

AOX

Principle

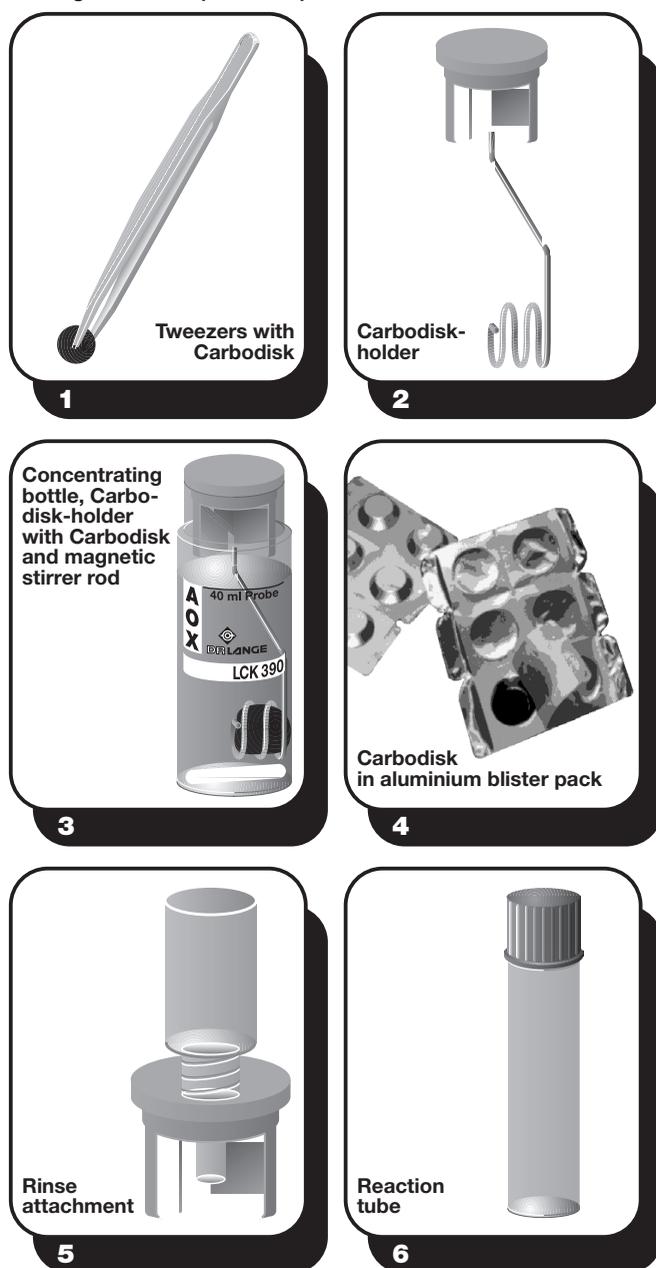
The analysis of **adsorbable organic halogens** (AOX) is carried out in several steps:

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.

Range of Application

Waste water, process water

Package contents (AOX-Test)



Starter-Set (LZC 905): (only supplied with a first order)

- Tweezers (Fig. 1)
- Magnetic stirrer rods (Fig. 3)

Concentrating (LZC 390):

- Aluminium blister packs with **Carbodisk** (Fig. 4)
- Rinse attachment (Fig. 5)
- Solution A (LCK 390 A)
- Rinsing solution B (LCK 390 B)
- Digestion solution C (LCK 390 C)
- Digestion reagent D (LCK 390 D)
- Reagent solution E (LCK 390 E)
- Dosing spoon
- Reaction tubes (Fig. 6)

Measurement (LCK 390):

- Cuvettes for the determination of AOX
- **Carbodisk**-holders (Fig. 2)
- Concentrating bottles (Fig. 3)
- Zero-solution cuvette

Note

Magnetic stirrers with an induction field are **not** recommended for the concentration step.

Storage Information

The test reagents are stable at +15 to +25°C up to the expiry date given on the package.

The reaction cuvettes must be stored in a refrigerator.

Interferences

Chloride concentrations of < 3 g/L (inorganic chloride in the waste-water 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 (see section on digestion), 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).

pH/Temperature

The pH of the sample must be between pH 4 and pH 9; the temperature of the sample and reagents must be between 15 and 25°C.

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.

Special note

■ Contamination of the Carbodisk from the atmosphere

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!

■ Contamination of the Carbodisk through contact

The **Carbodisk** should **not** be allowed to come into contact with the skin. **Always** use **tweezers** to remove the **Carbodisk** from the blister packs and to manipulate them during the analysis procedure.

■ Rinsing

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.

■ Thermostat

Preheat the thermostat to **100°C**. Then introduce the reaction tubes and set the reaction time to **2 hours**.

Safety Advice

On grounds of quality and reliability, the analysis should be carried out only with original HACH LANGE accessories.

CADAS 100 (≥ LPG 210)

If this test is not already stored in your instrument please ask your HACH LANGE Agency for programming instructions.

Disposal

The used rinsing solution B (LCK 390 B), the sample solution with added solution A (LCK 390 A), and the digested sample contain no environmentally harmful pollutants and can be disposed of on-site by pouring them down the drain. The **Carbodisk** can be disposed of with normal household refuse.

Procedure of the AOX Test

Edition 97/06

The AOX test procedure consists of 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)

Concentration

- Remove the HACH LANGE magnetic stirrer rod from the concentrating bottle and place it on a clean paper towel.
- Add the sample to the concentrating bottle until the 40 mL mark is reached. A beaker can be used for this purpose.
- Pipette **2 mL of solution A (LCK 390 A)** into the sample.
Take care – the solution is caustic.
- The pH of the sample must be between pH 2 and pH 3. If the pH is higher, it must be adjusted by adding concentrated nitric acid drop by drop.
- Use the tweezers to introduce the magnetic stirrer rod into the concentrating bottle.
- 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 (Fig. 7). The **Carbodisk**-holder should only be held by the rubber stopper.
- Immerse the **Carbodisk**-holder in the sample solution **immediately**. Close the concentrating bottle securely with the rubber stopper.
- Place the concentrating bottle on a magnetic stirrer.
- Gradually increase the stirring rate.
- The optimal stirring rate is reached when the movement of the liquid pulls down the meniscus by about 2 cm (Fig. 8).
- Stir for **15 min**.
- Then switch off the magnetic stirrer.

Preparing the reaction tubes

- Switch on the thermostat and set it to **100°C**.
- Add the following to a dry reaction tube (Fig. 6):
Digestion solution C (LCK 390 C): **3.6 mL**
Take care, the solution is caustic!
Digestion reagent D (LCK 390 D): **1 level dosing spoon**



- The amount of digestion reagent D (LCK 390 D) may not be significantly increased. For this reason, great care must be exercised to take only a level dosing spoon of the digestion reagent D (LCK 390 D).
- Dissolve the digestion reagent D (LCK 390 D) completely in the digestion solution C (LCK 390 C).
- Close reaction tube and allow to stand.

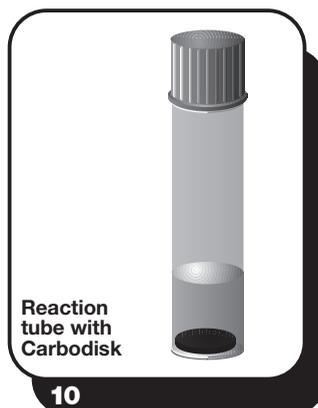
Rinsing

- Remove the rubber stopper with **Carbodisk**-holder from the concentrating bottle and remove the liquid out of the bottle down to the drain. The magnetic stirrer rod must remain in the bottle.
- Plug the rinse attachment onto the bottle.
- 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 (Fig. 9). Press down lightly on the **Carbodisk** with the tweezers.
- To remove the inorganic chloride, use a piston pipette (hold the pipette vertically) to **quickly** add **5 mL** rinsing solution B (LCK 390 B) **four times**, directing the solution onto the **centre** of the **Carbodisk** each time.
- 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 (LCK 390 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** (Fig. 9).
- 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.
- **Removal of the Carbodisk**
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) (Fig. 10).
- Close the reaction tube **immediately** and invert a few times.



Digestion

- Heat the reaction tube for **2 h** at **100°C** in the preheated thermostat.
- Remove the reaction tube, invert it briefly and allow it to cool to room temperature. The cooling process should take a least **45 min**.
Turbidities in the digested sample are eliminated by filtration through a 0.45 µm membrane filter (LCW 916). (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.



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**.

DR 1900

LCK 390

AOX

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Evaluation

- Select »Barcode-programs«.
- Select test number (see below).
- Control number must be **5**.
- Insert zero-solution cuvette and press "Zero".
- Insert sample cuvette and press "Read".

Parameter	Test-No.	Meas. range
AOX	390	0.05 – 3.0 mg/L

Procedure

LCK 390

Applies to all types of photometer

AOX

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Pipette into the cuvette test	
Reaction solution E (LCK 390 E)	0.3 mL
Of the clear supernatant liquid of the digested sample (reaction tube)	1.5 mL

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

Close cuvette and invert a few times. After **3 min** thoroughly clean the outside of the cuvette and evaluate. **(do not invert cuvette before carrying out the measurement!)**

Data table

LCK 390

LP2W	97/06
AOX • $F_1 = -2.501$ • $F_2 = 4.594$ • $F_3 = 2.671$ • $K = 0.025$	
CADAS 30/30S/50/50S	97/06
AOX • λ : 468 nm • Pro.: 15 • $F_1 = -1.892$ • $F_2 = 4.108$ • $F_3 = 2.689$ • $F_4 = 1.0$ • $K_1 = 0.025$ • $K_2 = 0$	
ISIS 6000/9000	97/06
AOX • λ : 465 nm • Pro.: 15 • $F_1 = -3.037$ • $F_2 = 5.048$ • $F_3 = 2.507$ • $F_4 = 1.206$ • $K_1 = 0.029$ • $K_2 = 0$	
CADAS 100 / \geq LPG 210	97/06
AOX • λ : 468 nm • $F_1 = -1.814$ • $F_2 = 3.945$ • $F_3 = 2.769$ • $F_5 = 0.032$	

LASA 20

LCK 390

AOX

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Evaluation

- Press any key.
- Check program control number: **__ : 38**
- Select test with \uparrow or \downarrow key.
- Insert zero-solution cuvette.
- Insert sample cuvette.

Parameter	Display	Meas. range
AOX	AOX LCK 390	0.05 – 3.0 mg/L

AOX

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Evaluation

This evaluation is possible from the Eprom-version 2.7.

1. Insert program filter **470 nm**.
2. Press "Tests" key until display (see below) appears.
3. Control number must be **1**.
4. Insert zero-solution cuvette and press "Null" (zero) key.
5. Insert sample cuvette and press "Ergebnis" (result) key.

Parameter	Display	Meas. range
AOX	AOX LCK 390	0.05 – 3.0 mg/L



AOX

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Evaluation

1. Check program control number:
__ : **38 (CADAS 30S/50S/200 Barcode)**
2. Insert zero-solution cuvette.
3. Insert sample cuvette.

This evaluation is possible from the Eprom-version:

Version 1.8 (CADAS 30)

Version 1.9 (CADAS 50)

Parameter	Meas. range
AOX	0.05 – 3.0 mg/L

AOX

Edition 97/06

Evaluation

1. Check program control number:
__ : **38 (CADAS 200)**
__ : **38 (ISIS 6000)** ⇒ Select »CUVETTE TEST« mode.
2. Select test number (see below).
3. Control number must be **5**.
4. Insert zero-solution cuvette and press blue key.
5. Insert sample cuvette and press green key.

Parameter	Test-No.	Meas. range
AOX	390	0.05 – 3.0 mg/L

AOX

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Evaluation

1. Select »TEST« mode.
2. Select symbol (see below).
3. Control number must be **9**.
4. Insert zero-solution cuvette and press "NULL" (zero) key.
5. Insert sample cuvette and press "MESS" (measure) key.

If more than one sample is to be measured start the next evaluation at point 5.

Parameter	Symbol	Meas. range
AOX	390	0.05 – 3.0 mg/L