

9. SIMPOZIJUM  
HEMIJA I ZAŠTITA  
ŽIVOTNE SREDINE

# ENVIROCHEM2023

*9<sup>th</sup> SYMPOSIUM  
CHEMISTRY AND ENVIRONMENTAL PROTECTION*

ENVIROCHEM2023

KNJIGA IZVODA

*4-7. jun 2023. godine, KLADOVO, SRBIJA*

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*BOOK OF ABSTRACTS*

9. simpozijum  
Hemija i zaštita životne sredine  
EnviroChem2023  
sa međunarodnim učešćem



*9<sup>th</sup> Symposium*  
*Chemistry and Environmental Protection*  
*EnviroChem2023*  
*with international participation*

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## Simplified validation and optimization methods for determination polychlorinated biphenyls in tuna fish

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Polychlorinated biphenyls (PCBs) represent important environmental pollutants from the group of persistent organic pollutants. Although the use of PCBs is banned in many countries, but they still figure in the environment. Due to its lipophilic character and accumulation in fats, food of animal origin is the one that is most exposed to accumulation and the possibility of the presence of PCB residues [2, 3]. It can be said that fish can be considered environmental indicators. Consumption of fish and fish products is growing globally, because consumers see fish as a natural and safe product. Fish safety control and testing for the presence of PCBs is important because it is an indicator of the state of the environment.

The aim of this research is to present the current determination of PCBs in fatty tissue of tuna fish. Gas chromatography coupled with mass spectrometry (GC/MS) was used for the detection of selected polychlorinated biphenyls. The work was performed for validation of the most important congeners (PCB 28, 52, 101, 153, 138 and 180) which are defined by national and international legislation.

The first step in the analysis was the validation and optimization of the determination method with simple and quick preparation using commercial extraction kits. The validation of basic parameters was carried out according to the guidelines of the SANTE 11312/2021 guide for precise control of pesticides in food (European Commission, 2021). Calibration curve, correlation coefficient, limit quantification (LOQ), recovery for the spike level of 10 µg/kg and precision are shown in Table 1. The tuna fish matrix was used for validation, because it is necessary that the matrix of all the examined samples be identical with the matrix used for calibration. The linear calibration coefficient was between 0.9968 and 0.9999 and limit quantification ranged between 2.5 and 3.8 µg/kg (Table 1). Table 1 shows the signal-to-noise ratios for individual PCBs obtained at the lowest point of calibration, i.e. the value of the PCB standard of 10 µg/kg. The value of S/N for the mass spectrum acquisition frequency of the examined congeners were greater than 50. The enhancement in S/N is largely attributed to moving the signal to a higher frequency domain, and a higher value confirms greater accuracy, selectivity and precision in quantification. The signal-to-noise ratio for the peak congeners of PCB 153 in the fish matrix is shown in Figure 1. The validated method was used for the analysis of PCBs present in 30 samples of frozen tuna from the Adriatic Sea.

Extraction and clean-up procedural steps were applied with some modifications of the original developed QuEChERS method. The analysis was conducted on fish samples that

were frozen before analysis and measured after homogenization. The content of polychlorinated biphenyls is expressed as the amount of micrograms of PCB on wet fish in order to comply with the units (ng/g of wet weight) prescribed by the regulation. The presence of tested PCBs was not confirmed in the tested tuna samples.

Table 1. Basic validation data for selected polychlorinated biphenyl obtained by using GC/MS

Congener	Equation	R <sup>2</sup>	LOQ, ( $\mu\text{g}/\text{kg}$ )	Recovery $\pm$ (RSD) % spike 10 $\mu\text{g}/\text{kg}$	Single to noise S/N (10 $\mu\text{g}/\text{kg}$ )
PCB 28	$y=1.7187x+8.1233$	0.9968	2.8	112.5 $\pm$ (12.8)	181.4
PCB 52	$y=0.9286x-1.0346$	0.9999	3.6	118.9 $\pm$ (14.1)	90.7
PCB 101	$y=1.0429x-5.2194$	0.9988	2.5	98.2 $\pm$ (11.6)	88.6
PCB 153	$y=0.6871x+14.507$	0.9984	3.8	97.7 $\pm$ (9.7)	98.1
PCB 138	$y=0.7099x-2.3009$	0.9981	3.6	108.9 $\pm$ (16.8)	51.6
PCB 180	$y=0.5010x+3.2130$	0.9974	3.5	119 $\pm$ (14.2)	52.6

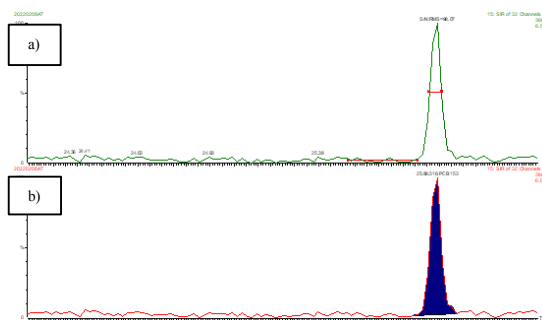


Figure 1. Chromatograms of PCB 153 in SIM mode. Spiked blank tuna fish sample after addition of 10  $\mu\text{g}/\text{kg}$  standard of congener, extraction and clean-up. a) Estimation of ratio single to noise (S/N) b) Peak area

The validated method can also be applied to routine analysis and monitoring of processed tuna fish products on the Serbian market and improve food safety.

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## Organic acids as solvents for leaching PAHs out of impregnated wood