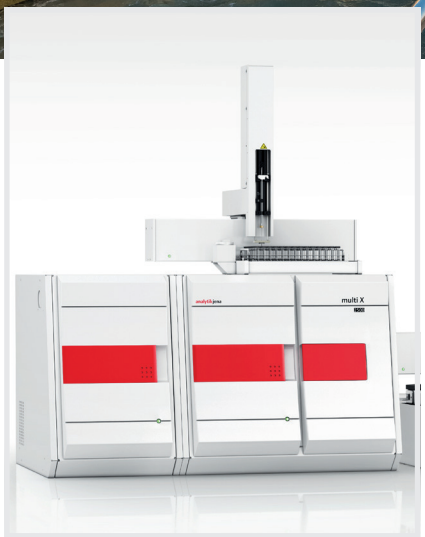

Challenge

To pass the linearity test according to DIN EN ISO 9562

Solution

Fast and cost-effective procedure applying adsorption of the AOX samples on an automatic preparation unit followed by analysis with an AOX analyzer

Linearity Test According to DIN EN ISO 9562 (Column Method)

Introduction

AOX (AOX - adsorbable organically bound halogens) is an analytical convention which represents the sum of all organically bound halogens (except fluorine), mainly used in water and waste water analysis. Solid samples like sludge or sediments can also contain AOX. The solid AOX however has to be determined by the batch method which is not part of this application. The compounds containing halogens are adsorbed onto the surface of activated carbon (charcoal) in dissolved or suspended form. The activated carbon is washed afterwards with aqueous nitrate washing solution to remove inorganic halides. During the combustion of the loaded charcoal in pure oxygen at high temperatures hydrogen halides are formed. After absorption of the halides the final detection takes place inside a coulometric cell by means of argentometric titration.

Instrumentation

A multi X® 2500 AOX analyzer equipped with an autoX36 auto sampler was used in combination with an automatic sample preparation unit of the APU series. Thanks to the perfect combination of sample preparation and analysis idle time is minimized and sample throughputvis increased. The multiWin software allows fast sample analysis and data evaluation simultaneously. In combination with the Self Check System the multi X® 2500 ensures trouble-free and fully automated operation.

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Samples and Reagents

The following reagents were used for sample and standard preparation

- Deionized water/ ultra-pure grade
- Nitric acid >65% p.a.
- p-chlorophenol aqueous stock solution; 1 mg/l Cl; (Bernd Kraft GmbH)

Standard preparation

The p-chlorophenol stock solution was diluted with ultra-pure water according the ISO 9562 to obtain different concentration as follows: 10, 50, 100, 200 and 250 µg/l.
100 ml of these solutions was adjusted to pH 2 with concentrated nitric acid. The low pH value inhibits bacterial growing and enables the storage of the samples at 0 - 4 °C.

Determination

The determination was carried out using a multi X® 2500 AOX analyzer. The adsorption of the AOX samples was performed on an automatic preparation unit of the APU series.
p-chlorophenol was used as organic standard solution to evaluate the system and method in terms of sample recovery. For this a linearity test was performed. According to the column method, two quartz glass containers filled with activated carbon were used. Both are positioned vertically, one behind the other. The different standard solutions were adsorbed with a flow rate of 3 ml/min. After rinsing with 25 ml washing solution at the same flow rate the columns with the loaded charcoal were combusted at 950 °C in pure oxygen stream.

Results and Discussion

According to DIN EN ISO 9562 the results are acceptable if the correlation coefficient is ≤ 0.999. The slope of the linearity should be within 0.95 and 1.05. The results of the determinations are shown in table 1.

Table 1: Results of the linearity test of the AOX determination of p-chlorophenol in water

Standard concentration [µg/l]	Abs. value 1st column [µg Cl]	Abs. value 2nd column [µg Cl]	Blank value [µg Cl]	Recovery [µg/l]	Average [µg/l]
10	1.08	0.06	0.265	8.75	**9.65**
10	1.08	0.24	0.265	10.55	
50	4.86	0.09	0.265	46.85	**47.7**
50	5.01	0.11	0.265	48.55	
100	9.63	0.17	0.265	95.35	**96.0**
100	9.79	0.14	0.265	96.65	
200	19.38	0.27	0.265	193.85	**194.1**
200	19.35	0.35	0.265	194.35	
250	24.02	0.33	0.265	240.85	**242.65**
250	24.43	0.28	0.265	244.45	

Two replicate measurements have been performed for each standard concentration level. According to the column method two pre-filled charcoal containers were used for each sample. Each container was filled with 50 mg of activated carbon. The absolute value shows the chlorine value of each single container. The result of standard concentration is already blank corrected. The average of the two replicates is given in bold numbers. For each standard recovery (shown in table 2) the obtained and the theoretical chlorine value should not differ more than 10 %; e.g. 90 % up to 110 %.

Table 2: Standard recovery of p-chlorophenole

Min. value [µg/l]	Theoretical AOX value [µg/l]	Max. value [µg/l]	Obtained AOX value [µg/l]
9	10	11	9.7
45	50	55	47.7
90	100	110	96.0
180	200	220	194.1
225	250	275	242.7

The recovery rates, correlation coefficient, and slope of the linearity test fulfill the specifications of the DIN EN ISO 9562. This confirms a correct adsorption behavior of the APU system and the excellent analytical performance of the analyzer.

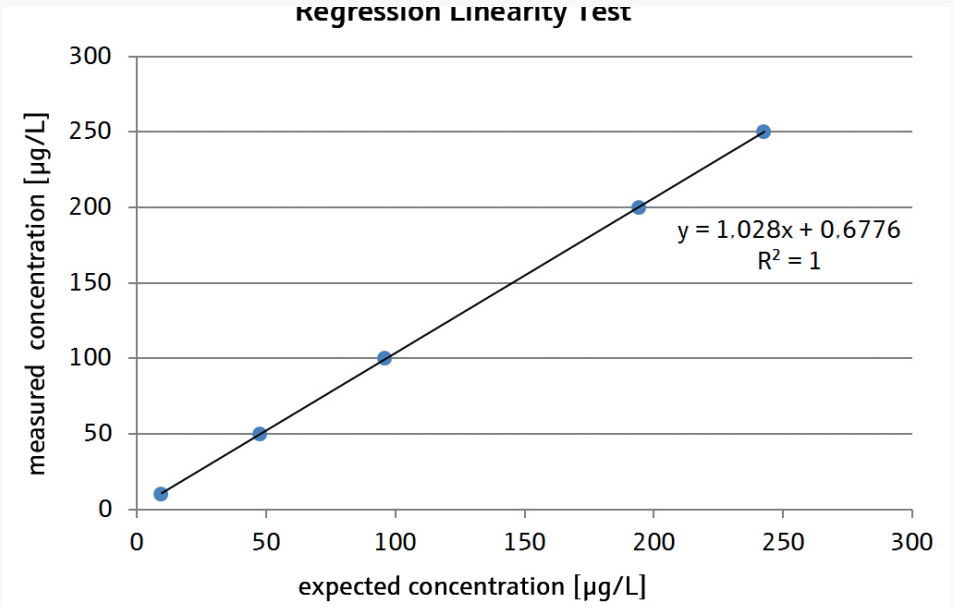

Figure 1: Regression Linearity Test

The parameters of the linear regression are as follows: $y = 0.9728x - 0.6565$ with $R^2 = 1$.

Conclusion

This linearity test shows that the multi X® 2500 AOX analyzer and the APU sample preparation unit provide accurate standard recovery. This enables fast and reliable analysis for water quality monitoring. Due to the high automation level, the APU sample preparation unit allows fast sample preparation without any manual handling. An overnight sample treatment followed by analysis with multi X® 2500 during day time is extremely timesaving. The AOX analysis by column method is cost-effective compared to the time intensive batch method.

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