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U.S. GEOLOGICAL SURVEY
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NATIONAL WATER QUALITY LABORATORY TECHNICAL MEMORANDUM 2024.02

September 23, 2025

Subject: Revision of prep dates and analysis dates reported for chlorophyll, plant pigments, MBAS, and pesticides, for 33,381 results from samples collected between May 7, 2020, and August 3, 2022. A revision to prep dates also changed the holding-time calculation for many results.

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Distribution: General Public

Revision: Not applicable

PURPOSE

This memorandum alerts users of the U.S. Geological Survey National Water Quality Laboratory (NWQL) of revisions to results provided through the Water Data for the Nation portal (WDFN, https://waterdata.usgs.gov/download-samples/#dataProfile=site).

BACKGROUND

The laboratory preparation (prep) dates and laboratory analysis (analysis) dates reported for the results in scope of this revision were revised in the National Water Quality Laboratory (NWQL) Laboratory Information Management System (LIMS) on July 14, 2025. Each of the methods impacted has an allowable hold time, which is defined as the maximum time(s) that a sample may be held prior to preparation or analysis and its analytical results still be considered valid. Results that are produced outside of the allowable hold time may be less accurate than results

produced within the allowable hold time due to degradation of the target compounds between sample collection and laboratory preparation. For some organic analysis, holding times can be reset from the date of sample extraction to the date of sample analysis. If no extraction or sample preparation is required, the analysis holding time is based on the date of sample collection. Revisions to preparation dates and analysis dates can affect the computation of actual time elapsed relative to allowable holding times. When the actual time elapsed exceeds the allowable holding time for the method, the NWQL releases the data with a qualifier of "[@ - holding time exceeded]" in the "LabInfo_LaboratoryComment" field. This revision impacts the application of the holding time exceedance qualifier as it corrects the preparation and analysis dates of the samples.

IMPACT ON NATIONAL WATER INFORMATION SYSTEM

The NWQL has identified an error that affects 33,381 results released from samples collected between May 7, 2020, and August 3, 2022. All numerical results transferred from the instruments to the NWQL LIMS were accurate; however, either the prep date or the analysis date (or in some cases, both) were incorrect. This led to some results that were within holding times being erroneously qualified with the "[@ - holding time exceeded]" and other results that exceeded holding times were not qualified with "[@ - holding time exceeded]." The resulting erroneous application of the "@" value-qualifier code to results within holding times and the non-application of the "@" value-qualifier code to results exceeding holding times could affect the interpretation of those results.

This communication may precede any changes to Water Data for the Nation; internal customers will be notified when the updated result files will be available for manual processing for final release to the public. If released to the public, analytical results within scope of this revision previously reported as "Accepted" in the "ResultStatusIdentifier" field will revert to "Provisional" as Water Science Centers re-review their data. In some instances, the preparation date and or the analysis date could not be verified. If the holding time could not be calculated due to this issue, the "@" value-qualifier code was added to the "LabInfo_LaboratoryComment" field out of an abundance of caution. Results will be updated with one of the following comments in the "DataQuality_ResultComment" field:

1. Corrected prep date and removed "@: holding time exceeded" value qualifier code on 07/14/2025. See NWQL Technical Memorandum 2024.02.
2. Corrected prep date and added "@: holding time exceeded" value qualifier code on 07/14/2025. See NWQL Technical Memorandum 2024.02.
3. Prep date could not be verified and the "@: holding time exceeded" qualifier was applied out of an abundance of caution on 07/14/2025. See NWQL Technical Memorandum 2024.02.
4. Corrected prep date on 07/14/2025. See NWQL Technical Memorandum 2024.02.
5. Analysis and prep date could not be verified and the "@: holding time exceeded" qualifier was applied out of an abundance of caution on 07/14/2025. See NWQL Technical Memorandum 2024.02.

6. Analysis date could not be verified and the "@: holding time exceeded" qualifier was applied out of an abundance of caution on 07/14/2025. See NWQL Technical Memorandum 2024.02.
7. Prep date could not be verified and the prep date was revised to "null" on 07/14/2025. See NWQL Technical Memorandum 2024.02.

SCOPE

This revision includes 33,381 results for environmental samples analyzed using laboratory method codes LCM60, 00050, FL016, SPEC2, GCM39, GCM14, GCM40, GCM35, and GCM32 for samples collected between May 07, 2020, and August 3, 2022. Method codes, along with citations and holding times, are outlined below:

- LCM60: Pesticides in filtered water by direct aqueous injection and high performance liquid chromatography/tandem mass spectrometry (LC/MS/MS) analysis (Method citation: https://doi.org/10.3133/tm5B11). Holding time from sample collection to laboratory preparation is 14 days.
- 00050: Plant pigments in algae by EPA Method 445.0 (fluorescence) modified by use of sonication for sample extraction. Holding time from sample collection to laboratory preparation is 24 days. Holding time from sample collection to laboratory analysis is 24 days.
- FL016: In vitro determination of chlorophyll a and pheophytin a in algae by maceration with acetone extraction and fluorescence analysis (EPA Method 445.0 modified by the use of acetone for the extraction). Holding time from sample collection to laboratory preparation is 24 days. Holding time from sample preparation to laboratory analysis is 1 day.
- SPEC2: MBAS by Spectrophotometry (Method citation: https://doi.org/10.3133/ofr95189). Holding time from sample collection to laboratory analysis is 7 days.
- GCM39: Pesticides, moderate use, filtered water, laboratory solid phase extraction, gas chromatography/mass spectrometry with selected ion monitoring (Method citation: https://doi.org/10.3133/ofr02462). Holding time from sample collection to laboratory preparation is 7 days. Holding time from sample preparation to laboratory analysis is 40 days.
- GCM14: Pesticides, moderate use, filtered water, solid phase extraction, gas chromatography/mass spectrometry with selected ion monitoring (additional constituents not included/described in the published method) (Method citation: https://doi.org/10.3133/ofr02462). Holding time from sample collection to laboratory preparation is 7 days. Holding time from sample preparation to laboratory analysis is 40 days.
- GCM40: Pesticides, moderate use, filtered water, solid phase extraction, gas chromatography/mass spectrometry with selected ion monitoring (NWQL custom method before Schedule 2002 method was approved) (Method citation: https://doi.org/10.3133/ofr02462). Holding time from sample collection to laboratory preparation is 7 days. Holding time from sample preparation to laboratory analysis is 40 days.

- GCM35: Pesticides, high use, filtered water, solid phase extraction at lab, gas chromatography/mass spectrometry with selected ion monitoring (Method citation: https://doi.org/10.3133/ofr02462). Holding time from sample collection to laboratory preparation is 7 days. Holding time from sample preparation to laboratory analysis is 40 days.
- GCM32: Pesticides in Water by Solid-Phase Extraction and GC-MS (Method citation: https://doi.org/10.3133/ofr02462). Holding time from sample collection to laboratory preparation is 7 days. Holding time from sample preparation to laboratory analysis is 40 days.

CONCLUSION

The NWQL strives to ensure that analytical results delivered to customers are of known and documented quality through adherence to its Quality Management System (QMS) and policies aligned with the USGS Fundamental Science Practices. The NWQL has implemented corrective actions to prevent reoccurrence of this issue.

Disclaimer: Any use of trade, product, or firm names is for descriptive purposes only and does not imply endorsement by the U.S. Government.

Contacts: Please direct feedback or questions to labhelp@usgs.gov.

Key words: revision; pesticides; holding-time violation; chlorophyll; pheophytin

Distribution: by Rapi-Note announcement posted to NWQL USGS-visible intranet, and https://water.usgs.gov/water-resources/nwql-memos/ (publicly accessible).

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