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Reagents

1. Introduction

Laboratory reagents are used in all procedural steps during the course of sample preparation. To ensure that reagents are of known quality and purity and free of contaminants and interferences the following procedures must be followed.

2. Reagents purchase and storage

2.1 All reagents are prepared as described in this SOP and/or each method.

2.2 New reagents are purchased in bulk to maintain a consistent lot number.

2.3 Whenever reagents with a new lot number are used, studies are undertaken to ensure the recovery of analytes is not impaired and that no contamination or interferences are present which may affect the analysis.

2.4 Upon receipt of new reagents, the date of receipt, quantity received, lot number and the name of the person receiving the goods, are entered into the reagent logbook. This date is also written on each reagent container.

2.5 Reagents are checked upon receipt to ensure quality is comparable with the last order.

2.6 Reagents are reordered from the same supplier with the same catalogue number whenever possible.

2.7 When a batch of reagent is prepared, the date of preparation and reagent lot number (s) are recorded in the reagent logbook.

2.8 Prepared reagents are stored in designated cabinets and labelled with reagent name, batch number if applicable, WHMIS label if required, date of preparation, date of expiry, and initials of preparer.

2.9 Expiry dates of reagents are typically 90 days from the date of preparation (except for XAD which is 30 days from date of drying).

2.10 Incompatible substances are stored apart (eg. acids and bases).

3. Preparation of Sodium Sulphate

Water can significantly alter the efficiency of adsorbents used for sample cleanup as well as affect the chromatographic separation of sample analytes by capillary gas

chromatography. Sodium sulphate is a critical component in the preparation of sample extracts to ensure adequate removal of water prior to sample cleanup and analysis.

3.1 Organic Lab

Granular anhydrous sodium sulphate, 10-60 mesh, Caledon catalogue # 8221-1-90 or equivalent

3.1.1 Pour sodium sulphate in a pre-cleaned column
3.1.2 Elute with two column volumes of dichloromethane
3.1.3 Discard dichloromethane
3.1.4 Elute with two column volumes of hexane
3.1.5 Discard hexane
3.1.6 Pour clean sodium sulphate into a pre-cleaned 1L beaker, cover loosely with acetone and hexane rinsed aluminum foil and allow to air dry overnight in fumehood
3.1.7 Dry in oven at approximately 110°C overnight
3.1.8 Store in oven at a temperature between 100°C and 300°C until ready for use

3.2 Ultra Trace Lab

Granular anhydrous sodium sulphate, 10-60 mesh, Caledon catalogue # 8221-1-90 or equivalent

3.2.1 Pour sodium sulphate in a pre-cleaned column
3.2.2 Elute with two column volumes of hexane
3.2.3 Discard hexane
3.2.4 Elute with two column volumes of dichloromethane
3.2.5 Discard dichloromethane
3.2.6 Pour clean sodium sulphate into a pre-cleaned 1L beaker, cover loosely with hexane and dichloromethane rinsed aluminum foil
3.2.7 Oven dry at 50°C for a minimum of one hour before conditioning overnight at 225°C
3.2.8 Store in a dessicator in a clean, labelled, screw-capped bottle fitted with a Teflon-lined cap

4. Preparation of Glasswool

Glasswool is used during sample filtration and cleanup for plugging the bottom of funnels and columns so that powder and granular reagents may be retained. Prior to use, glasswool must be properly cleaned to ensure that it is free of contaminants and moisture.

4.1 Organic Lab

Corning 3950, pyrex fibreglass, 8 micron or equivalent, Fisher catalogue # 11-388

4.1.1 Place glasswool in a large column
4.1.2 Elute with two column volumes of dichloromethane
4.1.3 Discard dichloromethane
4.1.4 Elute with two column volumes of hexane
4.1.5 Discard hexane
4.1.6 Place clean glasswool in a pre-cleaned 1L beaker, cover loosely with acetone and hexane rinsed aluminum foil and allow to air dry overnight in fumehood
4.1.7 Once dry, store in oven at a temperature ranging between 110° to 300°C, until ready for use

4.2 Ultra Trace Lab

Corning 3950, pyrex fibreglass, 8 micron or equivalent, Fisher catalogue # 11-388

4.2.1 Place glasswool in a large column
4.2.2 Elute with two column volumes of hexane
4.2.3 Discard hexane
4.2.4 Elute with two column volumes of dichloromethane
4.2.5 Discard dichloromethane
4.2.6 Transfer to a pre-cleaned 1L beaker, cover loosely with hexane and dichloromethane rinsed foil
4.2.7 Air dry in fumehood before conditioning overnight at 225°C in vented oven
4.2.8 Store in a clean, wide-mouth, glass-stoppered bottle.

5. Preparation of Silica

The following procedure ensures the removal of contaminants and the proper activation of silica used in the cleanup of sample extracts.

5.1 Organic Lab

Silica gel of 100-200 mesh size, Chromatographic grade, Fisher catalogue # S679-212

5.1.1 Pour silica into a large pre-cleaned glass column
5.1.2 Elute with the equivalent of two column volumes of methanol
5.1.3 Discard methanol
5.1.4 Repeat steps 5.1.2 and 5.1.3 using dichloromethane
5.1.5 Pour clean silica into a pre-cleaned 1 L beaker, cover loosely with acetone and hexane rinsed aluminum foil and allow to air dry, at least overnight, in fume hood

5.1.6 Place in oven at approx. 110°C overnight (minimum requirement) to dry
5.1.7 Prior to use, activate silica in an oven set between 240°C and 300°C for a minimum of 24 hours. Do not have more than half the beaker filled with silica to prevent the formation of noxious fumes from residual solvent
5.1.8 Store activated silica in the oven (240°C to 300°C) in properly labelled beaker

5.2 Ultra Trace Lab

Silica gel of 100-200 mesh size, Davisil grade 634, pore size 60A, Fisher catalogue # S734-1

5.2.1 Transfer approximately 350 g of silica (enough silica for 35 cleanup columns) to a large glass column
5.2.2 Elute with two column volumes of hexane
5.2.3 Discard hexane
5.2.4 Repeat steps 5.2.2 and 5.2.3 using dichloromethane
5.2.5 Oven dry the silica at 50°C for a minimum of one hour in a beaker loosely covered with hexane and dichloromethane rinsed aluminum foil before conditioning at 180°C for a minimum of 4 hours.
5.2.6 Store in a dessicator in a clean, labelled, screw-capped bottle fitted with a Teflon-lined cap

6. Preparation of XAD-2 Resin

XAD-2 resin is an adsorbent commonly used to trap semi-volatile organic compounds from stack emissions, ambient air and precipitation samples. It is essential that the resin be properly clean to remove potential interferences and any moisture that may affect its adsorbency. Amberlite XAD2, Supelco catalogue #1-0357

6.1 Extraction of XAD-2 Resin

6.1.1 Rinse XAD with the equivalent of three column volumes of deionized water and displace with methanol
6.1.2 Place clean glasswool on bottom of a clean soxhlet body, add the XAD resin and cover with more clean glasswool
6.1.3 Extract with methanol for 16 to 20 hours
6.1.4 Discard methanol and extract with dichloromethane for 16 to 20 hours
6.1.5 Discard dichloromethane and extract with cyclohexane for 16 to 20 hours
6.1.6 Rinse the XAD (while still in soxhlet body) with methanol (three cycles)
6.1.7 Store XAD under methanol into a pre-cleaned amber glass jar with a Teflon lined cap or cap lined with acetone and hexane rinsed aluminum foil

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6.2 Drying of XAD-2 Resin (for stack emissions and ambient air only)

6.2.1 Add clean XAD/methanol to a large column and elute with 2 column volumes of dichloromethane

6.2.2 Spread clean XAD onto a tray lined with acetone and hexane rinsed aluminum foil at a depth not exceeding 0.5 cm

6.2.3 Allow to air dry in fumehood for 1 to 2 hours to evaporate most of the solvent, then dry in an oven for 3 to 4 hours at approximately 110°C until dry (if required)

6.2.4 Transfer clean, dry, XAD to a clean amber glass bottle with a Teflon lined cap or cap lined with acetone and hexane rinsed aluminum foil

6.3 Proofing XAD-2 Resin

A portion of each batch cleaned is used as a method blank/proof.

7. Revisions

May 2008: Lead Reviewer: Alison Walkey
Section 2.8: dated with expiration date

Oct 2011 Lead Reviewer: Alison Walkey
Rearranged order of section 2
Section 2.2 Deleted "sample processing is interrupted"
Section 2.9 Changed expiry dates of reagents
Inserted Sections 3, 4, 5, and 6

June 2013: Lead Reviewer: Jennifer Verner
Section 6: Added: "and precipitation samples"
Section 6.2: added "for stack emissions and ambient air only"
Removed revision history 2001 and older

Lead Reviewer: Jennifer Verner
Title: Technologist, Organic Laboratory

Approved by: May Siu
Title: Supervisor, Organic Laboratory