# 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 $Se^{4+}$

## 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 EDTAsolution.**
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