

# ISO 13163:2021 (E)

## Water quality — Lead-210 — Test method using liquid scintillation counting

---

### Contents

|         |  |
|---------|--|
|         | Foreword   |
|         | Introduction   |
| 1       | Scope  |
| 2       | Normative references   |
| 3       | Terms, definitions and symbols   |
| 3.1     | Terms and definitions  |
| 3.2     | Symbols and abbreviated terms  |
| 4       | Principle  |
| 5       | Sampling and storage   |
| 6       | Procedure  |
| 6.1     | Sample preparation for measurement   |
| 6.2     | Sample measurement   |
| 7       | Quality assurance and quality control program  |
| 7.1     | General  |
| 7.2     | Variables that could influence the measurement   |
| 7.3     | Instrument quality control   |
| 7.4     | Reagent interferences  |
| 7.5     | Interference control   |
| 7.6     | Method verification  |
| 7.7     | Demonstration of analyst capability  |
| 7.8     | Calibration  |
| 8       | Expression of results  |
| 8.1     | General  |
| 8.2     | Sample recovery, activity and uncertainties  |
| 8.3     | Decision threshold   |
| 8.4     | Detection limit  |
| 8.5     | Limits of the coverage intervals   |
| 8.5.1   | Limits of the probabilistically symmetric coverage interval  |
| 8.5.2   | The shortest coverage interval   |
| 9       | Test report  |
| Annex A | (informative) Separation and purification of <sup>210</sup> Pb   |
| A.1     | Principle  |
| A.2     | Reagents and apparatus   |
| A.2.1   | Reagents   |
| A.2.1.1 | Lead-210 standard solutions  |
| A.2.1.2 | Standard solution of Pb(II)  |
| A.2.1.3 | Quenching agent  |
| A.2.1.4 | Laboratory water, distilled or deionized, complying with grade 3 of ISO 3696   |
| A.2.1.5 | Solution of stable iron carrier, approximately 1 g·l <sup>-1</sup> (for example in 0,5 mol·l <sup>-1</sup> HNO <sub>3</sub> ). |
| A.2.1.6 | Cationic exchange resin, e.g. sulfonic type 8 % cross-linking.   |
| A.2.1.7 | Hydrochloric acid solution, c(HCl) = 2 mol·l <sup>-1</sup> .   |
| A.2.1.8 | Nitric acid solution, c(HNO <sub>3</sub> ) = 1 mol·l <sup>-1</sup> .   |
| A.2.1.9 | Nitric acid solution, c(HNO <sub>3</sub> ) = 0,1 mol·l <sup>-1</sup> .   |

- A.2.1.10 ammonium hydroxide solution,  $c(\text{NH}_4\text{OH})$  concentrated = 280 g·l<sup>-1</sup>.
- A.2.1.11 Ammonium citrate or citric acid solution,  $c(\text{C}_6\text{H}_{11}\text{NO}_7)$  or  $c(\text{C}_6\text{H}_8\text{O}_7) = 0,01 \text{ mol}\cdot\text{l}^{-1}$  to 0,1 mol·l<sup>-1</sup>
- A.2.1.12 EDTA solution,  $c(\text{C}_{10}\text{H}_{16}\text{N}_2\text{O}_8) = 0,01 \text{ mol}\cdot\text{l}^{-1}$ .
- A.2.1.13 Chromatographic extraction resin, 18C6 Crown ether-type resins.
- A.2.1.14 Liquid scintillation cocktail
- A.2.2 Apparatus
  - A.2.2.1 Centrifuge or vacuum filtration system.
  - A.2.2.2 Membrane filter, of pore size 0,45 µm.
  - A.2.2.3 Analytical balance, accuracy 0,1 mg.
  - A.2.2.4 Equipment for the measurement of stable lead.
  - A.2.2.5 Measurement equipment: Liquid scintillation counter
  - A.2.2.6 Scintillation vials
- A.3 Procedure with 2 mol·l<sup>-1</sup> HCl loading medium
  - A.3.1 Preconcentration
    - A.3.1.1 Preparation
    - A.3.1.2 Preconcentration by iron (III) hydroxide co-precipitation
    - A.3.1.3 Preconcentration with cation exchange resin [10]
  - A.3.2 Separation of <sup>210</sup>Pb - Method with 2 mol·l<sup>-1</sup> HCl loading medium (see Figure A.1)
    - A.3.2.1 General
    - A.3.2.2 Preparation for the counting and the determination of the chemical recovery
    - A.3.2.3 Measurement
- A.4 Procedure with 1 mol·l<sup>-1</sup> HNO<sub>3</sub> loading medium
  - A.4.1 Preparation
  - A.4.2 Preconcentration
  - A.4.3 Separation of <sup>210</sup>Pb - Method with 1mol·l<sup>-1</sup> HNO<sub>3</sub> loading medium (see Figure A.2)

**Annex B (informative) Spectra examples**