REF 985 007 Test 0-07 04.17 NANOCOLOR® AOX 3

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Adsorbable organically bound halogens

Method

AOX determination is carried out in 3 steps

- 1. Solid phase extraction with NANOSORB for AOX
- 2 Decomposition of the concentrated adsorber medium
- 3. Determination as chloride with reagent set *NANOCOLOR®* AOX 3

0.01-0.30 mg/L AOX Measuring range: 0.1-3.0 mg/L AOX Method: (0)071 (0)072Wavelength (HW = 5-12 nm): 470 nm

Reaction time 3 min (180 s) Reaction temperature: 20-25 °C

Contents of the reagent set: 20 NANOSORB cartridges

- 1 preparation box containing
- 2 x 100 mL rinsing solution concentrate for preparation of AOX 3 R1 (fill up each to 1 L with dist. water) 1 tube NANOFIX AOX 3 R2
- 1 x 105 mL AOX 3 R3
- 1 x 75 mL AOX 3 R4
- 20 reaction tubes 16 mm OD
- 1 detection box containing
- 20 test tubes AOX 3
- 2 test tubes Chloride R2
- 1 test tube with blank value "NULL"

Hazard warning:

Reagent R2 contains sodium peroxodisulfate 20-100%, reagent R3 contains sodium hydroxide solution 0.5–2 %, reagent R4 contains nitric acid 3–5 %, test tubes contain nitric acid 5–20 %, reagent Chloride R2 contains mercury(II) thiocyanate 0.32–0.64 % in methanol 50–100 %.

H301, H311, H314, H317, H331, H334, H370 Toxic if swallowed. Toxic in contact with skin. Causes severe skin burns and eye damage. May cause an allergic skin reaction. Toxic if inhaled. May cause allergy or asthma symptoms or breathing difficulties if inhaled. Causes damage to organs.

P260, P261, P264, P270, P272, P280, P301+310, P301+330+331, P302+352, P303+361+353, P304+340, P305+351+338, P308+311, P333+313, P342+311, P361+364, P405, P501 Do not breathe vapors. Avoid breathing dust. Wash with water thoroughly after handling. Do no eat, drink or smoke when using this product. Contaminated work clothing should not be allowed out of the workplace. Wear protective gloves/eye protection. IF SWALLOWED: Immediately call a POISON CENTER/doctor/... IF SWALLOWED: Rinse mouth. Do NOT induce vomiting. IF ON SKIN: Wash with plenty of water/... IF ON SKIN (or hair): Take off immediately all contaminated clothing. Rinse skin with water/shower. IF INHALED: Remove person to fresh air and keep comfortable for breathing. IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing. IF exposed or concerned: Call a POISON CENTER/doctor/... If skin irritation or rash occurs: Get medical advice/attention. If experiencing respiratory symptoms: Call a POISON CENTER/doctor/... Take off immediately all contaminated clothing and wash it before reuse. Store locked up. Dispose of contents/container to regulated waste treatment. For further information ask for a safety data sheet.

COD-contaminated test solution (> 50 mg/L COD) must be diluted beforehand. Alternatively, the NANOCOLOR® Supplement kit for AOX (up to 1000 mg/L COD, REF 918 072) can be used.

When using 200 mL of rinsing solution, this method is also suitable for analyzing sea water.

Procedure:

Requisite accessories: Starter kit for AOX (REF 916 111), piston pipette with tips; optional: supplement kit (REF 918 072), pump set for AOX (REF 916 115)

1a. Manual extraction

Connect a NANOSORB cartridge to the syringe 50 mL with the aid of an adaptor. Pour 100 mL test sample (the pH value of the sample must be between pH 3 and 5) into a glass beaker 150 mL, dip the NANOSORB cartridge into the test sample and lift the syringe plunger up and down 20 times to adsorb the organically bound halogens from the sample (accessories: stand with clamp and boss)

After extraction disconnect NANOSORB cartridge from the adaptor and syringe. Rinse the

NANOSORB cartridge slowly in 4–5 portions with a total of

100 mL R1 rinsing solution in order to remove inorganic chloride. Connect the syringe to the cartridge once more and blow out any excess of water from the NANOSORB adsorber with 2 strong draughts of air

1b. Extraction using the pump set

Close valve of the flask. Pour

100 mL test sample (the pH value of the sample must be between pH 3 and 5) or 1000 mL test sample (the pH value of the sample must be between pH 3 and 5) for the sensitive range into the flask and connect a NANOSORB cartridge to the flask using the adaptor. Open valve and start pumping for 20 min to adsorb the organically bound halogens from the sample.

After extraction disconnect the NANOSORB cartridge from the adaptor and flask. Rinse the NANOSORB cartridge in 4-5 portions with a total of

100 mL R1 rinsing solution in order to remove inorganic chloride

Connect the syringe to the cartridge using the adaptor and blow out any excess of water from the NANOSORB adsorber with 2 strong draughts of air.



2a. Decomposition if COD content is low, without supplement kit, using a heating block

Add into a reaction tube 16 mm OD 1 NANOFIX R2 and

- 5 mL R3 close and mix

Open and insert the **NANOSORB** to this solution with help of a funnel, then press it down to the bottom of the tube with tweezers. Close the tube, place it into the heating block and heat at 120 °C for 30 min. Remove tube from heating block, shake gently and leave it to cool. Open tube, add

2b. Decomposition if COD content is low, without supplement kit, using a microwave

- 1 NANOFIX R2 and
- 5 mL R3, close and mix

Open and add the **NANOSORB** to this solution using tweezers. Add a glass rod to the vessel to prevent the **NANOSORB** from swimming on the surface. Close the decomposition vessel. Place it on the outer edge of the microwave revolving plate and heat 23 s at 900 VA or 28 s at 750 VA (always use the highest power rating of your microwave oven).

Remove vessel from microwave and let cool for about 10 min. Turn the pressure vessel upside down once and open it with caution. Add

3.5 ml B4 close and mix

2c. Decomposition if COD content is high, with supplement kit, using a heating block

Add into a reaction tube 16 mm OD

- 1 NANOFIX R2
- 1 black spoon R5 and
- 5 mL R3, close and mix

Open and insert the **NANOSORB** to this solution with help of a funnel, then press it down to the bottom of the tube using tweezers. Close the tube, place it into the heating block and heat at 120 °C for 30 min. Remove tube from heating block, shake gently and leave it to cool. Open tube, add

3.5 mL R4 and

1 orange spoon R6 (the solution becomes turbid), close and mix. Filter the solution with membrane or folded filters

2d. Decomposition if COD content is high, with supplement kit, using a microwave

Add to the decomposition vessel

- 1 NANOFIX R2
- 1 black spoon R5 and
- 5 mL R3, close and mix

Open and add the NANOSORB to this solution using tweezers. Add a glass rod to the vessel to prevent the NANOSORB from swimming on the surface. Close the decomposition vessel. Place it on the outer edge of the microwave revolving plate and heat 23 s at 900 VA or 28 s at 750 VA (always use the highest power rating of your microwave oven).

Remove the vessel from the microwave and let it cool for about 10 min. Turn the pressure vessel

upside down once and open it with caution. Add

3.5 mL R4 and

1 orange spoon R6 (the solution becomes turbid), close and mix. Filter the solution with membrane or folded filters.

3. Determination of AOX

Open test tube AOX and add

4.0 mL decomposition solution (let particles of adsorbent deposit or use membrane filters). Add

1.0 mL Chloride R2, close and mix.

Clean outside of test tube and measure after 3 min

Adjust photometer to zero by using blank value "NULL"

Measurement:

For using MACHEREY-NAGEL photometers see manual, test 0-07.

Photometers of other manufacturers:

For other photometers check whether measurement of round glass tubes is possible. Verify calibration curve for each type of instrument by measuring standard solutions

Analytical quality control:

NANOCONTROL AOX 3 (REF 925 07)

Reference:

German Standard Methods for the Examination of water, waste water and sludge (DIN EN 1485 H14 and DIN 38409 H22)