction step
  - Default control limits: 80% - 120% of expected value

Example records for QC evaluation

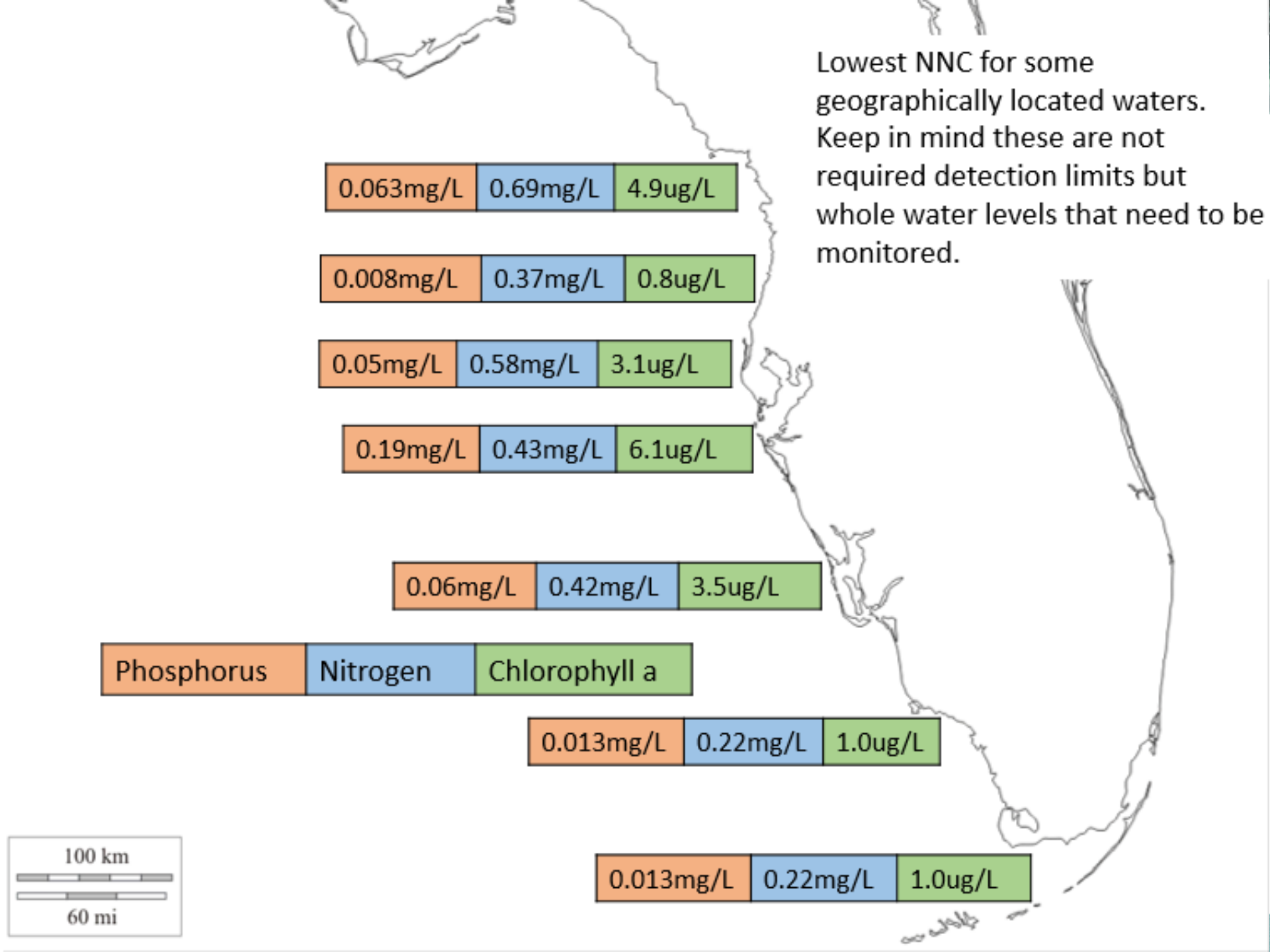
Evaluation of calculations, reporting and qualifying sample analysis results

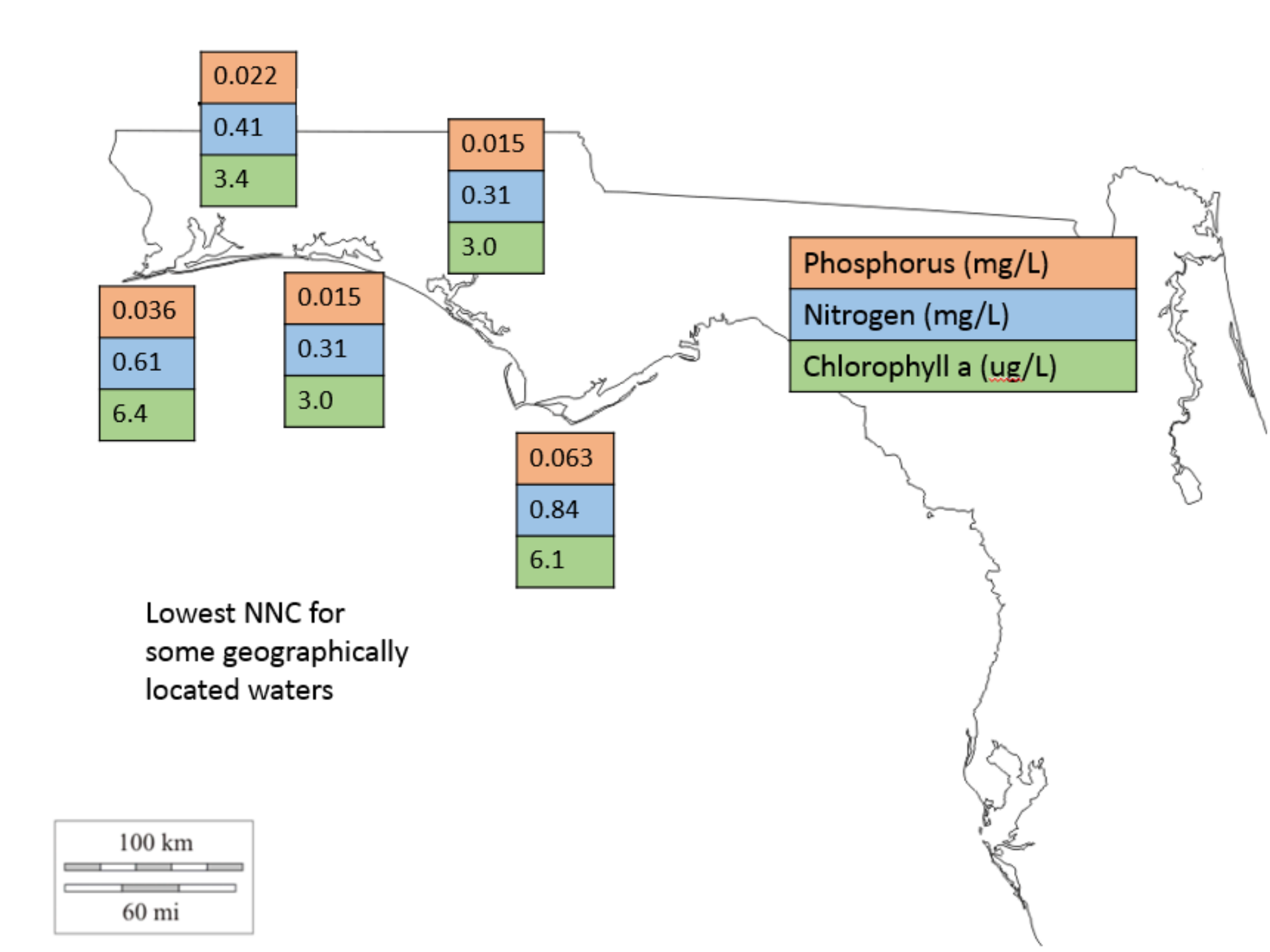
- Sample responses from the instrumentation are compared to standard responses (e.g. TKN, TP, NOx)
  - Sample responses or concentrations bracketed by standard responses
- Sample results calculated per method requirements
  - Corrections for sample dilutions applied to calculations
- Data qualifier codes (per 62-160) appropriately reported
- Comparison of reported results:
  - Reported data accurately reflect resulting calibration verifications, QC sample measurements and data reduction steps
  - Reported sample values and qualifier codes corroborate results reported to DEP
    - Comparison with STORET or other database records

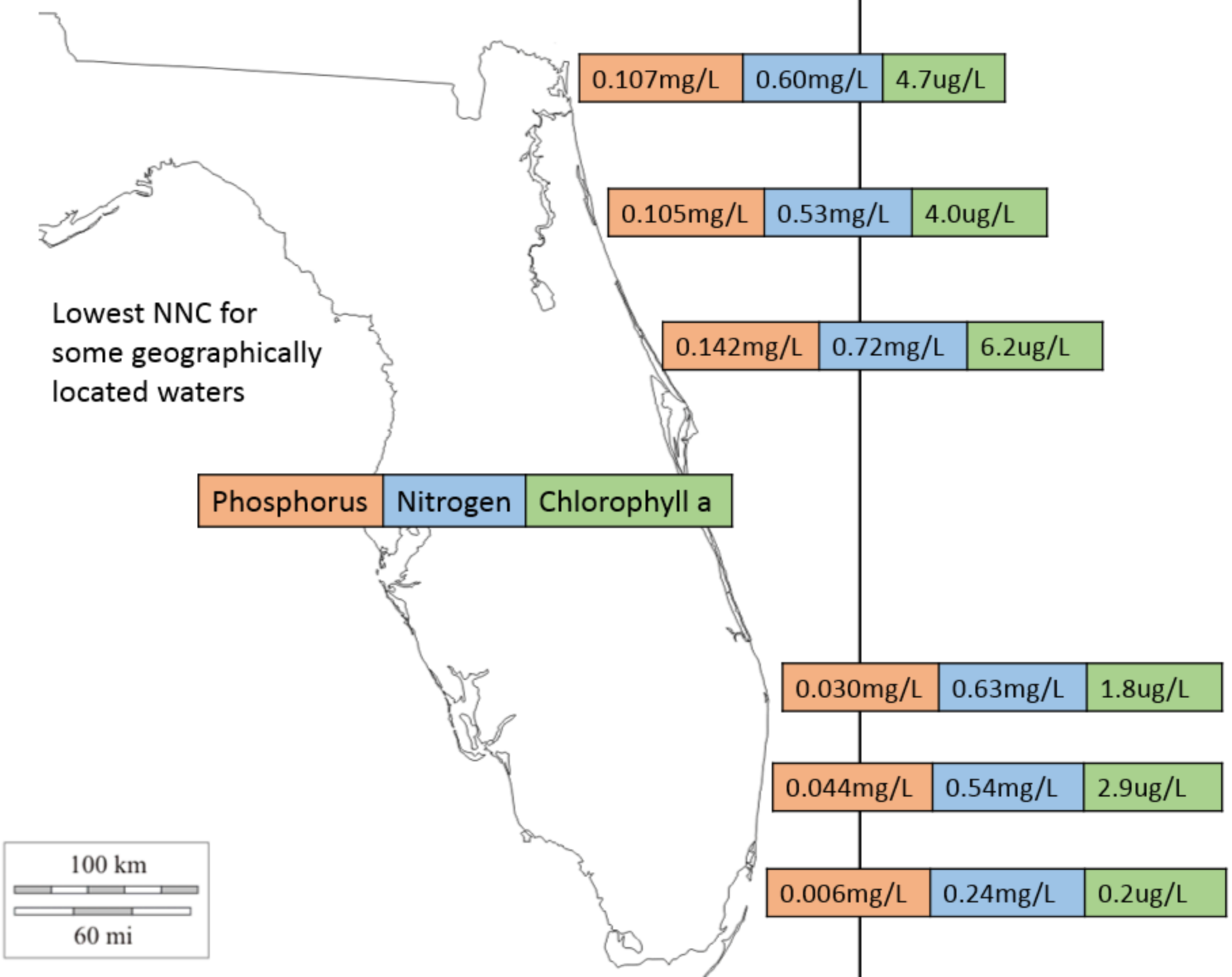
Example records for calculation and reporting of results

Evaluation of analytical sensitivity – MDLs & PQLs

- Reported laboratory **MDLs and PQLs evaluated for *data usability*** as specified by audit purpose and data quality objectives
- Example: **Sample analysis requires detections at appropriate limits for surface water monitoring for NNC criteria (62-302 FAC)**
  - Some or all typical analytical methods will have sufficient sensitivity to detect analytes at NNC criteria concentrations
    - Audit will evaluate whether most sensitive available method was used for analyses of audited samples *per data usability requirements for the selected samples*
  - TP methods may not be sensitive enough for some estuary samples
  - Chlorophyll fluorometric method may be required for some estuary samples


Lowest NNC for some geographically located waters. Keep in mind these are not required detection limits but whole water levels that need to be monitored.
0.063mg/L
0.69mg/L
4.9ug/L
0.008mg/L
0.37mg/L
0.8ug/L
0.05mg/L
0.58mg/L
3.1ug/L
0.19mg/L
0.43mg/L
6.1ug/L
0.06mg/L
0.42mg/L
3.5ug/L
Phosphorus
Nitrogen
Chlorophyll a
0.013mg/L
0.22mg/L
1.0ug/L
0.013mg/L
0.22mg/L
1.0ug/L
100 km
60 mi


0.022
0.41
3.4
0.015
0.31
3.0
0.036
0.61
6.4
0.015
0.31
3.0
0.063
0.84
6.1
Phosphorus (mg/L)
Nitrogen (mg/L)
Chlorophyll a (ug/L)
Lowest NNC for some geographically located waters
100 km
60 mi


Lowest NNC for some geographically located waters
Phosphorus
Nitrogen
Chlorophyll a
0.107mg/L
0.60mg/L
4.7ug/L
0.105mg/L
0.53mg/L
4.0ug/L
0.142mg/L
0.72mg/L
6.2ug/L
0.030mg/L
0.63mg/L
1.8ug/L
0.044mg/L
0.54mg/L
2.9ug/L
0.006mg/L
0.24mg/L
0.2ug/L
100 km
60 mi