

Selenium

M363

0.05 - 1.6 mg/L Se

3,3'-Diaminobenzidine in Toluene

Instrument specific information

The test can be performed on the following devices. In addition, the required cuvette and the absorption range of the photometer are indicated.

Instrument Type	Cuvette	λ	Measuring Range
SpectroDirect	□ 50 mm	445 nm	0.05 - 1.6 mg/L Se
XD 7000, XD 7500	□ 50 mm	445 nm	0.05 - 2 mg/L Se

Sampling

- Turbid samples must be filtered through a 0.45 μm pore size membrane filter.

Preparation

The following reagents need to be purchased:

1. Formic acid 98-100% for analysis (CAS-No.: 64-18-6)
2. 3,3'-Diaminobenzidine tetrahydrochloride-hydrate (CAS-No.: 868272-85-9)
3. Ammonia water 25% for analysis (CAS-No.: 1336-21-6)
4. EDTA disodium salt solution 0.1 mol/l (CAS-No.: 139-33-3)
5. Toluene for gaschromatography (CAS-No.: 108-33-3)
6. pH-indicator strips, pH 2.0 - 9.0
7. Sodium sulfate anhydrous for analysis (CAS-No.: 7757-82-6)
8. Water for analysis

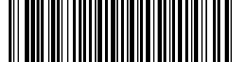
Other materials:

1. membrane filter (pore size: 0.45 μm)
- The pH-value of the sample should be almost neutral before the analysis.

Notes

- The result is given in mg/L Se⁴⁺





Determination of Selenium

Select the method on the device.

Reagent 1

- Bring 9.4 mL formic acid p.a. into a 100-ml-volumetric flask
- Fill with water p.a. up to the mark.

Reagent 2

- Solve 0.5 g 3,3'-diaminobenzidine tetrahydrochloride-hydrate in 100 mL cooled water p.a.
- This reagent needs to be freshly prepared per working day and stored in an amber bottle.

Reagent 3

- Bring 48 mL ammonia water 25% p.a. into a 100-ml-volumetric flask.
- Fill with water p.a. up to the mark.

1. Fill 50 mm cell with toluene.
2. Place cell in sample chamber, making sure the positioning is correct.
3. Press **Zero** key.
4. Remove the cell from the sample chamber. Empty the cell and dry completely.
5. Add **60 mL** of the **sample** into a beaker.
6. Add **4 mL Reagent 1**.
7. Add **4 mL EDTA solution**.
8. Add **4 mL Reagent 2**.
9. Mix reagents using a stirring rod.
10. Set the pH-value to **2.5 using Reagent 3**.
11. Store beaker at a dark place for **45 minutes**.
12. Set the pH-value to **7.0 using Reagent 3**.
13. Transfer the sample into a 250-ml-separatory funnel.
14. Add **30ml water for analysis**.
15. Add **14 mL toluene**.
16. Shake for **1 minute**.
17. Discard the lower aqueous phase.
18. Transfer the toluene phase into a small (25-50 mL) Erlenmeyer flask.
19. Add one spade point tip of **sodium sulfate anhydrous**.
20. Mix reagent by shaking the beaker gently.
21. Decant the toluene extract into a 50 mm cell.
22. Place cell in sample chamber, making sure the positioning is correct.
23. Press **Test** key.

The result in mg/L Selenium appears on the display.



Chemical Method

3,3'-Diaminobenzidine in Toluene