ISO 14966:2019-12 (E)

Ambient air - Determination of numerical concentration of inorganic fibrous pa rticles - Scanning electron microscopy method

Contents

Page

Foreword v

Introduction vi

1 Scope 1

2 Normative references 1

3 Terms and definitions 1

4 Abbreviated terms 4

5 Principle 4

6 Apparatus and materials 4
6.1 Air sampling 4
6.1.1 Sampling head 4
6.1.2 Sampling train 5
6.1.3 Sampling pump 5
6.1.4 Needle valve 6
6.1.5 Volumetric flowmeter (rotameter) 6
6.1.6 Timer 6
6.1.7 Dry type gas meter (optional) 6
6.1.8 Meteorological instruments (optional) 6
6.1.9 Instruments for unattended sampling (optional) 7
6.2 Preparation of filters 7
6.2.1 Vacuum evaporator 7
6.2.2 Plasma asher 8
6.3 Sample analysis 8
6.3.1 Scanning electron microscope (SEM) 8
6.3.2 Energy-dispersive X-ray system 8
6.3.3 Stereo-microscope 9
6.3.4 Gold-coated capillary-pore polycarbonate filters 9
6.3.5 Backing filters 9
6.3.6 Disposable plastic field monitors (optional) 9
6.3.7 Technically pure oxygen 9
6.3.8 Rubber connecting hoses 9
6.3.9 Filter containers 9
6.3.10 Routine electron microscopy tools and supplies 9
6.3.11 Sample for resolution adjustment 9
6.3.12 Sample for magnification calibration 10

7 Air sample collection and analysis 10
7.1 Measurement planning 10
7.2 Collection of air samples 10
7.3 SEM specimen preparation 13
7.4 Analysis in the scanning electron microscope 13
7.4.1 General instructions 13
7.4.2 Fibre-counting criteria 14
7.4.3 Fibre classification 19
7.4.4 Analysis using reference spectra and peak height ratios 26

7.4.5 Measurement of fibre dimensions 28
7.4.6 Recording of data on the fibre counting form 28

8 Calculation of results 28
8.1 Calculation of the mean fibre concentration 28
8.2 Calculation of the 95 % confidence interval 30

9 Performance characteristics 30
9.1 General 30
9.2 Measurement uncertainty 30

9.2.1 Systematic errors 30
9.2.2 Random errors 30
9.2.3 Errors due to sampling 31
9.2.4 Errors associated with the SEM examination 31
9.2.5 Total error of the measurement 31
9.2.6 Random errors due to fibre counting 32
9.3 Limit of detection 34

10 Test report 35

Annex A (normative) Preparation of filters for air sampling 37

Annex B (normative) Procedures for calibration and adjustment of the SEM 38

Annex C (informative) Characteristics and chemical composition of inorganic fibres 40

Annex D (informative) Poisson variability as a function of fibre density on sampling filter and area of filter analysed 45

Annex E (informative) Combination of the results from multiple samples 47

Bibliography 48