

ISO 13794:2019-10 (E)

Ambient air - Determination of asbestos fibres - Indirect-transfer transmission electron microscopy method

| Contents | Page |
|---|------|
| Foreword | vi |
| Introduction | vii |
| 1 Scope | 1 |
| 2 Normative references | 1 |
| 3 Terms and definitions | 1 |
| 4 Symbols and abbreviated terms | 7 |
| 5 Type of sample | 8 |
| 6 Range | 8 |
| 7 Limit of detection | 8 |
| 8 Principle | 8 |
| 9 Reagents | 9 |
| 10 Apparatus | 10 |
| 10.1 Air sampling | 10 |
| 10.1.1 Filter cassette | 10 |
| 10.1.2 Sampling pump | 10 |
| 10.1.3 Stand | 10 |
| 10.1.4 Personal sampling | 10 |
| 10.1.5 Flowmeter | 10 |
| 10.2 Specimen preparation laboratory | 11 |
| 10.3 Equipment for analysis | 11 |
| 10.3.1 Transmission electron microscope | 11 |
| 10.3.2 Energy dispersive X-ray analyser | 13 |
| 10.3.3 Plasma ashing | 13 |
| 10.3.4 Vacuum coating unit | 13 |
| 10.3.5 Sputter coater | 13 |
| 10.3.6 Beakers | 13 |
| 10.3.7 Vacuum source | 13 |
| 10.3.8 Glass filtration apparatus | 14 |
| 10.3.9 Solvent washer (Jaffe washer) | 14 |
| 10.3.10 Condensation washer | 15 |
| 10.3.11 Slide warmer or oven | 16 |
| 10.3.12 Ultrasonic bath | 16 |
| 10.3.13 Carbon grating replica | 16 |
| 10.3.14 Calibration specimen grids for EDXA | 16 |
| 10.3.15 Carbon rod sharpener | 17 |
| 10.3.16 Disposable tip micropipettes | 17 |
| 10.3.17 Thermometer | 17 |
| 10.3.18 Stopwatch | 17 |
| 10.4 Consumable supplies | 17 |
| 10.4.1 Copper or nickel electron microscope grids | 17 |

| | | |
|----------------------------|--|-----------|
| 10.4.2 | Gold or nickel electron microscope grids | 17 |
| 10.4.3 | Aluminium foil | 17 |
| 10.4.4 | Carbon rod electrodes | 17 |
| 10.4.5 | Routine electron microscopy tools and supplies | 18 |
| 10.4.6 | Reference asbestos samples | 18 |
| 10.4.7 | Reference samples of mineral fibres other than asbestos | 18 |
| 11 | Air sample collection | 18 |
| 11.1 | Calculation of analytical sensitivity | 18 |
| 11.2 | Sample collection procedure | 19 |
| 12 | Procedure for analysis | 20 |
| 12.1 | General | 20 |
| 12.2 | Cleaning of sample cassettes | 20 |
| 12.3 | Preparation of analytical filters | 20 |
| 12.3.1 | Selection of filter area for ashing | 20 |
| 12.3.2 | Ashing of sample collection filters | 20 |
| 12.3.3 | Aqueous dispersal of residual ash from sample collection filters | 21 |
| 12.3.4 | Assembly of system for filtration of aqueous dispersions | 21 |
| 12.3.5 | Filtration of aqueous dispersions | 21 |
| 12.4 | Preparation of TEM specimens from PC analytical filters | 22 |
| 12.4.1 | Selection of filter area for carbon-coating | 22 |
| 12.4.2 | Carbon-coating of filter portions | 22 |
| 12.4.3 | Preparation of the Jaffe washer | 23 |
| 12.4.4 | Placing of specimens into the Jaffe washer | 23 |
| 12.5 | Preparation of TEM specimens from cellulose ester analytical filters | 23 |
| 12.5.1 | Selection of area of filter for preparation | 23 |
| 12.5.2 | Preparation of solution for collapsing cellulose ester filters | 23 |
| 12.5.3 | Filter-collapsing procedure | 23 |
| 12.5.4 | Plasma etching of the filter surface | 23 |
| 12.5.5 | Carbon-coating | 24 |
| 12.5.6 | Preparation of the Jaffe washer | 24 |
| 12.5.7 | Placing of specimens in the Jaffe washer | 24 |
| 12.6 | Criteria for acceptable TEM specimen grids | 24 |
| 12.7 | Procedure for structure counting by TEM | 25 |
| 12.7.1 | General | 25 |
| 12.7.2 | Measurement of mean grid opening area | 25 |
| 12.7.3 | TEM alignment and calibration procedures | 26 |
| 12.7.4 | Determination of criterion for termination of TEM examination | 26 |
| 12.7.5 | General procedure for structure counting and size analysis | 26 |
| 12.7.6 | Estimation of mass concentration of asbestos fibres and bundles | 27 |
| 12.7.7 | Magnification requirements | 27 |
| 12.8 | Blank and quality control determinations | 29 |
| 12.9 | Calculation of results | 30 |
| 13 | Performance characteristics | 30 |
| 13.1 | General | 30 |
| 13.2 | Interferences and limitations of fibre identification | 30 |
| 13.3 | Precision and accuracy | 31 |
| 13.3.1 | Precision | 31 |
| 13.3.2 | Accuracy | 31 |
| 13.3.3 | Inter- and intra-laboratory analyses | 31 |
| 13.4 | Limit of detection | 32 |
| 14 | Test report | 32 |
| Annex A (normative) | Determination of operating conditions for plasma ashing | 36 |
| Annex B (normative) | Determination and standardization of operating conditions for ultrasonic bath | 37 |

| | |
|--|-----------|
| Annex C (normative) Calibration procedures | 39 |
| Annex D (normative) Structure counting criteria | 42 |
| Annex E (normative) Fibre identification procedure | 52 |
| Annex F (normative) Determination of the concentration of asbestos fibres and bundles longer than 5 µm and PCM equivalent asbestos fibres | 68 |
| Annex G (normative) Calculation of results | 69 |
| Annex H (normative) Test procedure to determine suitability of cellulose ester sample collection filters | 76 |
| Annex I (informative) Strategies for collection of air samples | 77 |
| Bibliography | 78 |