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LINEARITY STUDIES IN DETERMINATION OF TRIHALOMETHANES IN DRINKING WATER BY GAS CHROMATOGRAPHY

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Introduction

To ensure the of drinking water quality, during the treatment process, one of the most important steps is the disinfection of water.

Depending on the composition of the treated water, during the chlorination stage, several secondary compounds can appear, such as trihalomethanes, which in concentrations exceeding the allowed limits can bring negative effects on the population's health. Trihalomethanes (THMs) are environmental pollutants, chemical compounds, in which three of the four hydrogen atoms of methane (CH₄) are replaced by halogen atoms. Due to the great impact that trihalomethanes can have on health, the need to monitor them as well as the need to optimize gas chromatographic methods of determination have been intensely deepened lately.

The aim of this study was to demonstrate the linearity relationship between the concentrations of the analyte solutions of interest that are injected into the chromatographic column and the areas of the peaks generated in the corresponding chromatograms. It is desired to determine the analyte concentration that generates a distinct signal in relation to the background noise, but without allowing its accurate dosing (LOD detection limit).

Materials and methods

THMs (trichlor-methane (CHCl₃), brom-dichlor-methane (CHBrCl₂), (chlorodibromomethane) CHBr₂Cl, bromoform (CHBr₃)) was extracted with isooctane and detected using gas chromatography (Agilent Technologies, USA). The final concentrations were calculated using the method of the external standard such as STAS 12997-91.

The method operational parameters was: chromatographic column 60 m × 0.25 mm, fused silica, oven temperature of 35°C, injection volume of 1 μL and flow rate of 5 mL/min.

In this study the source water was Argeș river, and the analyzed sample was obtained after the chlorination stage.

The determination of THM was made by gas chromatographic technique according to STAS 12997-91.

The stock solutions of 200 ppm was diluted to obtain different concentrations of 5 ppb, 10 ppb, 15ppb, 40 ppb, 50 ppb, 70 ppb, 80 ppb, 90 ppb, 100 ppb and 150 ppb. Linearity was detected by injecting a series of stock solution/diluted stock solution standards using the solvent/mobile phase at a minimum of five different concentrations in the range of 50 - 150% of the expected working range. All the studies were performed using the gas chromatograph (HP 6890, Agilent) and the individual standards for THMs used in the linearity studies were from RESTEK (France).

Results and conclusions

As it results from the graphical representations, the correlation coefficient respected the imposed conditions, R Square > 0.99 (Figure 1).

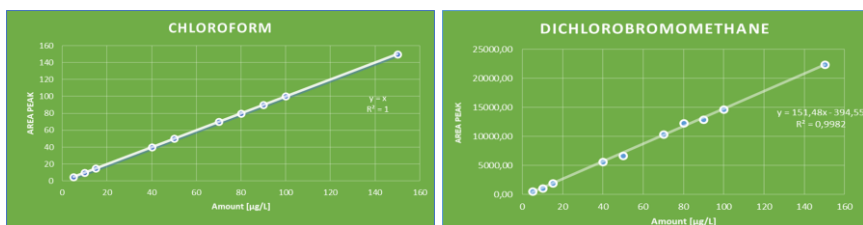


Fig. 1. Calibration curves for chloroform and dichlorobromomethane

The range of THM concentration to verify the linearity was 5 - 150 ppb. Over the studied range, the proposed chromatographic method showed linearity between the areas of the chromatographic peaks and the analyte concentration (amount). Linearity was verified for all the THMs components (CHCl_3 , CHBrCl_2 , CHBr_2Cl , CHBr_3) and indicated that the best results were obtained for chloroform (R Square=1).

The detection and quantification limits were calculated according with the following equation: $\text{LOD} = (3,3 * S_{y/x}) / b$, were $S_{y/x}$ =standard deviation
 b = slope

The obtained values for the detection limits were: 8,73 ppb for chloroform; 8,64 ppb for dichlorobromomethane; 9,22 ppb for dibromochloromethane and 8,59 ppb for bromoform. These values were within the legislation Low 458/2002 for THMs from water.