

# ISO 14966:2019-12 (E)

## Ambient air - Determination of numerical concentration of inorganic fibrous particles - Scanning electron microscopy method

---

<b>Contents</b>		<b>Page</b>
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