

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	$\lambda$	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  $\mu\text{m}$  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

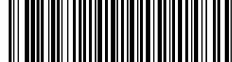
Other materials:

1. membrane filter (pore size: 0.45  $\mu\text{m}$ )
- The pH-value of the sample should be almost neutral before the analysis.

## Notes

- The result is given in mg/L Se<sup>4+</sup>





## 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