

LZC390/LCK390 AOX

Adsorbable organic halogen

DOC312.53.94124

0.05–3.00 mg/L AOX

LCK390

Scope and application: For wastewater and process water.



Test preparation

Test storage

Storage temperature: 2–8 °C (35–46 °F)

pH/Temperature

The pH of the water sample must be between pH 4–9.

The temperature of the water sample and reagents must be between 15–25 °C (59–77 °F).

The pH of strongly buffered samples should be determined separately before the concentration step (i.e. after the addition of solution A (LCK390 A)), and if necessary concentrated nitric acid should be added to adjust the pH to 2.

Before starting

For reliable and quality results, only use accessories from the manufacturer.

Review safety information and expiration date on the package.

Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.

Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

The analysis of adsorbable organic halogens (AOX) is carried out in several steps:

- Concentration (of the AOX compounds)
- Rinsing (removing the inorganic chloride from the water sample)
- Preparing the reaction tubes (for wet-chemical digestion)
- Digestion (wet-chemical digestion of the AOX compounds)
- Measurement (of the digested, originally organically bonded, chloride with the cuvette test).
- Cleaning (the materials used)

Items to collect—Starter-Set (LZC905—only supplied with a first order)

Description
Tweezers
Magnetic stirrer rods

Before starting I—Concentration

Remove the Carbodisk from the blister packs immediately before the measurements are carried out. The Carbodisk must not come into contact with the atmosphere for any length of time. Carbodisk that have been stored in a damaged condition or in accidentally opened blister packs must not be used. There should be no smoking in the workplace!

The Carbodisk should not be allowed to come into contact with the skin! Always use tweezers to remove the Carbodisk from the sealed wrapper and to manipulate it during the analysis procedure.

Items to collect I—Concentration

Description

Aluminium blister packs with Carbodisk

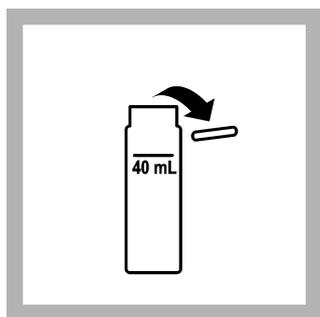
Magnetic stirrer rods

Tweezers

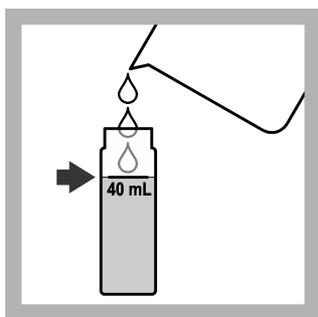
Solution A (LCK390 A)

Reaction tubes

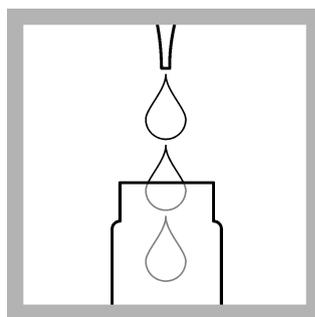
Procedure I—Concentration



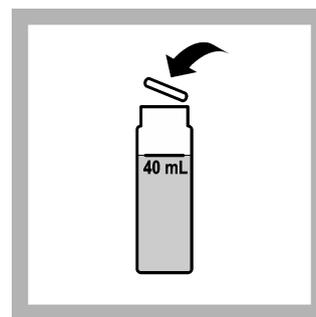
1. Remove the magnetic stirrer rod from the concentrating vessel and place it on a clean paper towel.



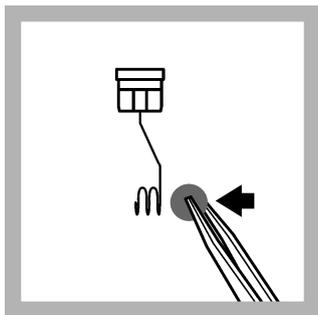
2. Add the sample to the concentrating bottle until the **40 mL mark** is reached. A beaker can be used for this purpose.



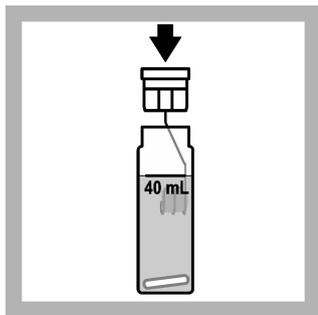
3. Pipet into the sample: **2.0 mL solution A**. The pH of the sample must be between pH 2 and pH 3. If the pH is higher, adjust it by adding concentrated nitric acid drop by drop.



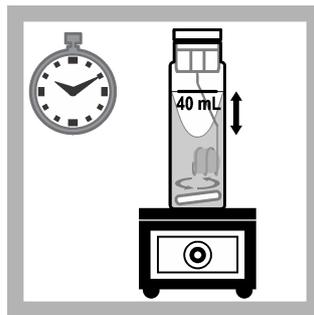
4. Use the tweezers to introduce the magnetic stirrer rod into the concentrating bottle.



5. Open an aluminium blister pack by gripping the aluminium flap at the side and pulling it towards the centre of the pack. Use the tweezers to remove the Carbodisk and push it into the open end of the mat holder.



6. **Immediately** immerse the Carbodisk holder in the sample solution and close the concentrating bottle securely with the rubber stopper.



7. Place the concentrating bottle on a magnetic stirrer. Increase the stirring speed gradually. The optimal stirring speed is reached when the movement of the liquid pulls down the meniscus by about **2 cm**. Stir for **15 minutes**. Then switch off the magnetic stirrer.

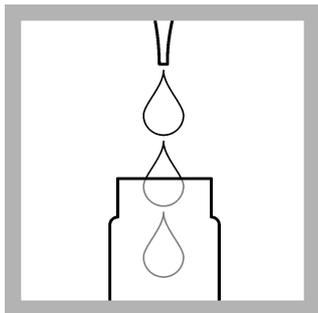
Before starting II—Preparing the reaction tubes

The amount of digestion reagent D (LCK390 D) may not be significantly increased. For this reason, take good care to take only a **level dosing spoon** of the digestion reagent D.

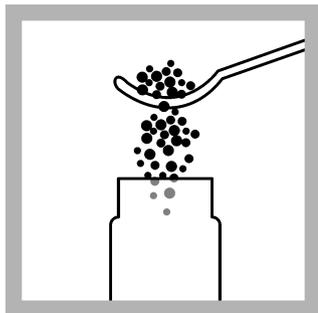
Items to collect II—Preparing the reaction tubes

Description
Digestion solution C (LCK390 C)
Digestion reagent D (LCK390 D)
Dosing spoon
Reaction tubes

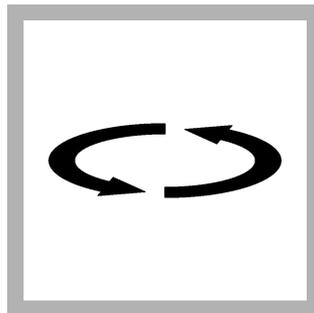
Procedure II—Preparing the reagent tubes



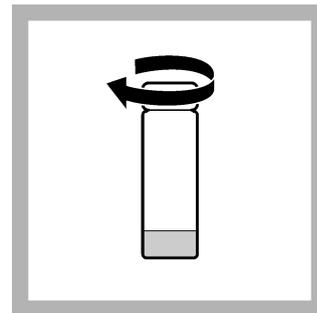
1. Add to a dry reaction tube:
3.6 mL of digestion **solution C**.



2. Add to the same reaction tube:
1 level dosing spoon of digesting **reagent D**.



3. Dissolve the digestion **reagent D** completely in the digestion **solution C**.



4. Close reaction tube and allow to stand.

Before starting III—Rinsing and Digestion

Up to 3 g/L inorganic chloride are removed from the waste water sample during the rinsing stage. If the chloride concentration is higher than this, the sample should be diluted.

The Carbodisk must **not be swept up by the liquid** and must be **completely wetted**, otherwise there is no guarantee of adequate rinsing.

Blockages in the rinse attachment (air bubbles or particles) should be removed by tapping lightly on the side of the bottle.

The total amount of rinsing solution B (LCK390 B) should not be less than 20 mL, otherwise the amount of chloride measured will be too high (= high-bias AOX) if the waste water sample contains a high concentration of chloride. If the exact amount of rinsing solution B is added (4 x 5 mL), **the upper meniscus of the rinsing solution B should be level with the upper edge of the white label bar**.

When rinsing is complete, the rinsed Carbodisk must be **immediately** transferred to the prepared reaction tubes; contact with the atmosphere must be avoided as far as possible.

To remove the Carbodisk, use **one arm of the tweezers** (tip) to push it from the bottom of the bottle onto the side. Remove the **damp** Carbodisk with the tweezers and transfer it to the prepared digestion solution (reaction tube).

Turbidities in the digested sample are eliminated by filtration through a 0.45 µm membrane filter (LCW916). (Screw the membrane filter onto the Luer attachment of the syringe and remove the plunger. Introduce the digested sample into the syringe. Refit the plunger, then depress the plunger to filter the sample into a clean, dry beaker.)

Avoid stirring up the sediment if possible, otherwise allow suspended particles to settle.

Items to collect III—Rinsing and Digestion

Description

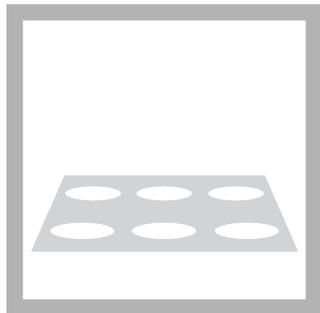
Tweezers

Rinse attachment

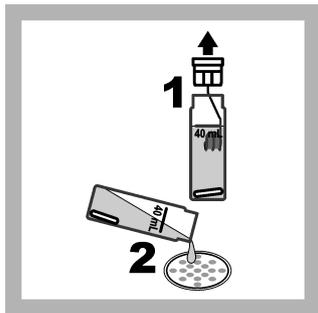
Rinsing solution B (LCK390 B)

Reaction tubes

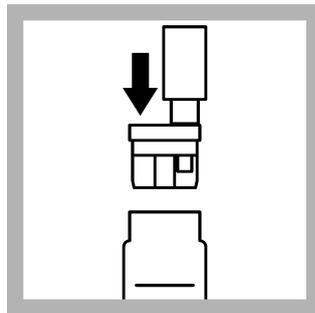
Procedure III—Rinsing and Digestion



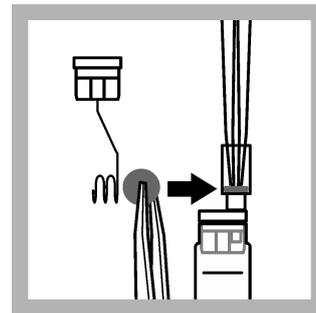
1. Preheat the thermostat to **100 °C (212 °F)**.



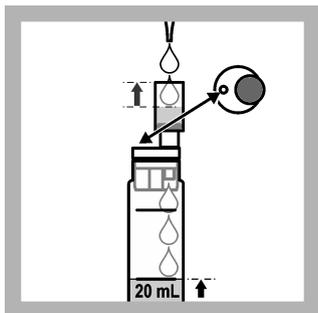
2. Remove the rubber stopper with Carbodisk-holder from the **concentrating bottle** refer to: [Procedure I—Concentration](#) on page 2 and remove the liquid from the bottle down to the drain. The magnetic stirrer rod **must remain** in the bottle.



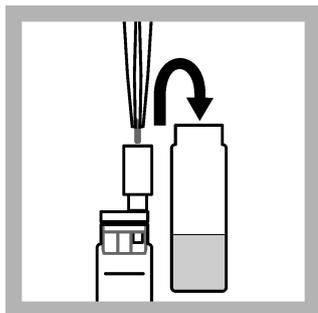
3. Plug the rinse attachment onto the bottle.



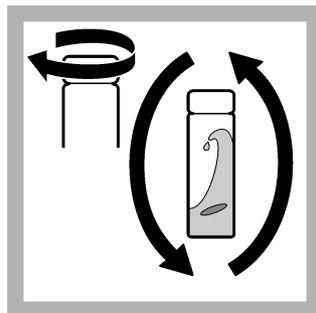
4. Use the tweezers to draw the Carbodisk out of the holder (open side of the spiral) and place it on the bottom of the rinse attachment. Press the Carbodisk down lightly with the tweezers.



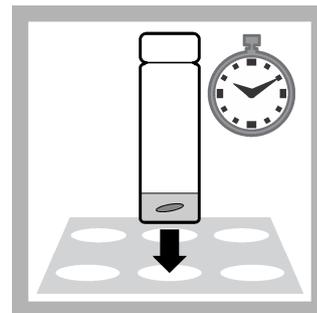
5. To remove the inorganic chloride, use a piston pipette (hold the pipette vertically) to **quickly add 5 mL rinsing solution B four times**, directing the solution onto the centre of the Carbodisk each time.



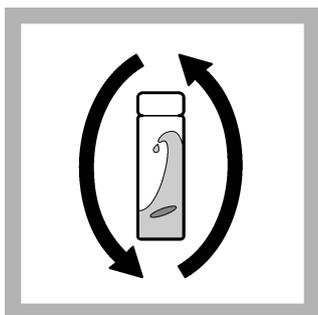
6. To remove the Carbodisk, use one arm of the tweezers (tip) to push it from the bottom of the bottle onto the side. Remove the damp Carbodisk with the tweezers and transfer it to the prepared digestion solution.) refer to: [Procedure II—Preparing the reagent tubes](#) on page 3.



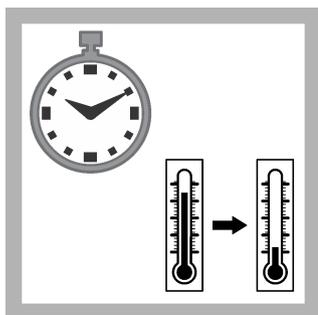
7. Close the reaction tube **immediately** and invert a few times.



8. Heat the reaction tube for **2 hours at 100 °C** in the preheated thermostat.



9. Remove the reaction tube and invert it.



10. Allow the reaction tube to cool to room temperature. The cooling process should take a least **45 minutes**.

Before starting IV—Cuvette test

Attention! Allow disturbed Carbodisk-fibres to settle in the reaction tube. The pipetted solution must not contain any fibres.

Items to collect IV—Cuvette test

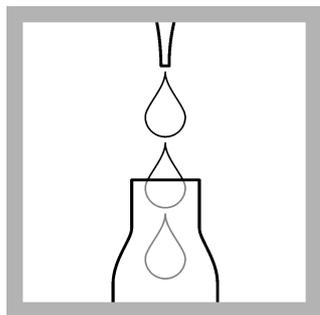
Description

Cuvettes for determining AOX

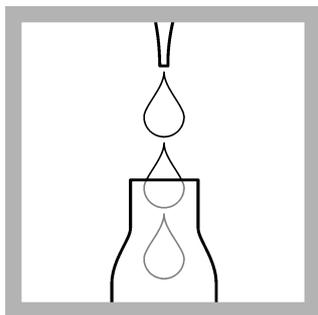
Reagent solution E (LCK390 E)

Zero solution cuvette

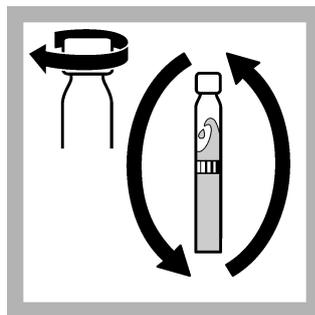
Procedure IV—Cuvette test



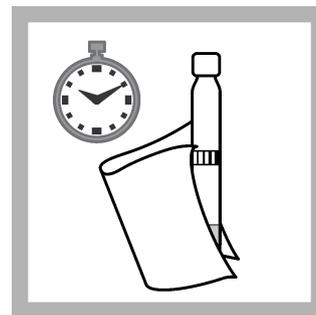
1. Add **0.3 mL** of the reaction **solution E** to the cuvette test LCK390.



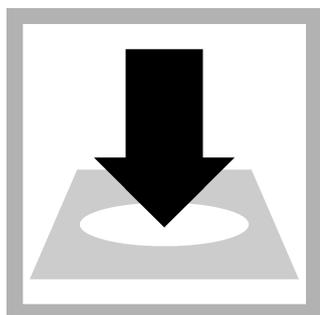
2. Add **1.5 mL** of the **clear supernatant liquid** of the digested sample (reaction tube) to the cuvette refer to: [Procedure III—Rinsing and Digestion](#) on page 4.



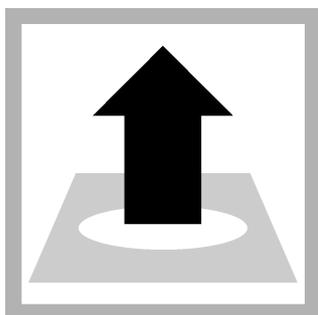
3. Close the cuvette and invert a few times.



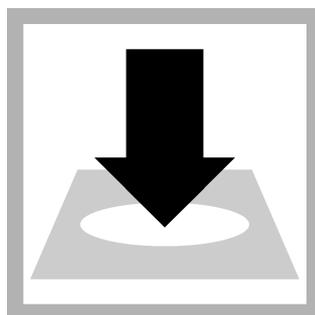
4. After **3 minutes** thoroughly clean the outside of the cuvette. **Do not invert cuvette before carrying out the measurement!**



5. Insert the zero cuvette into the cell holder.
DR1900: Go to LCK/TNTplus methods, Select the test, push **ZERO**.



6. Remove the zero cuvette.



7. Insert the sample cuvette into the cell holder.
DR1900: Push **READ**.

Interferences

Chloride concentrations of < 3 g/L (inorganic chloride in the wastewater sample) do not interfere with the AOX analysis. If the DOC content (Dissolved Organic Carbon) is greater than 100 mg/L, less concentration can be expected. In general turbidities and intrinsic colors in the digested sample interfere with the photometric measurement and cause high-bias results. Turbidities are therefore eliminated with the 0.45 μm membrane filter (refer to: [Procedure III—Rinsing and Digestion](#) on page 4), and yellow colorations are eliminated by diluting the initial sample and repeating the concentration step.

The measurement results must be subjected to plausibility checks (dilute and/or spike the water sample).

Summary of method

The analysis of adsorbable organic halogens (AOX) is carried out in several steps:

1. Concentration of the AOX compounds by adsorption on an active carbon mat (Carbodisk) with the help of a magnetic stirrer.
2. Rinsing out (removing) the inorganic chloride.
3. Digesting the adsorbed AOX compounds on the Carbodisk in a thermostat.
4. Photometric measurement of the AOX.

Cleaning the materials used

Concentrating bottle, magnetic stirrer rod, Carbodisk-holder

Rinse the concentrating bottle, the magnetic stirrer rod and the Carbodisk-holder **manually** (not in a dishwasher) with **distilled** water.

When the magnetic stirrer rod is dry (allow to dry, or use a paper towel), return it to the concentrating bottle and close the bottle with the stopper of the Carbodisk-holder.

Any attached Carbodisk-fibres should be removed from the stopper of the Carbodisk-holder with a paper towel as necessary.

Rinse attachment

The rinse attachment should be rinsed thoroughly with distilled water after use. When it is dry (allow to dry, or use a lint-free paper towel), the rinse attachment should be kept in the AOX test pack. Remove any Carbodisk-fibres with a paper towel.

Tweezers

Rinse with distilled water after use and dry as above. Keep in the AOX-test pack. Any fibres should be removed with a clean paper towel.

Reaction tube

After the digestion the reaction tube and its lid should be **carefully rinsed** with distilled water and **dried**. After drying, fit the lid to the tube and store it in the AOX package. The reaction tubes should not be used more than **12 times**.



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