

Title: PAH Analysis of Urban Dust Material (NIST_1649b)		Copy No: ##
SOP No.: 5.03/3.4/S	Effective Date: October 11, 2013	Location: ###

QSM Approval: _____

PAH Analysis of Urban Dust Material (NIST_1649b)

1. Introduction

The following procedure is used for the determination of PAHs in urban dust material (NIST 1649b). Urban dust material is analyzed periodically as a quality control measure in the determination of PAH Total C1 to C4 alkylated PAHs, dibenzothiophene (DBT) and Total C1 to C4 alkylated dibenzothiophenes (DBTs) in ambient air samples. Note from this point forward any reference to C1 to C4 alkylated PAHs also includes DBT and C1 to C4 alkylated DBTs. Reference material should be processed using same procedures as for samples.

2. Sample Preparation

2.1 Soxhlet Extraction

- 2.1.1 Accurately weigh approximately 0.100 g of urban dust material (NIST 1649b) onto a Teflon coated glass fibre filter for PAH, 0.300 g for PAH and PCDD/F analysis or 0.010g for NPAH analysis. Once weighed, fold the filter carefully into a beaker.
- 2.1.2 Spike with surrogates.
- 2.1.3 Place in a Soxhlet and extract 16-20 hours using dichloromethane with approximately 10% acetone for PAH, 80/20 cyclohexane/acetone for PAH and PCDD/F or dichloromethane for NPAH.
- 2.1.4 Filter through sodium sulphate into a round bottom flask.

2.2 MAP Extraction

- 2.2.1 Accurately weigh approximately 0.100 g (PAH analysis), 0.300 g (PAH and PCDD/F analysis) or 0.010 g (NPAH analysis) of urban dust material (NIST 1649b) onto a Teflon coated glass fibre filter.
- 2.2.2 Spike with surrogates.
- 2.2.3 Place in microwave extraction vessel, add 25 mL of 1:1 acetone:hexane.
- 2.2.4 Extract samples at 50% power to a maximum temperature of 100 °C for a duration of 10 minutes.
- 2.2.5 Allow samples to cool for at least 30 minutes (30 °C or below) before opening the vessels.

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2.2.6 Filter sample extracts along with three rinses of the extraction flask (20-30 mL hexane) through Na₂SO₄/filter paper apparatus to remove particulate matter, into a round bottom flask.

2.3 Cleanup Procedure

- 2.3.1 Concentrate raw extract to approximately 2 mL by rotary evaporation.
- 2.3.2 Follow the cleanup procedure as described in the appropriate method 3.02/*.*M

3 GC/MS Analysis

- 3.1 Sample extracts are submitted along with the corresponding sample tracking sheet to the Instrumental Analysis Laboratory.
- 3.2 Analyze the final extracts as described in method 3.03/*.*M.

4 Revisions

- Sept 1995: Author: Mylaine Tardif. New Document (SOP no. OL 10)
- Sept 1997: Revisions not recorded
- July 1999: Section 3.1: Word "Supervisor" removed
 2.1.1/2.2.1: add data for PAH and PCDD
 2.1.2/2.2.2: remove word PAH
 2.1.3: (add cyclohexane/acetone)
 Removed sections 2.3.3-2.3.6.
- 2001: New header added
- Nov 2003: 0.010 g for NPAH analysis added to section 2.1.1
 DCM for NPAH added to section 2.1.3
 0.010 g (NPAH analysis) added to section 2.2.1
 Header change to reflect change in ETC address
- Oct 2009: Lead Reviewer: David Harnish
 changed all references to NIST 1649 to NIST 1649b
- Oct 2013: Lead Reviewed: David Harnish
 Added reference to C1-C4 alkylated PAHs and DBTs in Section 1
 Changed cyclohexane to DCM w/10% Acetone in Section 2.13

Lead Reviewer: David Harnish
Title: Chemist, Chemical Analysis and Methods

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