Title: Preparation and Analysis of Sample Extract Dilutions by GC/MS – Instrumental Analysis Laboratory		Copy No: ##
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Preparation and Analysis of Sample Extract Dilutions by GC/MS - Instrumental Analysis Laboratory

1. Introduction

The following procedure ensures adequate dilution of samples for accurate target analyte measurement by GC/MS. This procedure applies to all sample extracts with analyte concentrations exceeding the dynamic calibration range of the instrument.

2. Procedure

Verify that the reported concentrations fall within established calibration ranges and that integration of analyte peaks was properly performed. Ensure that the performance/ recovery standard is present at an appropriate response level. Absent or low response may indicate high loading of closely eluting target analytes. Dilutions are required for any sample with a target analyte concentration exceeding the pre-established dynamic calibration range.

2.1 Preparation of Dilutions

2.1.1 All dilutions are prepared in autosampler vials.
2.1.2 Each dilution is typically prepared to a final volume of 400-500 µL.
2.1.3 For each dilution the corresponding information is recorded in the Sample Dilution Log Book and includes:

- Dilution factor
- Sample ID
- Batch ID
- TS#
- Date prepared
- Analyst
- Pipette(s) used
- Sample Extract volume
- Dilution solvent type and volume.

- Performance/recovery standard type and volume (if applicable i.e. dilutions >10)
- Final dilution volume

2.1.4 Dilutions ≤ 10 fold

2.1.4.1 For dilutions up to 10 fold do not add any additional performance/recovery standard unless omitted in the original extract.

2.1.4.2 Use the same dilution solvent as used in the corresponding daily calibration standard.

2.1.4.3 Example dilution (4 fold dilution of PAH extract)
100 µL sample extract
300 µL toluene
Final volume = 400 µL

2.1.4.5 No correction factor needs to be applied to results when using ISTD quantitation (Internal standard quantitation).

2.1.5 Dilution > 10 fold

2.1.5.1 Samples requiring a greater than 10 fold dilution require the addition of the corresponding performance/recovery standard to maintain a concentration level consistent with the original extract, typically 1ppm.

2.1.5.2 Example dilution (20 fold dilution of PAH extract)
20 µL sample extract
*38 µL d10-FLT (10 ppm)
342uL toluene
Final volume = 400 µL

2.1.5.3 (*Note: The corresponding aliquot of the original extract will contain a proportional quantity of recovery/performance standard which must be accounted for)

2.1.6 ISTD quantitation using performance/recovery standard (PAH):

The addition of performance/recovery standard when preparing extract dilutions requires that the appropriate correction factor be applied to the quantitative results, surrogate recoveries and detection limits

2.1.7 ISTD quantitation using surrogate standards (PAH, HPO, HCBD/CB, WHO PCB, Total PCB):

Addition of recovery standard will affect reported surrogate recoveries to which the appropriate correction factor must be applied according to the dilution performed. Do not apply any correction factor to the quantitative results of native analytes as both surrogates and native analytes are diluted proportionately.

3. Revisions

Aug 1999:	Author: Mylaine Tardif. New Document.
Oct 2001:	New header added Section 3 added
Nov 2003:	Small changes made to sections 2.1.4, 2.1.5.1, 2.1.6 and 2.1.7
Oct 2005:	Lead Reviewer:David Harnish Small grammatical and punctuation changes to text Section 2.1.6: Add "and detection limits" Section 2.1.7: Add 'WHO PCB' to section header
Oct 2009:	Lead Reviewer:David Harnish Section 2.1.7:Add PAH to section header
Oct 2013:	Lead Reviewer: David Harnish Removed reference to NAPS Arvida site in Section 1

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