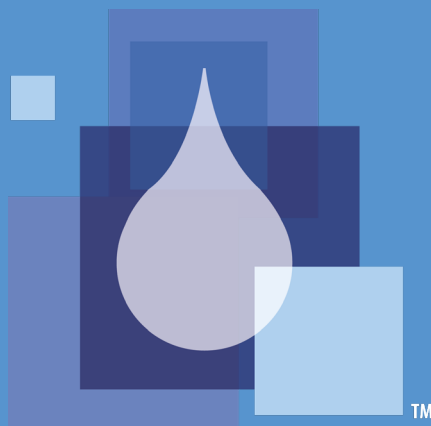


Selenium Quantification and Speciation

OV-AN-0015



OndaVia

Purpose

This OndaVia test provides rapid concentration and speciation information of selenium in mine water samples with concentrations within the 5- to 100-ppb range. By using this kit and the OndaVia Analysis System a technician can obtain selenate concentrations in 30 minutes and total selenium concentrations in 45 minutes with a $\pm 10\%$ accuracy above 10 ppb and ± 2 ppb below 10 ppb.

Materials and Supplies

- OndaVia software with selenium analyte file
- Column vacuum (OV-PP-A003)
- Selenium Analysis Kit:
- OndaVia Cartridge
 - Reagent 1
 - Reagent 2
 - Nanoparticles
- Solid Phase Extraction (SPE) columns:
 - BaAgH column
- Luer-lock syringe *-not supplied*
- Deionized water *-not supplied*
- 1M Magnesium chloride solution (MgCl_2) *-not supplied*
- 40mM Sodium carbonate solution (Na_2CO_3) *-not supplied*
- Hydrochloric Acid (HCl) and Sodium Hydroxide (NaOH) solutions to adjust sample pH *-not supplied*
- 1% Sodium Hypochlorite solution (NaClO) *-not supplied*
- 5% Hydrogen Peroxide solution (H_2O_2) *-not supplied*
- pH paper or meter *-not supplied*
- Pipettor and tips *-not supplied*
- 1-mL Microcentrifuge Tube *-not supplied*
- 12mm glass analysis vial *-not supplied*
- 50-mL Centrifuge Waste Tube

Important Note

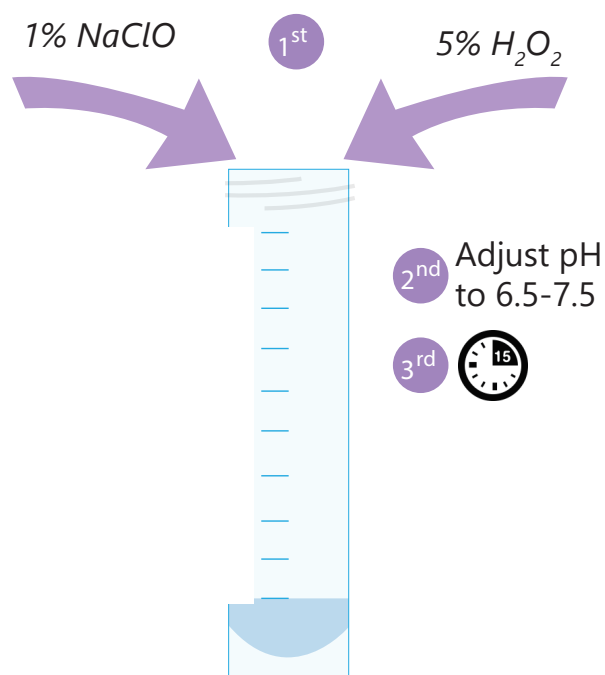
Reagent 2, part of the Selenium Analysis kit, should be stored in the freezer (at approximately -18°C). For optimal results Reagent 2 can only be thawed once. Once thawed, Reagent 2 should be used within a month and stored at 4°C .

Sample Preparation for Total Selenium

- Oxidize the 40-ml sample following the Selenium Oxidation procedure described below:
 - Add 1.2 ml 1% sodium hypochlorite (NaClO) to the sample tube
 - Adjust the pH of the sample solution to between 6.5-7.5 using HCl or NaOH solutions
 - Shake well and let the sample react for 15min

Tip: During the 15-minute wait required for the selenium to oxidize, it is possible to start the DI water conditioning of the Ba column

- Add 1.2 mL of 5% hydrogen peroxide to stop the oxidation reaction



BaAgH Column Preparation

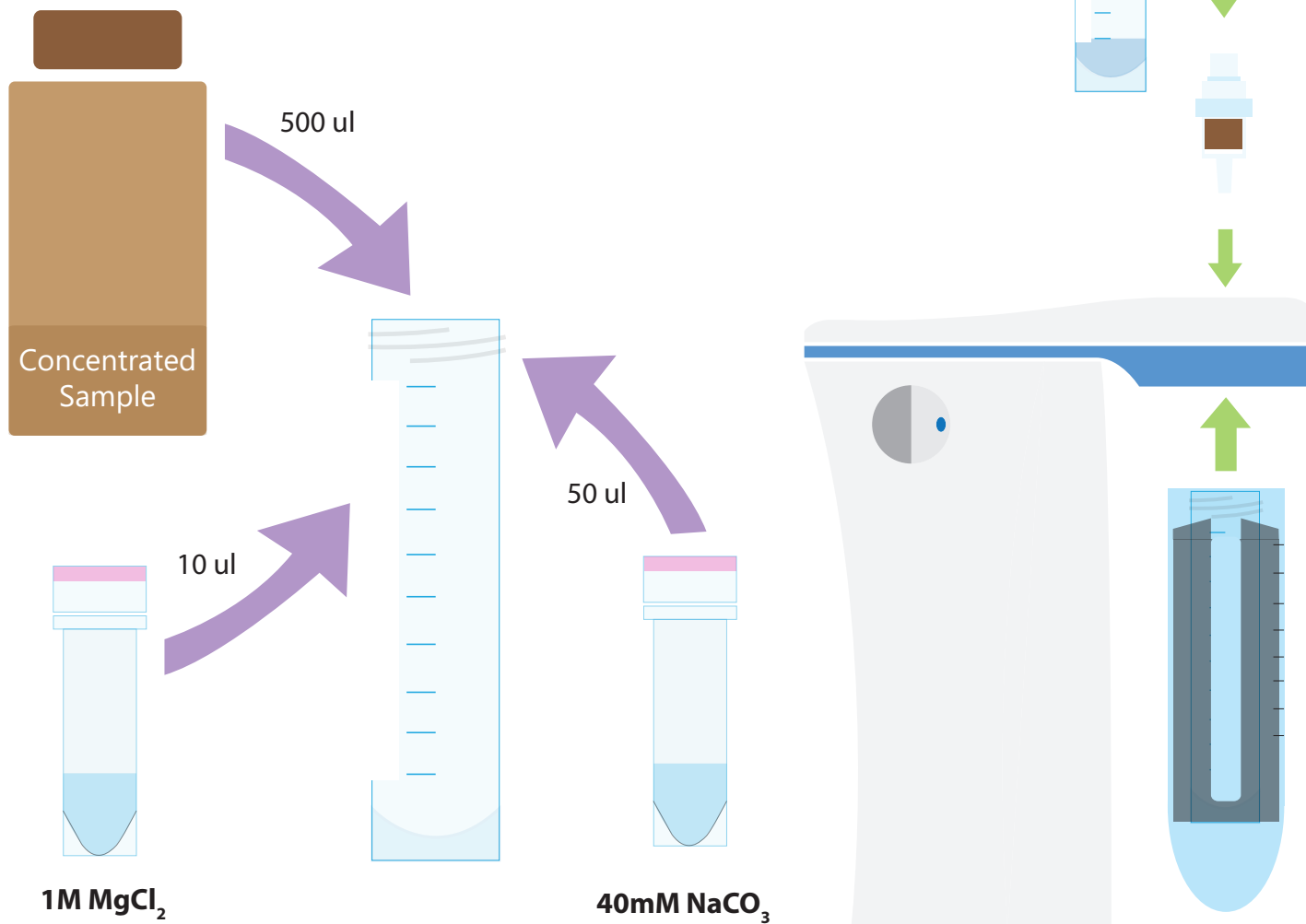
- Remove a column from the clear bag and insert it tip-first through the top opening on the column vacuum
- Obtain a syringe with a capacity of at least 5 mL, pull the plunger out of the syringe and screw the syringe barrel to the top of the column
- Condition the column by passing 20 mL of deionized water (DI):
 - Screw an empty 50-mL centrifuge waste tube into the column vacuum (*this tube will be used to capture the liquid from the pre-treatment cycles*).
 - Fill the syringe with 5 mL of DI water and turn on the vacuum to pull the water through the sorbent bed into the 50-mL waste tube. Add an additional 5 mL of DI water three more times. Allow the fluid to pass through the bed and collect in the waste tube. When the fluid level in the syringe reaches the top of the sorbent bed switch off the vacuum.

Sample Concentration

- Place a 50- or 100-ml glass beaker on an analytical balance and tare the instrument.
- Weight exactly 30.0 mL of sample into the glass beaker.
- Add 300 μ L of Reagent 1 to the beaker
- Heat the sample and beaker on a hot plate until the volume has reduced by at least 10 \times . The higher the reduction factor the stronger the signal from the analyte will be.

Sample Interference Removal

- Transfer 500 μL of sample to a clean vial.
- Add 10 μL of 1M MgCl_2 , followed by 50 μL of 40mM NaCO_3 .
- Prepare the BaAgH column as described above
- Pour the sample mixture into the BaAgH syringe.
- Swap the 50 mL centrifuge waste tube for the tube adapted to fit a 1-mL collection tube. Power on the vacuum to collect the solution in a 1-mL microcentrifuge tube
- Add 10 μL of 1M MgCl and 50 μL of 40mM NaCO_3 to the collected solution and pass it again through the same BaAgH column.
- Collect the final solution in a new 1-ml tube



Final Preparation Steps

- Prepare Reagent 2, if not already hydrated
- Transfer 200µL of sample to a clean, 12mm glass analysis vial. Add 40 µL of 100mM HCl and 40µL of Reagent 2.
- Add 200µL of Nanoparticle solution to the glass vial.
- Proceed with analysis in the OPAL-100 Series instrument.

