

# ISO/FDIS 17735 ISO 17735:2019 (E)

## Workplace 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	8.1	Instrumental settings
	8.2	HPLC programme
		Data handling
9	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
		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