Preparation and Testing of Cadmium Chloride and Copper Chloride Reference Toxicant Stock Solutions

1. SCOPE AND APPLICATION

1.1. This method is based on procedures utilized within the FDEP Biology Laboratory. It is used to prepare solutions of toxicants used in bioassay standard reference toxicant (SRT) tests. With proper substitution and corresponding calculations, it can be used to prepare other metal solutions such as selenium.

2. SUMMARY OF THE METHOD

2.1. Materials are gathered and placed on lab table and cart. The chemical compound is weighed, placed in a labeled flask and dissolved in deionized water. The stock solution and working solutions prepared from the stock solution are stored in the walk-in refrigerator. An aliquot of the working solution is diluted and submitted for chemical analysis to confirm the proper concentration of the toxicant. Preparation and analysis information is recorded in the *Tox Record of Preparation Logbook*.

WARNING: DO NOT USE ALUMINUM WEIGH BOATS – A violent chemical reaction can occur especially when copper chloride is placed into an aluminum weigh boat and rinsed into a flask.

3. INTERFERENCES

3.1. Calibration errors or improper weighing procedures. Contaminated spatulas, weigh boats, or glassware. Contaminants in the water or chemical compound being used. Failure to properly store prepared solutions.

4. EQUIPMENT AND SUPPLIES

4.1. Supplies – one set per toxicant being prepared

4.1.1. PLASTIC Weigh boat only

4.1.2. Volumetric flasks with stoppers, 100 mL, 2 each

4.1.3. Magnetic stir bar

4.1.4. Magnetic stir bar retriever

4.1.5. 10 mL pipette

4.1.6. Colored tape, 2 strips

4.1.7. *Tox Record of Preparation* logbook and computer with access to the Laboratory Information Management System (LIMS)

4.1.8. Permanent black marker

4.1.9. Black waterproof pen

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4.1.10. Labeling tape

4.1.11. Parafilm®, glass or rubber stoppers to seal flasks NO ALUMINUM

4.2. Equipment

4.2.1. Balance capable of weighing 0.01 mg such as Mettler Toledo XA105DU (Calibrated per SOP BB-36)

4.2.2. Autoclave

4.2.3. Magnetic stir plate

5. REAGENTS AND STANDARDS

5.1. Reagents

5.1.1. Cadmium chloride ($CdCl_2$) - Use ACS grade at a minimum; purer compounds can and should be used when available.

5.1.2. Copper chloride ($CuCl_{2)}$ - Use ACS grade at a minimum; purer compounds can and should be used when available. Copper sulfate can be substituted for copper chloride if the amount needed to yield the same concentration of copper is calculated.

5.1.3. Autoclaved deionized (DI) water

6. METHOD – Prepare one toxicant at a time.

6.1. Use the black marker and colored tape to label each 100 mL volumetric flask with the name of the toxicant being prepared, the batch number, an expiration date, and the preparer's initials. Each toxicant should have two flasks. For each toxicant, label one flask "Stock" and one flask "Working Solution".

Note: The "batch #" is the date that the solution is prepared. Format is mm/dd/yy. The expiration date is 1 year from preparation date. The stock solution can be stored indefinitely if the QA/QC chemistry results of the working solution prepared from the stock solution are within acceptable limits (± 10% of nominal). The working solution is usually depleted within 3 months of preparation, but it can be stored for up to one year.

6.2. For the $CdCl_2$ "Stock" write "1 mg/mL cadmium" on the label. For the "Working Solution" write "100 µg/mL cadmium" on the label.

6.3. For the $CuCl_2$ "Stock" write "1mg/mL copper" on the label. For the "Working Solution" write "100 µg/mL copper" on the label.

6.4. Use the balance and PLASTIC weigh boat to weigh out the required amount of chemical compound and place in the appropriate 100 mL volumetric flask labeled "Stock". For cadmium chloride, weigh out 163 mg. For copper chloride, weigh out 268 mg.

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Note: WARNING – DO NOT USE ALUMINUM WEIGH BOATS. Do not weigh out the two toxicants at the same time. Prepare one toxicant first from start to finish and then the next toxicant to avoid any cross contamination between the two. If necessary, use autoclaved DI water to rinse the compound into the flask.

6.5. Fill the "Stock" flask containing the chemical compound with autoclaved DI water to the 100 mL mark. Place the stir bar in the "Stock" flask and place on the magnetic stir plate. Turn the stir plate on, setting the speed control to mid-range (adjust speed as necessary to achieve a moderate rotation of the stir bar).

6.6. When the compound has dissolved (no visible particles), turn off the stir plate and remove the flask. Use the magnetic stir bar retriever to remove the stir bar from the flask.

6.7. Using the 10 mL pipet, remove 10 mL of the solution from the flask and place it into the "Working Solution" flask. Fill the "Working Solution" flask to the 100 mL mark with autoclaved DI water. Stopper/Seal the flask and shake to mix the contents.

DANGER – DO NOT USE ALUMINUM to SEAL the FLASKS- USE GLASS, RUBBER, or PLASTIC (Parafilm®) only.

6.8. Store both flasks on the top, rear shelf of the walk-in refrigerator.

6.9. Record the preparation in the LIMS Standard Prep Tracker module. Print a label of the serial number created in Standard Prep Tracker and affix it to the appropriate flask.

6.10. For each new batch of stock solution or annually on the current stock solution, ask the Toxicity Workgroup supervisor to request in LIMS that a QA/QC analysis for the toxicant (cadmium or copper) be performed.

6.10.1. Place 1 mL of working solution into one liter of DI water, mix well and submit this sample to the Chemistry Section for analysis. The acceptable range is 100 ± 10 µg/L of toxicant (cadmium or copper as appropriate). If an analysis is out of this range, discard the prepared solutions and make new stock solutions. New solutions should then be analyzed by the Chemistry Section.

6.10.2. Record the analysis results in the *Tox Record of Preparation* logbook beside the proper batch number. Analysis results should also be recorded in the LIMS Standard Prep Tracker. File the analysis results (LIMS report) in the Toxicity QA/QC folder located in the file cabinet in office A216N.

7. DATA ARCHIVAL

7.1. Use a black, waterproof pen for all bench sheets and logbooks.

7.2. Record information in *Tox Record of Preparation* logbook and in the LIMS Standard Prep Tracker.

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7.3. To correct raw data entries, place a single line through the incorrect entry, write the corrected entry near the error, date and initial the correction. Do not overwrite entries.

7.3.1. The corrected entry is written as near its appropriate space as possible or else in the comment box on the appropriate bench sheet.

7.3.2. If the correction requires an explanation, write a comment and reference the error. Initial and date the explanation.

7.3.3. Use sequential alphabetical annotation to identify errors and corresponding comments (use A for first error and use A for corresponding explanatory comment; use B for second error, etc.).

8. QUALITY CONTROL

8.1. Refer to QA manual for corrective action and handling out-of-control data.

9. SAFETY/HAZARDOUS WASTE MANAGEMENT

9.1. Refer to the FDEP Laboratory Health and Safety Plan at https://www.fldepnet.org/content/dear/bureau-laboratories-safety-documents.

9.2. Read MSDS information for reagent handling and disposal.

10. REFERENCES

10.1. Laboratory Quality Manual http://publicfiles.dep.state.fl.us/DEAR/DEARweb/Labs/Lab_Library/lab_qualitymanual_2020.pdf

10.2. Laboratory Health and Safety Plan https://www.fldepnet.org/content/dear/bureau-laboratories-safety-documents

10.3. DEP SOP BB-36 *Calibration Use and Maintenance of Mettler Toledo XA105DU Balance*

APPENDIX OF CHANGES

01/25/2008 Format change to add Scope and Application, Summary of Method and Appendix of Changes

01/12/2009 Format change to add Interferences and References.

10/09/2012 Major Revision: Combined SOP's TA-09.19 and TA-09.20 and reformatted.

10/08/2013 Added XA105DU balance and XA105DU reference.

09/11/2014 Removed OHAUS balance reference.

02/11/2015 Formatted for 508 compliance.

08/18/2015 Added warning and updated links.

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09/15/2016 Updated format and links.

03/05/2018 This SOP was previously deactivated. It is now being reactivated. As a result, the SOP ID will change from TA-09.19-2.5 to TA-09.21-1.0. Revisions include LIMS Standard Prep Tracker; updated format and links.

02/28/2020 Updated links, SOP references, and made minor formatting changes.

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