

Title: Preparation, Use and Storage of Standard Solutions		Copy No: ##
SOP No.: 3.09/4.6/S	Effective Date: September 9, 2013	Location: ###

QSM Approval: _____

Preparation, Use and Storage of Standard Solutions

1. Introduction

This procedure provides instructions on the proper use, storage and preparation of standard solutions for the Chemical Analysis and Methods Unit.

2. Procedures

- 2.1 Stock solutions are prepared in a fumehood by authorized personnel.
- 2.2 Standard solutions are prepared from properly labelled stock solutions and certified reference material by designated personnel.
- 2.3 Upon receipt, stock solutions are recorded into the standards logbook including date of receipt, lot number, expiry date and supplier. The original copy of the Certificate of Analysis is dated with the time of receipt and archived with the Unit Head. A copy of the certificate may be kept in the laboratory.
- 2.4 Standard solutions are labelled accordingly with proper ID, concentration and dilution solvent. The initials of the analyst that prepared the solution and date of preparation and expiry are also recorded on the vial containing the solution.
- 2.5 The pipettes used to prepare standard solutions are recorded in the Standards Logbook.
- 2.6 The date, preparation procedure, the name of the analyst and if applicable the certificate expiry date are also recorded in the Standards Logbook.
- 2.7 Prior to use, standards are verified by GC/MS.
 - 2.7.1 If any individual analyte concentration in a standard solution is outside of the designated value range ($\pm 20\%$), the standard solution is re-calibrated or new solutions are prepared. The mean relative error in concentration for all analytes within a given standard solution must not exceed 10% or the standard must be recalibrated or a new solution prepared.
 - 2.7.2 Re-calibrated standards are re-labelled indicating the contents, concentrations and new calibration date or preparation date.
 - 2.7.3 Working standard solutions are verified at least every 6 months using standard reference materials, whenever possible.
 - 2.7.4 Any suspicious standards are also verified before use.

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- 2.8 Following verification of analyte concentrations, the standard is assigned a unique ID which is then recorded in the Standards Preparation Logbook and on the actual vial containing the standard.
- 2.9 Vials containing standard solutions are placed in a supporting device at all times.
- 2.10 All standard solutions are sealed and refrigerated at less than 10°C when not in use.
- 2.11 Prior to use, standard solutions are allowed to warm up to ambient temperature.
- 2.12 After each use, the volume will be marked on the vial.
- 2.13 Standards are never to be lent to other laboratories. Instead an aliquot will be given to the lab in need.
- 2.14 Outdated standard solutions are disposed of as contaminated waste. Standards are considered outdated at the certificate expiry date or two years after the date of preparation, whichever comes first.
- 2.15 Outdated standard solutions may be re-calibrated using certified reference standards for use beyond the two-year period. Records of date prepared and new expiry date are maintained.

3. Standard Preparation

Whenever possible standards are prepared from stock solutions purchased from certified suppliers. When solutions are not available, the standards are prepared gravimetrically from pure compounds (at 98% purity or better) purchased from certified suppliers. Prior to use, standard concentrations are verified using GC/MS. The instrument used and the date concentrations were verified are recorded in the appropriate standard preparation logbook.

The following applies to the preparation of native, surrogate and recovery standard stock solutions and secondary dilution standard mixtures, and to the preparation of the instrument calibration standards for the target analytes specified.

3.1 PAH Stock Solutions

If stock solutions from certified suppliers are not available, PAH stock solutions are prepared from pure standard materials (98% purity or better). Each compound is allowed to equilibrate to room temperature and then transferred quantitatively into a pre-weighed or tared clean amber vial. Typically 3 mg of each compound is transferred using a clean spatula into the amber vial (3.5 mL capacity) and an appropriate volume of toluene is added via volumetric pipette (i.e. 3 mL for 3.000mg) to achieve a concentration of 1 mg/mL. The volume of toluene delivered is adjusted according to the weight of the compound in the vial. The vial is then capped and the stock standard solution is mixed thoroughly by shaking for at least 1 minute to ensure homogeneity prior to use.

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3.2 PAH Standard mixtures

PAH standard mixtures are prepared by the addition of the corresponding volumes of the individual stock solutions containing the compounds of interest made up to the appropriate volume with toluene for a desired concentration; 10 ppm typically. Additional dilution of the PAH standard mixture may be subsequently prepared using toluene. The volumes of each stock solution added and respective concentrations are recorded in the standard preparation logbook. Each mixture is prepared in toluene with a final volume ranging from 10 mL to 20 mL typically.

3.3 PAH Instrument Calibration Standards

Instrument calibration standards in toluene spanning the dynamic range of the GC-MS are prepared by combining the appropriate volumes of the surrogate, recovery and native standard stock solutions and/or mixtures and toluene for final concentrations ranging from 0.01 ppm to 25 ppm (i.e. typical native concentrations are 0.01, 0.05, 0.25, 1.0, 4.0, 10.0, 16.67, and 25 respectively and recovery and performance standard concentrations are typically 1 ppm). Instrument calibration standards are prepared in clean amber vials at a volume of 1.0 mL (typically). All volumes and solutions used as well as final concentrations are recorded in the standard preparation logbook.

3.4 PCB Standards

PCB stock solutions are prepared in the same way as the PAH stock solutions. Individual congeners are weighed out into pre-weighed or tared clean amber glass vials and the appropriate volume of iso-octane added to achieve a concentration of 1 mg/mL.

PCB mixtures are prepared from the PCB stock solutions by combining a known volume of each stock solution and adjusting the final volume accordingly using iso-octane. Typically a 100x dilution is prepared (i.e. 10 ppm).

Instrument calibration standards are prepared by further dilution of the PCB standard mixtures to achieve the necessary concentrations as per described in the corresponding method(s).

3.5 Dioxin/Furan Standards

Individual dioxin and furan stock solutions are purchased from a certified supplier typically at a concentration of 50 µg/mL. Each stock solution is transferred into a clean amber vial using a clean Pasteur pipette. A Dioxin/Furan mixture is prepared by combining know volumes of each stock solution into a clean amber

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vial and making up to volume using toluene to achieve a known concentration of each individual compound (typically from 1 to 4 µg/mL).

Instrument calibration standards are then prepared by dilution of the standard mixture to span the dynamic range of the instrument.

3.6 HPO Standards

HPO Stock solutions are prepared in the same way as the PAH stock solutions. Individual compounds are weighed out into pre-weighed or tared amber glass vials and the appropriate volume of iso-octane added to achieve a concentration of 1 mg/mL. Dilution of the HPO stock solutions may be subsequently prepared using iso-octane. Acetylated PCP and ¹³C₆-PCP solutions are prepared by dilution of the corresponding stock solution with iso-octane (typically a 100x dilution is prepared, 10ppm) followed by acetylation (for the acetylation procedure, see Method 3.09/*.*/*M, section 7.2).

Instrument calibration standards are prepared by combining the appropriate volumes of diluted stock solutions for HCB, ¹³C₆-HCB, OCS; acetylated PCP and ¹³C₆-PCP; d10-Fluoranthene (see Section 3.1) and iso-octane to achieve the necessary concentrations as per described in the corresponding method (3.09/*.*/*M).

4. GC/MS Analysis

- 4.1 Each standard solution prepared or received is assigned a unique identifier (CALYY-####) and entered in the appropriate logbook along with corresponding information.
- 4.2 When in use standard solutions are calibrated at least every six months.
- 4.3 For every batch of samples submitted for analysis for a given analyte group, a standard control solution consisting of the appropriate recovery (ies) and surrogate(s) standard(s) is also submitted. Stock solutions used and their respective volumes are clearly indicated on the corresponding tracking sheet.
- 4.4 Each standard control solution prepared is given a unique identifier (SRS- or SC-###).
- 4.5 After analysis all SRS or SC controls are stored in a refrigerator in a separate container labelled QA/QC.

5. Revisions

- Nov. 2003: Section 4.3 changed from every second batch to every batch.
Minor changes to document made to reflect current procedure.
- Nov. 2005: Lead Reviewer: David Harnish

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Section 3.3 – Updated concentrations data

- Feb. 2006: Insert Section 2.3: specify date of receipt, lot #, expiry date and supplier is documented in standards logbook and that certificate of analysis is stored with Section Head.
Section 2.4: Add date of preparation and initials of analyst to standard label.
- Feb. 2010: Lead Reviewer: Alison Walkey
Added Sections 2.7.4, 2.11, and 2.12.
Backdated Revisions history to original version.
- Feb. 2012 Lead Reviewer: Gary Poole
Insert Section 2.8: specify Unique ID is recorded on standard vial and in standard preparation logbook
- June 3, 2013 Lead Reviewer: Jennifer Verner
Section 2.3: corrected punctuation
Section 2.4: Add date of expiry to standard label
Removed revision history 2001 and older

Lead Reviewer: Jennifer Verner
Title: Technologist, Organic Laboratory

Approved by: May Siu
Title: Supervisor, Organic Laboratory