ANALYSIS OF MERCURY IN WATER BY THERMAL DECOMPOSITION, AMALGAMATION, AND ATOMIC ABSORPTION SPECTROPHOTOMETRY

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1. SCOPE AND APPLICATION

This standard operating procedure (SOP) details the analysis of mercury in water samples by thermal decomposition, amalgamation, and atomic absorption (TDA/AAS). The calibration range is 0.03 ng – 15 ng (0.1 µg/L – 50 µg/L). The method detection limit (MDL) is 0.03 ng (0.1 µg/L). The practical quantitation limit (PQL) is 0.09 ng (0.3 µg/L). Laboratory tests covered by this SOP are W-HG-TDA-R and W-HGTDA-F-R.

2. SUMMARY OF THE METHOD

2.1 This SOP is based on EPA method 7473, Revision 0.

2.2 Controlled heating in an oxygenated decomposition furnace is used to liberate mercury from aqueous samples in the Milestone DMA-80 instrument. The sample is pipetted into a quartz boat and placed on the autosampler. The sample is dried and then thermally and chemically decomposed within the decomposition furnace. The decomposition products are carried by flowing air to the catalytic section of the furnace. Oxidation is completed and halogens and nitrogen/sulfur oxides are trapped. The remaining decomposition products are then carried to an amalgam trap that selectively traps mercury. The analytical system is flushed with flowing air to remove the remaining gases and decomposition products, followed by the rapid heating of the amalgam trap, releasing the mercury vapor. The mercury vapor is carried by flowing air through absorption cells positioned in the light

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path of a single wavelength atomic absorption spectrophotometer. Absorbance is measured at 253.7 nm as a function of mercury concentration.

2.3 The Tricell DMA-80 contains three absorption cells with different path lengths for measuring varying concentrations of mercury in the sample. The DMA-80 software automatically chooses which peak to use for sample measurement based on the absorbance. Cell 0 is used for absorbance results of up to 0.5. Cell 1 is used for absorbance of up to 0.85. Cell 2 is used for absorbance of up to 1.0.

Note: Cell 2 is not used during the analysis of water samples. Samples with absorbance greater than 0.85 will be diluted to bring the absorbance into the range of Cell 1.

2.4 Co-absorbing gases, which are common interferences, such as free chlorine and certain organics, are purged from the analytical system after release by the decomposition furnace and the mercury vapor is selectively trapped on the amalgam trap.

2.5 Memory effects: Mercury vapor may remain in the decomposition tube, amalgam trap, or absorbance cells and be released in a subsequent analysis resulting in a positive bias. For example, this may occur when a low concentration sample is analyzed after a sample of high mercury content. To prevent memory effects, the DMA-80 automatically runs a cleaning cycle after samples that contain more than 9 ng of mercury in the water matrix and will continue to analyze auto blanks until the auto blank value is below 0.01 ng.

3. APPARATUS AND EQUIPMENT

3.1 Milestone DMA-80 instrument, serial number 14041581.

3.2 Milestone DMA-80 instrument, serial number 15081813.

3.3 Quartz Boats, part number, DMA8347.

3.4 Eppendorf pipette - 100 – 1000 µL adjustable volume.

3.5 Finnpipette, 2 – 10 mL adjustable volume.

3.6 Class A Volumetric Flasks – various volumes.

4. REAGENTS AND CHEMICALS

See DEP SOP MT-069 for standard and reagent documentation, labeling requirements, and expiration dates. All bottles of reagents and standards (purchased and prepared) in the laboratory are required to have laboratory information management system (LIMS) labels which includes a serial number.

4.1 Catalyst – The catalyst is a consumable and is considered a reagent. Conditioning procedure for the catalyst if it is new or has not been used for a long time:

4.1.1 Analyze 100 mg of flour using a max start temperature of 200 °, a one minute drying time at a temperature of 200 °C, and a purge time of 60 s.

4.1.2 Analyze 0.1 mL of 5 % HNO_3 using a max start temperature of 200 °C, a 2 minute drying time at a temperature of 650 °C, and a purge time of 60 s.

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The catalyst does not have an expiration date. The DMA-80 software will display a message when it is time to install a new catalyst. This is a general message based on the number of the cycles the instrument has run, but the lifetime of the catalyst depends on the type of samples analyzed and will vary for each catalyst.

When a new catalyst is installed, log the catalyst in to the LIMS standard prep tracker to generate a unique ID. Enter the identity, manufacturer, and lot number. Print the label which contains the unique ID for the catalyst and place it in the maintenance logbook. Document the catalyst installation date in the maintenance logbook.

4.2 Deionized (DI) water - Available from plastic spigots in the laboratory.

4.3 Trace Metal Grade Hydrochloric Acid (HCl) - The purchased acid is stored in the fume hood cabinet and the expiration date is provided by the manufacturer.

4.4 Standards - Preparation of all Hg standards must be documented in the LIMS standard prep tracker. A label should be printed from the standard prep tracker that shows the identity of the standard, preparation date, expiration date, and serial number. See DEP SOP MT-069 for standard documentation requirements and expiration dates.

4.4.1 Primary Commercial Stock Standard 1000 mg/L - Commercially available inorganic mercury stock from SPEX, Accustandard, Baker, Baxter, Fisher, VSR, etc. The expiration date is assigned by the manufacturer and the standard is stored at room temperature. This primary stock standard is used to prepare the calibration standards.

4.4.2 WTDA-1, 10 mg/L standard - Pipette 1.0 mL of the 1000 mg/L purchased primary standard into a 100 mL volumetric flask containing 50 mL of DI water. Add 5.0 mL of HCl. Dilute to volume with DI water and mix well. The expiration date for this prepared standard is 60 days from preparation and it is stored at room temperature.

4.4.3 WTDA-2, 1.0 mg/L standard – Pipette 0.1 mL of the 1000 mg/L purchased primary standard into a 100 mL volumetric flask containing 50 mL of DI water. Add 5.0 mL of HCl. Dilute to volume with DI water and mix well. The expiration date for this prepared standard is 60 days from preparation and it is stored at room temperature.

4.4.4 WTDA-3, 100 µg/L standard - Pipette 1.0 mL of WDTA-1 (10 mg/L) into a 100 mL volumetric flask containing 50 mL of DI water. Add 5.0 mL of HCl. Dilute to volume with DI water and mix well. The expiration date for this prepared standard is 60 days from preparation and it is stored at room temperature.

4.4.5 WTDA-4, 50 µg/L standard - Pipette 0.5 mL of WTDA-1 (10 mg/L) into a 100 mL volumetric flask containing 50 mL of DI water. Add 5.0 mL of HCl. Dilute to volume with DI water and mix well. The expiration date for

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this prepared standard is 60 days from preparation and it is stored at room temperature.

4.4.6 WTDA-5, 40 µg/L standard - Pipette 0.4 mL of WTDA-1 (10 mg/L) into a 100 mL volumetric flask containing 50 mL of DI water. Add 5.0 mL of HCl. Dilute to volume with DI water and mix well. The expiration date for this prepared standard is 60 days from preparation and it is stored at room temperature.

4.4.7 WTDA-6, 30 µg/L standard - Pipette 0.3 mL of WTDA-1 (10 mg/L) into a 100 mL volumetric flask containing 50 mL of DI water. Add 5.0 mL of HCl. Dilute to volume with DI water and mix well. The expiration date for this prepared standard is 60 days from preparation and it is stored at room temperature.

4.4.8 WTDA-7, 25 µg/L standard - Pipette 0.25 mL of WTDA-1 (10 mg/L) into a 100 mL volumetric flask containing 50 mL of DI water. Add 5.0 mL of HCl. Dilute to volume with DI water and mix well. The expiration date for this prepared standard is 60 days from preparation and it is stored at room temperature.

4.4.9 WTDA-8, 20 µg/L standard - Pipette 0.2 mL of WTDA-1 (10 mg/L) into a 100 mL volumetric flask containing 50 mL of DI water. Add 5.0 mL of HCl. Dilute to volume with DI water and mix well. The expiration date for this prepared standard is 60 days from preparation and it is stored at room temperature.

4.4.10 WTDA-9, 15 µg/L standard - Pipette 0.15 mL of WTDA-1 (10 mg/L) into a 100 mL volumetric flask containing 50 mL of DI water. Add 5.0 mL of HCl. Dilute to volume with DI water and mix well. The expiration date for this prepared standard is 60 days from preparation and it is stored at room temperature.

4.4.11 WTDA-10, 10 µg/L standard - Pipette 0.1 mL of WTDA-1 (10 mg/L) into a 100 mL volumetric flask containing 50 mL of DI water. Add 5.0 mL of HCl. Dilute to volume with DI water and mix well. The expiration date for this prepared standard is 60 days from preparation and it is stored at room temperature.

4.4.12 WTDA-11, 5.0 µg/L standard - Pipette 0.5 mL of WTDA-2 (1.0 mg/L) into a 100 mL volumetric flask containing 50 mL of DI water. Add 5.0 mL of HCl. Dilute to volume with DI water and mix well. The expiration date for this prepared standard is 60 days from preparation and it is stored at room temperature.

4.4.13 WTDA-12, 3.0 µg/L standard - Pipette 0.3 mL of WTDA-2 (1.0 mg/L) into a 100 mL volumetric flask containing 50 mL of DI water. Add 5.0 mL

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of HCl. Dilute to volume with DI water and mix well. The expiration date for this prepared standard is 60 days from preparation and it is stored at room temperature.

4.4.14 WTDA-13, 1.0 µg/L standard - Pipette 0.1 mL of WTDA-2 (1.0 mg/L) into a 100 mL volumetric flask containing 50 mL of DI water. Add 5.0 mL of HCl. Dilute to volume with DI water and mix well. The expiration date for this prepared standard is 60 days from preparation and it is stored at room temperature.

4.4.15 WTDA-14, 0.4 µg/L standard - Pipette 0.4 mL of WTDA-3 (100 µg/L) into a 100 mL volumetric flask containing 50 mL of DI water. Add 5.0 mL of HCl. Dilute to volume with DI water and mix well. The expiration date for this prepared standard is 60 days from preparation and it is stored at room temperature.

4.4.16 WTDA-15, 0.2 µg/L standard - Pipette 0.2 mL of WTDA-3 (100 µg/L) into a 100 mL volumetric flask containing 50 mL of DI water. Add 5.0 mL of HCl. Dilute to volume with DI water and mix well. The expiration date for this prepared standard is 60 days from preparation and it is stored at room temperature.

4.4.17 WTDA-16, 0.1 µg/L standard - Pipette 0.1 mL of WTDA-3 (100 µg/L) into a 100 mL volumetric flask containing 50 mL of DI water. Add 5.0 mL of HCl. Dilute to volume with DI water and mix well. The expiration date for this prepared standard is 60 days from preparation and it is stored at room temperature.

4.4.18 Calibration Standards - The calibration standards are prepared from standards WTDA-4 – WTDA-16. Pipette 300 µL of each WTDA standard into a sample boat. The concentration for each calibration standard is shown below.

Note: The absorption cell used for sample measurement is automatically chosen by the DMA-80 software based on the absorbance. Cell 0 is used for absorbance of up to 0.5. Cell 1 is used for absorbance of up to 0.85.

4.4.18.1 Cell 0 Calibration Standards:

WSTD1 (0.03 ng / 0.1 µg/L) [300 µL of WTDA-16]

WSTD2 (0.06 ng / 0.2 µg/L) [300 µL of WTDA-15]

WSTD3 (0.12 ng / 0.4 µg/L) [300 µL of WTDA-14]

WSTD4 (0.3 ng / 1.0 µg/L) [300 µL of WTDA-13]

Note: WSTD4 also serves as WSTD4CCV

WSTD5 (0.9 ng / 3.0 µg/L) [300 µL of WTDA-12]

WSTD6 (1.5 ng / 5.0 µg/L) [300 µL of WTDA-11]

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WSTD7 (3.0 ng / 10 µg/L) [300 µL of WTDA-10]

4.4.18.2 Cell 1 Calibration Standards:

WSTD1 (0.03 ng / 0.1 µg/L) [300 µL of WTDA-16]

WSTD2 (0.06 ng / 0.2 µg/L) [300 µL of WTDA-15]

WSTD3 (0.12 ng / 0.4 µg/L) [300 µL of WTDA-14]

WSTD4 (0.3 ng / 1.0 µg/L) [300 µL of WTDA-13]

WSTD5 (0.9 ng / 3.0 µg/L) [300 µL of WTDA-12]

WSTD6 (1.5 ng / 5.0 µg/L) [300 µL of WTDA-11]

WSTD7 (3.0 ng / 10 µg/L) [300 µL of WTDA-10]

WSTD8 (4.5 ng / 15 µg/L) [300 µL of WTDA-9]

WSTD9 (6.0 ng / 20 µg/L) [300 µL of WTDA-8]

WSTD10 (7.5 ng / 25 µg/L) [300 µL of WTDA-7]

WSTD11 (9.0 ng / 30 µg/L) [300 µL of WTDA-6]

Note: WSTD11 also serves as WSTD11CCV

WSTD12 (12 ng / 40 µg/L) [300 µL of WTDA-5]

WSTD13 (15 ng / 50 µg/L) [300 µL of WTDA-4]

4.4.19 Second Source Standard (1000 mg/L) - Commercially available inorganic mercury stock from SPEX, Accustandard, Baker, Baxter, Fisher, VSR, etc. The expiration date is assigned by the manufacturer and the standard is stored at room temperature. This purchased standard must be purchased from a different vendor than the primary standard used to prepare the calibration standards.

4.4.19.1 WTDA-1C, 1.0 mg/L standard - Pipette 0.1 mL of the 1000 mg/L purchased second source standard into a 100 mL volumetric flask containing 50 mL of DI water. Add 5.0 mL of HCl. Dilute to volume and mix well. The expiration date for this prepared standard is 60 days from preparation and it is stored at room temperature.

4.4.19.2 WTDA-2C, 30 µg/L standard - Pipette 3.0 mL of TDA-1C (1 mg/L) into a 100 mL volumetric flask containing 50 mL of DI water. Add 5.0 mL of HCl. Dilute to volume and mix well. The expiration date for this prepared standard is 60 days from preparation and it is stored at room temperature.

4.4.19.3 WTDA-3C, 3.0 µg/L standard - Pipette 0.3 mL of WTDA-1C (1 mg/L) into a 100 mL volumetric flask containing 50 mL of DI water. Add 5.0 mL of HCl. Dilute to volume and

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mix well. The expiration date for this prepared standard is 60 days from preparation and it is stored at room temperature.

4.4.19.4 The second source standard is analyzed at two different concentrations for absorption cells 0 and 1.

4.4.19.4.1 WTDA-CA (0.9 ng / 3.0 µg/L) - Pipette 300 µL of WTDA-3C into a quartz boat.

4.4.19.4.2 WTDA-CB (9.0 ng / 30 µg/L) - Pipette 300 µL of WTDA-2C into a quartz boat.

5. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

5.1 Water samples are collected in 500 mL or 1000 mL polyethylene bottles with screw caps and preserved by acidifying with nitric acid to a pH below 2. The preservation is done immediately after the samples are collected, prior to shipment to the laboratory. The samples are stored at room temperature. The holding time is 28 days from sample collection to analysis.

6. SAMPLE ANALYSIS PROCEDURE

6.1 Open the air supply.

6.2 Turn the DMA-80 on with the switch on the front panel of the instrument.

6.3 Turn on laptop computer and open the DMA-80 software. Log in as an administrator with the password 123456, then press enter.

6.4 The instrument will require about 15 to 20 minutes of warm up time.

6.5 Calibration:

The calibration is performed initially and when any significant instrument maintenance is performed. For example, a new calibration should be performed after the decomposition tube or amalgam trap is replaced. A new calibration must also be performed if the daily calibration check does not pass (see section 6.6.4).

6.5.1 To create a new calibration curve, choose from ‘Method’ WaterStandard.m80 and go to ‘Calibration’. Clear the old calibration (do not delete) using the blank page icon and save the new one in the calibration folder.

6.5.2 Go to the ‘Measurement Sample’. Clear the old measurements using the blank page icon and create a new file for calibration analysis. Analyze each calibration standard twice. Open the small balance icon and choose ‘calibration’. The software will mark the small balance icon with letter ‘C’.

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6.5.3 To complete the program for calibration switch to the 'Measurement Results' window. Enter the concentration for each standard used along with the volume in mL in the 'Amount' column.

6.5.4 Save the file.

6.5.5 Push START to start analyzing calibration standards.

6.5.6 After measurements are finished, the instrument will stop. Save the results.

6.5.7 Go to 'Calibration' and choose fit calibration in every cell. Save the results. The calibration curve is non-linear. Select the 'square' fit.

6.6 Sample Analysis:

6.6.1 Under 'Sample Measurement' create a new file for the analysis.

6.6.2 Select the appropriate calibration under 'Measurement Links'.

6.6.3 Select the Water.m80 method under 'Measurement/Links'.

6.6.4 Daily Calibration Check for water samples - Analyze the following calibration standards at the beginning of every analysis. The results must be within 10% of their true value.

6.6.4.1 Cell 0 daily calibration check - WSTD4 (0.3 ng / 1.0 µg/L) and WSTD6 (1.5 ng / 5.0 µg/L).

6.6.4.2 Cell 1 daily calibration check - WSTD9 (6.0 ng / 20 µg/L) and WSTD12 (12 ng / 40 µg/L).

6.6.5 The sequence of samples and quality control samples to be analyzed, including the daily calibration check, are typed into the sample file. See Appendix A for an example sequence.

6.6.6 Invert each water sample several times to thoroughly mix the sample. Pour approximately 0.5 mL of sample into a disposable beaker and then pipette 300 µL from the disposable beaker to a quartz boat and place on the autosampler.

7. QUALITY CONTROL

See DEP SOP MT-058 for calculation of relative percent difference (RPD) and percent recovery. Contingencies for out-of-control data are listed in DEP SOP LB-027. The equations used for calculating recovery and relative percent difference are in the Laboratory's Quality Manual.

7.1 One method blank per batch of 20 samples. Prepare a solution of 5% HCl in DI water. Pipette 0.3 mL of the acidified DI water into a quartz boat. The result of the method blank must be below the MDL.

7.2 One practical quantitation level standard (PQL) 0.09 ng (0.3 µg/L). The PQL standard is prepared by adding 0.3 mL of WTDA-3 to a 100 mL flask half-filled with DI water and 5 mL of HCl.

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7.3 One laboratory fortified blank (LFB) per batch of 20 samples. Pipette 0.3 mL of WTDA-11 (5.0 µg/L) into a quartz boat. The acceptable recovery limits for the LFB is 80 – 120%.

7.4 Matrix spikes – Prepare a matrix spike by pipetting 5.0 mL of sample and 0.25 mL of WTDA-3 into a 15 mL centrifuge tube and mix well. Transfer 0.3 mL to a quartz boat with a pipette. Repeat with the same sample to create a duplicate matrix spike. The acceptable recovery limits for the matrix spikes is 80 – 120%. The RPD should be below 20%.

7.5 Continuous Calibration Verification (CCV) - Every 10 samples, standards WSTD4CCV and WSTD11CCV are analyzed to verify the calibration. The WSTD4CCV is used for cell 0. The STD11CCV is used for cell 1. If any of the CCV standards are outside of the acceptable recovery limits, data cannot be reported from the absorption cell affected without appropriate qualification. The acceptable recovery limits for CCVs is 80 – 120%.

7.6 Continuous Calibration Blank (CCB) - An instrument blank (5% HCl) is analyzed every 10 samples to verify no contamination is present and that no drift has occurred. Acceptable CCB results are between -0.03 ng and 0.03 ng.

7.7 Second Source Standard - The second source standard (see section 4.3.8) is used to verify the calibration. This standard is analyzed on every analytical run and the acceptable recovery limits are 90 – 110%.

7.8 The correlation coefficient of the calibration curve must be ≥ 0.995.

8. DATA ARCHIVAL

The Milestone instrument software does not automatically save the results from the analytical run. To distinguish files between the two DMA-80 instruments, a suffix is used. The ‘DMA’ suffix is for S/N 14041581. The ‘DMA2’ suffix is for S/N 15081813. See below for details.

8.1 DMA-80 (S/N 14041581): After measurements are complete, click File > Save and save the instrument software file on the hard drive of the instrument computer with the format 20170805w1-DMA.d80. The file name reflects the year, month, and day the samples were analyzed on. The letter following the date in the file name reflects the matrix of samples that were analyzed (‘w’ for water samples). The number after the letter in the file name indicates the sequence of analyses performed on that day. If more than one analysis is performed on one day, then the second file name should have a ‘2’ following the letter, and so on. Then, the file needs to be saved as a text file with ‘DMA’ appended to the file name, for example, 20170805w1-DMA.txt. The text file is saved to \\FLDEP1\dear\labs\lab3\Chem_Data\Inorganic\Mercury\DMA-80.

8.2 DMA-80 (S/N 15081813): After measurements are complete, click File > Save and save the instrument software file on the hard drive of the instrument computer

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with the format 20170805w1-DMA2.d80. The file name reflects the year, month, and day the samples were analyzed on. The letter following the date in the file name reflects the matrix of samples that were analyzed ('w' for water samples). The number after the letter in the file name indicates the sequence of analyses performed on that day. If more than one analysis is performed on one day, then the second file name should have a '2' following the letter, and so on. Then, the file needs to be saved as a text file with 'DMA2' appended to the file name, for example, 20170805w1-DMA2.txt. The text file is saved to \\FLDEP1\dear\labs\lab3\Chem_Data\Inorganic\Mercury\DMA-80.

8.3 Save the absorbance peaks for each analysis on the laboratory's computer network. From the Signal tab, click the print icon. Then click the icon that looks like multiple pieces of paper to print more than one sample per page. Click the print icon. Change the printer to 'XPS Document Write' and click OK. Save the file with the format '20170805w1-DMA Peaks'. Saving the file will take several minutes.

9. MAINTENANCE

9.1 During routine maintenance of the DMA-80, both the instrument and laptop computer should be turned off and disconnected from the power supply. Turn off the air supply. Allow the instrument to cool to room temperature before beginning maintenance.

9.2 Refer to the DMA-80 Operator Manual for replacement of the catalyst and the amalgam trap.

10. SAFETY/HAZARDOUS WASTE MANAGEMENT

10.1 Always wear a lab coat and safety glasses or protective eyewear while working in the lab. Nitrile gloves and a lab coat should be worn while working in the laboratory. Concentrated acids should be handled in a fume hood.

10.2 Many mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. Extreme care must be exercised in the handling of concentrated mercury standards.

10.3 Samples with a high organic content (e.g. oil saturated soil) should be analyzed with caution. The sample size should be scaled down as too large of a sample size may result in ignition of the sample within the decomposition tube.

11. REFERENCES

11.1 Milestone operator's manual MA122.

11.2 EPA Method 7473, Mercury in Solids and Solutions by Thermal Decomposition, Amalgamation and Atomic Absorption Spectrophotometry.

11.3 DEP SOP LB-027, Standard Operating Procedure for Reporting Qualified Data.

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11.4 DEP SOP MT-058, Procedure for Metals Data Review and Authorization.

11.5 DEP SOP MT-069, Metals Standard and Reagent Preparation Procedure.

11.6 Quality Manual for State of Florida Department of Environmental Protection Laboratory, located on the Chemistry Section's website.

11.7 Laboratory Health and Safety Manual, located on the Division of Environmental Assessment and Restoration intranet site.

11.8 DEP-SOP-001/01, FS 1000, General Sampling Procedures.

APPENDIX A

Example Sample Sequence

WSTD4
WTDA-CA
WSTD6
WSTD9
WTDA-CB
WSTD12
WSTD11CCV
WSTD4CCV
Method Blank (batch 1)
W-PQL
LFB
Sample
Matrix spike
Matrix spike duplicate
Sample
Sample
Sample
Sample
Sample
WSTD11CCV
WSTD4CCV
CCB
etc.

Appendix of Significant Changes

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