ISO/FDIS 17735ISO 17735:2019 (E)

Workplace atmospheres — Determination of total isocyanate groups in air using 1-(9-anthracenylmethyl)piperazine (MAP) reagent and liquid chromatographyWorkplace atmospheres — Determination of total isocyanate groups in air using 1-(9-anthracenylmethyl)piperazine (MAP) reagent and liquid chromatography

Contents

Foreword

Introduction

Foreword

Introduction

1 Scope

2 Normative references

3 Terms and definitions

4 Principle

5 Reagents and materials

5.1 General
5.2 MAP reagent
5.3 Reagent solutions
5.3.1 Impinger solution
5.3.2 Solution for filter impregnation
5.3.3 Filter extraction solution
5.3.4 Stability of reagent solutions
5.4 Standard matching solutions
5.4.1 General
5.4.2 Preparation of monomer derivatives
5.4.3 Preparation of standard solutions of monomer derivatives for HPLC analysis
5.4.4 Preparation of monomer derivatives for solid-phase extraction (SPE)
5.4.5 Preparation of derivative solutions of bulk isocyanate products
5.5 HPLC mobile phase
5.5.1 General
5.5.2 Mobile phase buffer solutions
5.5.3 Primary mobile phases
5.5.4 Post-column acid mobile phase

6 Apparatus

6.1 General
6.2 Sampler
6.2.1 General
6.2.2 Filters
6.2.3 Midget impingers
6.3 Sampling pump
6.4 Tubing
6.5 Flowmeter
6.6 Filtration and solid-phase extraction equipment
6.7 Liquid chromatographic system
6.7.1 Autosampler
6.7.2 Pumping system
6.7.3 Analytical column
6.7.4 Column oven

6.7.5 Post-column acid delivery pump
6.7.6 Detectors

7 Air sampling

7.1 Pre-sampling laboratory preparation
7.1.1 Cleaning of sampling equipment
7.1.2 Preparation of MAP-coated filter samplers
7.1.3 Preparation of extraction solution jars
7.2 Pre-sampling field preparation
7.2.1 Calibration of pump
7.2.2 Preparation of samplers
7.3 Collection of air samples
7.3.1 Filter sampling
7.3.2 Impinger sampling
7.3.3 Sampling with an impinger followed by a filter
7.4 Blanks and negative controls
7.5 Bulk products
7.6 Shipment of samples
7.7 Filter test samples
7.8 Impinger test samples

8 HPLC analysis

8.1 Instrumental settings
8.2 HPLC programme

9 Data handling

9.1 Monomer measurement
9.2 Oligomer measurement (total detectable isocyanate)

10 Calibration and quality control

10.1 Standard matching solutions
10.2 Calibration curves
10.3 Blank tests
10.4 Bulk products
10.5 Quality control spikes

11 Calculations

11.1 Monomer
11.2 Oligomers (total detectable isocyanate)

12 Interferences

13 Determination of performance characteristics

13.1 General
13.2 Assessment of performance characteristics
13.2.1 Collection efficiency relative to particle size distribution
13.2.2 Air sampling
13.2.2.1 Sampling volume
13.2.2.2 Sampling time
13.2.2.3 Variations in flow rate during sampling
13.2.2.4 Conversion of sample volume to STP
13.2.2.5 Combined uncertainty of sample volume
13.2.3 Analysis
13.2.3.1 Sampled mass
13.2.3.2 Analyte stability
13.2.3.3 Reaction/extraction efficiency
13.2.3.4 Response factor
13.2.3.5 Uncorrected analytical mass of compound
13.2.3.6 Standard matching solutions
13.2.3.7 Lack-of-fit of calibration function
13.2.3.8 Drift in detector response
13.2.3.9 Precision of the analysis
13.2.3.10 Analytical selectivity
13.2.3.11 Combined uncertainty in the analytical mass of isocyanate
13.2.3.12 Combined uncertainty in the sampled mass of isocyanate

13.2.4 Mass of compound in sample blank
13.2.5 Between-laboratory uncertainty contributions
13.2.6 Combined uncertainty
13.2.7 Expanded uncertainty
13.2.8 Uncertainty from performance criteria

1 Scope

2 Normative references

3 Terms and definitions

4 Principle

5 Reagents and materials

5.1 General
5.2 MAP reagent
5.3 Reagent solutions
5.3.1 Impinger solution
5.3.2 Solution for filter impregnation
5.3.3 Filter extraction solution
5.3.4 Stability of reagent solutions
5.4 Standard matching solutions
5.4.1 General
5.4.2 Preparation of monomer derivatives
5.4.3 Preparation of standard solutions of monomer derivatives for HPLC analysis
5.4.4 Preparation of monomer derivatives for solid-phase extraction (SPE)
5.4.5 Preparation of derivative solutions of bulk isocyanate products
5.5 HPLC mobile phase
5.5.1 General
5.5.2 Mobile phase buffer solutions
5.5.3 Primary mobile phases
5.5.4 Post-column acid mobile phase

6 Apparatus

6.1 General
6.2 Sampler
6.2.1 General
6.2.2 Filters
6.2.3 Midget impingers
6.3 Sampling pump
6.4 Tubing
6.5 Flowmeter
6.6 Filtration and solid-phase extraction equipment
6.7 Liquid chromatographic system
6.7.1 Autosampler
6.7.2 Pumping system
6.7.3 Analytical column
6.7.4 Column oven
6.7.5 Post-column acid delivery pump
6.7.6 Detectors

7 Air sampling

7.1 Pre-sampling laboratory preparation
7.1.1 Cleaning of sampling equipment
7.1.2 Preparation of MAP-coated filter samplers
7.1.3 Preparation of extraction solution jars
7.2 Pre-sampling field preparation
7.2.1 Calibration of pump
7.2.2 Preparation of samplers
7.3 Collection of air samples
7.3.1 Filter sampling
7.3.2 Impinger sampling
7.3.3 Sampling with an impinger followed by a filter

7.4 Blanks and negative controls
7.5 Bulk products
7.6 Shipment of samples
7.7 Filter test samples
7.8 Impinger test samples

HPLC analysis

8.1 Instrumental settings
8 8.2 HPLC programme

Data handling

9.1 Monomer measurement
9 9.2 Oligomer measurement (total detectable isocyanate)

10 Calibration and quality control

10.1 Standard matching solutions
10.2 Calibration curves
10.3 Blank tests
10.4 Bulk products
10.5 Quality control spikes

Calculations

1 11.1 Monomer
1 11.2 Oligomers (total detectable isocyanate)

12 Interferences

13 Determination of performance characteristics

13.1 General
13.2 Assessment of performance characteristics
13.2.1 Collection efficiency relative to particle size distribution
13.2.2 Air sampling
13.2.2.1 Sampling volume
13.2.2.2 Sampling time
13.2.2.3 Variations in flow rate during sampling
13.2.2.4 Conversion of sample volume to STP
13.2.2.5 Combined uncertainty of sample volume
13.2.3 Analysis
13.2.3.1 Sampled mass
13.2.3.2 Analyte stability
13.2.3.3 Reaction/extraction efficiency
13.2.3.4 Response factor
13.2.3.5 Uncorrected analytical mass of compound
13.2.3.6 Standard matching solutions
13.2.3.7 Lack-of-fit of calibration function
13.2.3.8 Drift in detector response
13.2.3.9 Precision of the analysis
13.2.3.10 Analytical selectivity
13.2.3.11 Combined uncertainty in the analytical mass of isocyanate
13.2.3.12 Combined uncertainty in the sampled mass of isocyanate
13.2.4 Mass of compound in sample blank
13.2.5 Between-laboratory uncertainty contributions
13.2.6 Combined uncertainty
13.2.7 Expanded uncertainty
13.2.8 Uncertainty from performance criteria

Annex A (informative) Performance characteristics

A.1 Uncertainty estimates
A.2 Combined uncertainty
A.3 Expanded uncertainty
Annex A (informative) Performance characteristics

A.1 Uncertainty estimates
A.2 Combined uncertainty
A.3 Expanded uncertainty