Sample preparation for liquid AOX samples by column method

AOX sample preparation

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Find your way for AOX sample preparation

Column sample preparation for fast, easy, and automated analysis.

AOX analysis for wastewater, surface water, and river water is regulated by many international standards. Sample preparation is usually done either by batch method or column method. But which is the best way for sample preparation?

Column method	*ISO 9562; 100 mL sample*	Batch method
Fit pre-filled column into column. Fill vial with 100 mL water sample. APU sim: 6 samples in < 10 min APU 28 connect: 28 samples in < 30 min	Steps before adsorption starts	Add 50 mg charcoal into Erlenmeyer flask. Fill vial with 100 mL water sample. 1 batch sample ~ 1 min
APU sim: 6 samples in 45 min APU 28 connect S: 28 samples in 12 hours APU 28 connect SPE: 28 samples in 24 hours	Sample preparation, adsorption, and rinsing	Shake each sample for 60 min and filter each sample ~ 1 min (particles may impact time). Rinse each sample.
▪ Scheduled workflow ▪ Fully automated routine ▪ Overnight operation for higher throughput	Conclusion	▪ Total time per sample is not predictable (particles) ▪ Operator's full attention is required

While batch method is often the method of choice, it is very tedious, requires the full attention of an operator, and the total time per sample is not predictable, especially when particle-containing samples need to be analyzed.

The Analytik Jena solution for column method in turn, can easily deal with particles, offers different degrees of automation, and reduces handling steps and likely errors. This makes AOX results more comparable and saves time in your lab work, as well as costs for analysis.

Figure 1: APU 28 connect

Your benefits using the column method

- Defined time per sample
- Automated workflow
- Most cost-effecient routine
- Comparable results

Challenges in AOX analysis of aqueous samples

Water samples that are analyzed according to e.g., ISO 9562, EPA 1650C, and EPA 9020B can contain particles and/or a high level of salts. Inorganic salts in water samples greatly interfere with adsorption on the surface of activated carbon. Particles lead to the fact that filtration steps in the batch method cannot be terminated. Besides, hoses and/or columns of conventional column systems can become clogged. Analytik Jena offers solutions for both problems.

Particle-containing samples

The column method utilizes at least two pre-filled quartz columns with 2 x 50 mg of active carbon that can be tested individually and give an indication about completion of adsorption (breakthrough). Hence, operators gain ensurance of correct AOX results.

In addition to the duplex column holder, Analytik Jena offers a triplex solution that improves handling of particle-rich samples. The third column is also made from quartz, containing quartz wool and is installed in line with the AOX columns (pre-filter).
Particles are trapped by the quartz wool, and the AOX from aqueous samples can readily adsorb to the active carbon, avoiding clogging of columns.

Finally, active carbon columns loaded with AOX are ready for analysis and the pre-filter can also be submitted to the AOX analysis. By doing that, operators can further distinguish between particle-bound AOX and dissolved AOX.

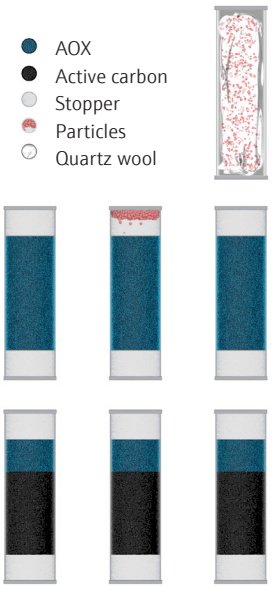

Figure 2: left: columns with particle-free AOX sample, middle: columns with particles clogging first column, right: columns with pre-filter (triplex) separating high particle load from AOX sample

Salt-containing samples

For samples with high inorganic salt load, the AOX content of an aqueous sample is often found to be more evenly distributed among both AOX columns. As inorganic chloride competes for adsorption sites on the active carbon of the first column, the resulting AOX value is not reliable. Hence, the inorganic chloride ought to be separated from aqueous samples prior to the AOX adsorption, and for that operators make use of the SPE-AOX approach.

The SPE-AOX approach comprises the following steps:

1) Conditioning of SPE cartridge with methanol
2) Addition of sample containing inorganic salt and AOX, the latter adhering to polymer
3) Rinsing with H_2O or suitable washing solution containing nitrate to remove inorganic salt
4) Elution of AOX from polymer into transfer flask

Subsequently, the obtained sample is submitted to AOX adsorption onto the active carbon in the AOX columns.

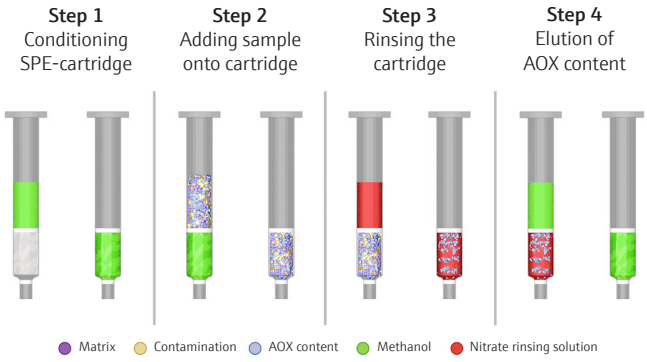

Figure 3: SPE – preparation steps prior adsorption onto active carbon

Disadvantages of batch method (particle-containing samples)

- 1 x 50 mg only
- No information on completion of adsorption (breakthrough)
- Uncertainty of correctness of results
- No means to separate particles
- No species information (bound versus dissolved)
- No automation of workflows

Disadvantages of batch method (SPE-AOX)

- Extra accessory for SPE sample preparation is required in addition
- No automation of workflow
- Uncertainty of correctness of results
- Risk of exposure to volatile methanol

Product information

Automation is a decisive factor for an efficient sample preparation process. The ideal partner for **APU 28 connect** is Analytik Jena's AOX analyzer **multi X 2500**. Together with the autoX 112 autosampler, the setup allows for the processing of 112 samples in 24-hour operation.
The multi X 2500 is universally applicable for the analysis of samples prepared by column or batch method. Other applications include EOX, POX, determination of TOC in wastewater, and TCl in organic solids and liquids.

Also from Analytik Jena's portfolio of AOX sample preparation systems is the **APU sim**. It is ideal for small sample numbers, offering simultaneous enrichment of up to 6 samples by column method. The system is well suited to handle particulate and saline samples.

Figure 4: APU sim

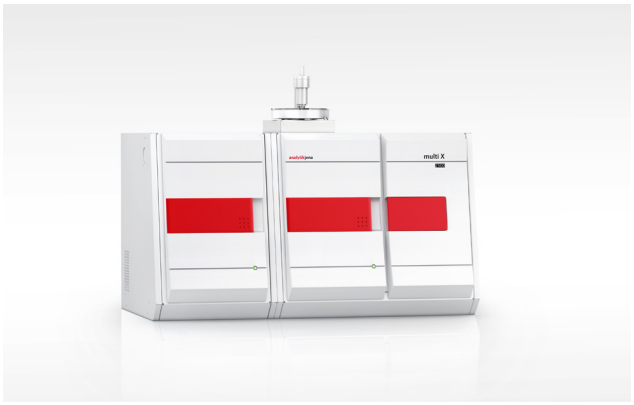

Figure 5: multi X 2500 AOX analyzer

www.analytik-jena.com/aox-samplepreparation

Headquarters

Analytik Jena GmbH+Co. KG
Konrad-Zuse-Straße 1
07745 Jena / Deutschland

Tel +49 3641 77 70
Fax +49 3641 77 9279
info@analytik-jena.com
www.analytik-jena.com

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