Diisopropanolamine (DIPA) in Water and Soil Samples by HPLC - PBM

Parameter

Diisopropanolamine (DIPA) in Water and Soil

Analytical Method

Analysis by High Performance Liquid Chromatography with UV Detector (HPLC/UV).

Introduction

DIPA is a secondary alkanolamine which is a hygroscopic polar solvent that is completely miscible in water. DIPA has a wide variety of applications such as a solvent used in the Sulfinol process by the petroleum industry to remove acid gasses from natural gas streams through chemical absorption. The neutralizing capacity of DIPA salts, their high foaming properties and low level of skin irritation allow them to be commonly used as components of cosmetics, personal care products and detergents.

Method Summary

This method uses a derivatization step which transforms mono and di-ethanolamines into products with strong UV absorbency by reaction with 9-fluorenylmethylchloroformate (FMOC-CL). The derivatized products are injected directly into the liquid chromatography with UV detector and separated on an ODS column.

This method is performance-based. Laboratories may adopt alternative options to improve performance or efficiency provided that all stated performance requirements and prescribed (mandatory) elements are met.

MDL(s) and EMS Analyte Codes

Analyte	CAS Number	Approx. MDL	EMS Analyte Code
Diisopropanolamine (DIPA)	110-97-4	100 ug/L 0.1 mg/kg	defined on request

EMS Method Code(s)

***Refer to EMS Parameter Dictionary on the ministry website for all current EMS codes.

Matrix

Freshwater, Groundwater, Drinking water, Soil and Sediment

Interferences and Precautions

Solvents, reagents, glassware and other sample processing materials must be demonstrated to be free form interferences by analyzing a method blank. Any compounds that reacts with FMOC-CL and having a similar retention time as analysed ethanolamines and responds to a UV detector is an interference. Retention time alone is not proof of chemical identity. Analysis by an alternate HPLC column, rationing between fluorescence and UV detectors and confirmation by mass spectrometry are additional means of identification.

Contamination of the analytical system can occur after high level samples are analyzed. Analysts should be aware of the degree of carryover that occurs on their instrument system, and should take appropriate steps to prevent the occurrence of false positives.

Sample Handling and Preservation

Sampling should be done by qualified personnel. Samples must be collected and stored such that degradation or alteration of the sample is minimized. Collect sample in 500mL Polypropylene, Polyethylene Terephthalate (PET), High-Density Polyethylene (HDPE) or 250mL glass amber bottle with a Teflon-lined lid.

Soil samples require 120 ml or 250 ml glass jar with a Teflon-lined lid containing at least 50 g of soil.

Preservation: No preservation

Stability

Holding Time:
Water samples are to be extracted as soon as possible upon receipt and within 14 days of the original sampling date.. The analysis of extracts is to be completed within 10 days of the extraction date.

Soil samples are to be extracted within 14 days of the original sampling date and the

analysis of extracts is to be completed within 10 days of the extraction date.

Storage: Refrigerate in the dark at ≤ 6°C (do not freeze)

Procedure

Borate buffer Preparation

Prepare a solution of 19.108g Na2B4O7·10H2O in 1L of water, and a second solution of 12.404g H3BO3 and 9.925g NaCl in 1L of water. Mix equal volumes of the two solutions and adjust to pH 9.5 using 20% NaOH.

Derivatizing reagent Preparation

Prepare 2.5 mmol FMOC-Cl solution in acetonitrile. Store FMOC-CL solution in a freezer, shelf life is one year.

Liquid Samples

Water samples can be used as received, filter a small portion of the sample into a 10mL test tube using a 3cc syringe with a 0.45um syringe filter. Using a pipette, transfer 0.5mL of the filtered sample into a PTFE filter vial. Pipette from the middle of the via and be sure not to shake the vial or it could disturb particulate matter that has settled to the bottom.

Solids Extraction

Transfer 10g of solids sample into a 40 mL vial. Add 10mL of borate buffer solution. Shake samples for 30 minutes on a wrist action shaker then allow to settle or centrifuge at 1800rpm for 10 minutes. Filter a small portion of the extract into a 10mL test tube using a 3cc syringe with a 0.45um syringe filter. Using a pipette, transfer 0.5mL of the filtered extract into a PTFE filter vial.

Derivatization

Add 25uL of borate buffer for every 0.5mL of sample, add 100uL of FMOC-CL derivatization agent of every 0.5mL of sample. Incubate the vials under low heat for about 30 minutes.

After incubation, remove the filter vial caps and add 10uL of 30% HCl for every 0.5mL of sample, vortex and analyze the samples by HPLC-UV or DAD.

Performance Requirements

Any analytical method options selected for this analysis must meet or exceed the performance requirements specified below.

Accuracy and Precision requirements are distinct from daily QC requirements, and apply to measures of long term method performance (averages and standard deviations). Achievement of these requirements is to be demonstrated during initial and ongoing method re-validation studies. For Initial Validations, averages of at least 8 Lab Control Samples or RMs must be assessed. Ongoing Re-validations (performance reviews) should assess QC data encompassing longer timeframes (e.g. 6 months to 1 year). A minimum frequency of 2 years is recommended for Ongoing Re-validations.

Accuracy Requirement: Laboratories must demonstrate method accuracy (measured as average recovery) of 80-120% or better for Lab Control Samples or Certified Reference Materials at concentrations above ten times the MDL.

Precision Requirement: Laboratories must demonstrate method precision equal to or better than 20% relative standard deviation for clean matrix spikes at concentrations above ten times the MDL.

Sensitivity Requirement: Where possible, the method should support Reporting Limits (and MDLs) that are less than 1/5 of applicable numerical standards. The method is not fit-for-purpose if an MDL exceeds a guideline, standard, or regulatory criteria against which it will be used for evaluation of compliance.

Quality Control

Summary of QC Requirements		
QC Component	**Minimum Frequency**	**Minimum Data Quality Objectives**

Internal Standard Area Checks	All samples and QC	Within 50% of initial calibration or last CCV
Surrogate Compounds	Optional	80 - 120% recovery
Calibration Verification Standard (CVS)	1 per initial calibration	80 - 120% recovery
Method Blank (MB)	One per batch (max 20 samples)	Less than reported DL
Lab Control Sample (LCS) or Reference Material (RM)	One per batch (max 20 samples)	70 – 130%
Lab Duplicates (DUP)	One per batch (max 20 samples)	20% RPD [or within 2x reported DL for low level results]
Matrix Spike (MS)	One per batch (max 20 samples)	60 – 140%
Continuing Calibration Verification (CCV)	At least every 12 hours (max 20 samples), and at end of each batch.	80 - 120% recovery for mid-level standards
If DQOs are not met, repeat testing or report qualified test results. DQOs do not apply to MS results where sample background exceeds spike amount.		

Prescribed Elements

The following components of this method are mandatory:

1. Sample holding times must be adhered to. Samples analyzed beyond the stated holding time must be qualified.
2. Samples that exceed the calibration range must be diluted and re-analyzed, or reported as estimated or minimum values.
3. All stated Performance Requirements and Quality Control requirements must be met.

Apart from these limitations, and provided performance requirements are met, laboratories may introduce modifications to this method in order to improve quality or efficiency. Laboratories must disclose to their clients where modified or alternative methods are employed.

Revision History

Mar 17, 2017 First version of method.