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## EVALUATION OF THE CONTENT OF CHLOROPHENOLS AND THEIR DERIVATIVES IN WATER SUPPLY SYSTEMS

### OCENA ZAWARTOŚCI CHLOROFENOLI I ICH POCHODNYCH W WODACH WODOCIĄGOWYCH I RZECZNYCH

**Abstract:** There is currently great environmental interest in chlorinated phenol derivatives on account of their high toxicity and wide industrial use. Surface water contains many inorganic and organic compounds both of natural and anthropogenic origin. The composition of the specified compounds in aquatic ecosystems is related to the influence of different natural factors or a variety of human activity. Aromatic compounds, due to their toxicity and persistency in the environment, establish serious danger not only for living organisms but also for the biocenosis. One of the most important group of ecotoxins are chlorophenols. They exhibit high toxicity including genotoxic, mutagenic and cancerogenic activity. Moreover, substitution of these compounds by chlorine atoms may increase their toxic action and prolong the period of their bioaccumulation in living organisms. Chlorination is one of the method which lets to remove pathogens, organic matter and xenobiotic from water. This process uses oxidants like chlorine or chlorine dioxide. Chlorination leads to transformation part of recycled compounds in forms which can be more toxic and dangerous than their precursors. This process leads also to formation of new chloroorganic compounds like halomethanes, chlorinated aromatic hydrocarbons and a lot of other organic compounds which can be toxic. Their concentration in drinking water depends on the concentration of the precursors and dose of chlorine which is used in the water purification process. The aim of this work was to determine the occurrence of phenol, chlorophenols, chlorocatechols and chlorinated metoxyphenols both in drinking water of Warsaw and Tomaszow Mazowiecki, and surface water taking from Vistula and Pilica Rivers. The chromatographic analyses were made using a gas chromatograph connected with a mass spectrometer.

**Keywords:** phenol, chlorophenols, chlorination, chlorine oxidants

Water is one of the main elements of the environment which determine the existence of life on the Earth, affect the climate and limit the development of civilization.

Water resources management requires constant monitoring in terms of its qualitative-quantitative values. Proper assessment of the degree of water pollution is the basis for conservation and rational utilization of water resources.

Aromatic compounds, due to their toxicity and persistence in the environment, establish serious danger towards living organisms, including humans. One of the most important group of ecotoxins are phenol and chlorophenols [1, 2]. The presence of phenol compounds in water significantly impairs the taste and smell of the water. Thus, phenol can be organoleptically detected in water at concentration of  $10\div 30 \mu\text{g}/\text{dm}^3$  [3].

Phenol (1-hydroxybenzene) is a colorless, crystalline substance of characteristic odour, soluble in water and organic solvents [4]. Phenol was one of the first compounds inscribed into The List of Priority Pollutants by the US Environmental Protection Agency (US EPA). Phenol is synthesized on an industrial scale by extraction from coal tar as it is formed by transformation of high quantities of cumene present in plants that were used for tar production. Hydroxybenzene is also obtained in a reaction between chlorobenzene and sodium hydroxide, toluene oxidation and synthesis from benzene and propylene [5, 6].

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Chlorophenols are the most widespread and the largest group of phenols, that are used in a number of industries and products. Those compounds are formed as a result of use and degradation of phenolic pesticides such as 2-chlorophenol, 4-chlorophenol, phenoxyherbicides such as 2,4-dichlorophenoxyacetic acid (2,4-D). Exposure to high levels can cause damage to the liver and immune system. These compounds are also formed in the environment by chlorination of mono- and polyaromatic compounds present in soil and water. In drinking water there are present as a result of the chlorination of phenols during disinfection, as by-products of the reaction of hypochlorite with phenolic acids, as biocides, or as degradation products of phenoxy herbicides. Those most likely to occur in drinking water as by-products of chlorination are 2-chlorophenol, 2,4-dichlorophenol and 2,4,6-trichlorophenol.

Chlorophenols are also present in drinking water due to the substitution of organic matter and low molecular weight compounds (present in purified water) with chlorine atoms derived from inorganic chlorine oxidants [7].

The appearance of metoxyphenols including those substituted with methoxy residues in the second (guaiacols) and in the second and sixth positions (syringols) is related with plant decay in which lignin contained in cell's walls transformed. In a few last decades the intensive usage of wood in combustion processes also led to the excessive emission of metoxyphenols to the atmosphere and thus to other systems of the environment. The processes of wood pulp chlorination employed during paper production are a very important source of these compounds (including chlorinated) in water. This type of activity generates many chlorinated compounds including high substituted chlorinated metoxyphenols. Phenols, chlorophenols, and chlorocatechols are usually used as precursors and components of many chemicals including dyes, plastics, resins and pharmaceuticals. The compounds are also formed as a result of use and degradation of pesticides and biphenyls. Some of described compounds have natural origin. Phenol is formed during decomposition of organic matter, catechol is biosynthesized by plants and fungi create chlorinated metoxyphenols [8].

Phenol, catechol, chlorophenols and metoxyphenols exhibit high toxicity including mutagenicity and carcinogenicity [9, 10]. Most of these compounds reveal acute toxicity as they easily penetrate cell membrane and finally exert necrosis of skin and organ within the body. Clinical investigation showed that people exposed to drinking water contaminated with chlorophenols suffer from infections, dermatitis, irritation of digestive tract and strong exhaustion. Moreover, substitution of these compounds by chlorine atoms may increase their toxic action and prolong the period of their bioaccumulation in living organisms.

Described compounds may be transformed in water as they undergo the influence of many abiotic and biotic factors including UV irradiation, metal oxides and temperature influence and microbes and plants activity. The transformation processes lead to increase in the diversity of phenols and some of them are characterized by strong toxicity (stronger, than their precursors) towards water organisms.

### **Collecting samples**

Samples of drinking water and river water were collected in Warsaw and Tomaszów Mazowiecki six times within a year (in period of January, March, May, July, September and November 2008).

In Warsaw tap water was collected from the area supplied by Central Water Supply that pumps it from the Vistula River. Drinking water in Tomaszow Mazowiecki was collected from the area supplied by Water Supply "Tomaszow" that pumps it from the Pilica River.

Tap water was collected after final purification process-chlorination which uses:

- chlorine dioxide ( $0.3\div 0.4$  active chlorine/ $m^3$  water) in Warsaw,
- chlorine ( $0.3\div 0.4$  active chlorine/ $m^3$  water) in Tomaszow Mazowiecki.

Selected areas of the cities were supplied with water exposed to the strongest anthropogenic contamination. The water taken from these sources potentially should have contained the highest amounts and the diversity of compounds analyzed and their precursors.

### Materials and methods

The standards of these compounds (purity: 98÷99%) were bought from Fluka AG: 1-hydroxybenzene (phenol), 2-chlorophenol, 4-chlorophenol, 2,4-dichlorophenol, 2,3,6-trichlorophenol, 2,4,5-trichlorophenol, 2,4,6-trichlorophenol, 2,3,4,5-tetrachlorophenol, pentachlorophenol, 4-methylphenol, 4-chlorocatechol, 3,4,5-trichlorocatechol, tetrachlorocatechol, 2-methoxyphenol (guaiacol), 4,6-dichloroguaiacol, 3-chlorosyringol, trichlorosyringol, 3,4,5-trichloroveratrole. Methanol, dichloromethane, diethyl ether, methylene chloride, acetone, hexane and phosphoric acid of HPLC purity were obtained from Baker JT, USA.

### Preparation of samples

Samples were collected in 1  $dm^3$  volumes in dull HD polyethylene bottles. To inhibit microbes development and oxidation of phenols, 10  $cm^3$  of methanol and 0.1 g of ascorbic acid were added to the samples. Next, 200 g of sodium chloride was added and samples were acidified with phosphoric acid to pH 2.0 to decrease solubility of investigated compounds in water (higher effectiveness of adsorption of phenols by octadecyl layer). Then, the inner standard was added (1.5  $\mu g$  of 2,3,6-trichlorophenol in 1  $cm^3$  of acetone). Finally, samples were mixed using an electromagnetic stirrer (750 r.p.m.) for 30 minutes.

### Solid-Phase Extraction

Adsorption of chlorophenols and their derivatives was performed on "EMPORE Extraction Disks" in a Baker Separex system. The system "EMPORE spe" of extraction filters (disks) was equipped with its binding phase octadecyl C18, diameter of 50 mm and layer thickness of 1.0 mm. The filters were used in a special vacuum extraction apparatus "Baker Separex" made of borosilicate glass. The octadecyl layer was conditioned using 10  $cm^3$  volumes of diethyl ether, methylene chloride, methanol and bidistilled water. Elution was made with two 5  $cm^3$  volumes of diethyl ether and methylene chloride, respectively. Eluent was evaporated, individual phenols and their derivatives were dissolved in hexane, acetylated (derivatized) and concentrated down to a volume of 0.1  $cm^3$  for gas chromatography separation.

## Gas Chromatography - Mass Spectrometry

Chlorophenols are usually determined by use of chromatographic techniques such as HPLC [11] or GC [12]. However, because of their high polarity, they give broad, tailed peaks if separated directly (without prior derivatization) by GC.

The chromatographic analysis were made using a gas chromatograph (Hewlett-Packard type 5890) connected with a quadruple mass spectrometer type 5972 (temperature MS - 162°C) equipped with capillary column HP 5 (60 m x 0.25 mm). Chromatographic separations were led in programmed heating conditions in the range of the temperature from 60°C which was keeping by 1 min then increased to 80°C at the rate of 5°C/min and to 210°C at the rate of 10°C/min. Finally increased to 260°C. The temperature of the splitless injector was 260°C, the carrier gas was helium (rate of flow - 1cm<sup>3</sup>/min). Qualitative estimation was made in SCAN system (identification of individual compounds by comparison with standards of spectra from spectra library type NBS 75KL) and quantitative estimation was done in SIM system (identification by comparison with calibrated standards).

## Results and discussion

Surface water contains many organic compounds both of natural and anthropogenic origin. The composition of the particular compounds in water is related to the influence of different natural factors or a kind of human activity on the aqueous ecosystems. It is considered that the occurrence of chlorophenols and their derivatives in the environment is strictly related to human activity including mainly industry, production and degradation of pesticides and disposal of the communal sewages to open waters [13].

Obtained results evidenced the presence in collected samples of phenol, chlorophenols (4-chlorophenol, 2,4,5-trichlorophenol, 2,4,6-trichlorophenol, tetra- and pentachlorophenol), 4-chlorocatechol, 4,6-dichloroguaiacol, 3-chlorosyringol, trichlorosyringol.

Results of analyses have exerted, that water of Vistula River is significantly contaminated with phenolic compounds.

The highest concentrations were noted for: 4,6-dichloroguaiacol (2.83 µg/dm<sup>3</sup>), phenol (1.67÷2.70 µg/dm<sup>3</sup>), 2,4,5-trichlorophenol (0.25÷2.72 µg/dm<sup>3</sup>), in particular for the pentachlorophenol (0.71÷641.3 µg/dm<sup>3</sup>). The lowest concentrations were observed for the: 2,4-dichlorophenol (0.02÷0.03 µg/dm<sup>3</sup>), trichlorosyringol (0.03 µg/dm<sup>3</sup>) and 4-metylophenol (0.04 µg/dm<sup>3</sup>) (Tab. 2).

Smaller number of organic compounds and lower concentration of phenol identify in samples which were taken from the Pilica River. Following compounds were detected: phenol, 2,4-dichlorophenol, 2,4,5-trichlorophenol, 2,4,6-trichlorophenol, tetrachlorophenol, pentachlorophenol, 4-metylophenol and guaiacol. The highest concentrations were observed for: pentachlorophenol (5.60 µg/dm<sup>3</sup>) and 2,4,5-trichlorophenol (2.49 µg/dm<sup>3</sup>), in turn the lowest for 4-metylophenol (0.03 µg/dm<sup>3</sup>) (Table 1).

In tap water (treated water) destined for the city of Warsaw following compounds were noted: phenol, 2-chlorophenol, 2,4-dichlorophenol, 2,4,5-trichlorophenol, 2,4,6-trichlorophenol, tetrachlorophenol, pentachlorophenol, 4-metylophenol, 4-chlorocatechol, 3,4,5-trichlorokatechol, tetrachlorokatechol and 4,6-dichloroguaiacol. The highest concentration was noted for pentachlorophenol (3.27 µg/dm<sup>3</sup>), phenol (0.43÷1.86 µg/dm<sup>3</sup>) and 2,4,6-trichlorophenol (1.01÷1.14 µg/dm<sup>3</sup>), whereas the lowest

established for 4-metylophenol ( $0.02 \div 0.09 \mu\text{g}/\text{dm}^3$ ), tetrachlorocatechol ( $0.03 \mu\text{g}/\text{dm}^3$ ) and 2,4,5-trichlorophenol ( $0.03 \div 0.04 \mu\text{g}/\text{dm}^3$ ) (Table 2).

Table 1

The means concentrations [ $\mu\text{g}/\text{dm}^3$ ] and their standard deviation obtained for phenol, chlorophenols, chlorocatechols and guaiacols determined in the drinking water of Tomaszów Mazowiecki and surface water from Pilica River six times during 2008

Determined compounds	January		March		May	
	Pilica River	Tomaszów Mazowiecki	River Pilica	Tomaszów Mazowiecki	Pilica River	Tomaszów Mazowiecki
phenol	1.37±0.252	1.20±0.361	0.05±0.021	2.23±0.252	0.15±0.025	1.80±0.400
2,4-dichlorophenol	0.04±0.012	-	-	-	-	-
2,4,5-trichlorophenol	2.49±0.170	-	0.53±0.252	-	0.62±0.104	-
2,4,6-trichlorophenol	0.06±0.015	0.83±0.252	0.89±0.259	0.62±0.076	0.19±0.051	0.10±0.015
tetrachlorophenol	-	-	-	-	0.28±0.057	0.35±0.058
pentachlorophenol	0.03±0.000	-	-	-	5.60±1.253	0.70±0.100
4-methylphenol	-	-	-	0.38±0.040	-	-
4-chlorocatechol	-	-	-	-	-	-
3,4,5-trichlorocatechol	-	0.12±0.031	-	-	-	-
tetrachlorocatechol	-	-	-	-	-	-
guaiacol	-	-	0.59±0.036	-	-	-
4,5,6-trichloroguaiacol	-	-	-	-	-	0.48±0.076
3-chlorosyringol	-	-	-	-	-	-

Determined compounds	July		September		November	
	Pilica River	Tomaszów Mazowiecki	Pilica River	Tomaszów Mazowiecki	Pilica River	Tomaszów Mazowiecki
phenol	0.05±0.010	7.07±1.172	1.13±0.252	-	0.15±0.014	1.25±0.071
2,4-dichlorophenol	-	0.22±0.085	1.03±0.153	-	-	-
2,4,5-trichlorophenol	-	0.53±0.153	-	0.13±0.038	0.05±0.007	-
2,4,6-trichlorophenol	-	0.24±0.125	-	0.16±0.015	0.03±0.000	0.09±0.014
tetrachlorophenol	-	0.33±0.058	0.57±0.153	0.79±0.110	-	0.04±0.000
pentachlorophenol	-	-	2.35±0.304	-	-	-
4-methylphenol	-	-	-	-	0.03±0.000	0.03±0.000
4-chlorocatechol	-	-	-	0.70±0.100	-	-
3,4,5-trichlorocatechol	-	-	-	0.43±0.058	-	-
tetrachlorocatechol	-	-	-	0.67±0.058	-	-
guaiacol	-	1.37±0.321	-	0.44±0.032	-	-
4,5,6-trichloroguaiacol	-	-	-	-	-	-
3-chlorosyringol	-	0.33±0.115	-	-	-	-

Presence of numerous phenolic derivatives evidenced also in samples from tap water from Tomaszów Mazowiecki. For example: phenol, 2,4-dichlorophenol, 2,4,5-trichlorophenol, 2,4,6-trichlorophenol, tetrachlorophenol, pentachlorophenol, 4-metylophenol, 4-chlorocatechol, 3,4,5-trichlorocatechol, tetrachlorocatechol, guaiacol, 4,5,6-trichloroguaiacol and 3-chlorosyringol. The highest concentrations among the phenolic derivatives were noted for phenol ( $1.20 \div 7.07 \mu\text{g}/\text{dm}^3$ ), guaiacol ( $0.44 \div 1.37 \mu\text{g}/\text{dm}^3$ ) and 2,4,6-trichlorophenol ( $0.09 \div 0.83 \mu\text{g}/\text{dm}^3$ ) (Table 1).

The presence of phenol of natural origin is related with photochemical decomposition of different natural substances that are contained in cell walls of plants under the influence of UV irradiation. For example, aromatic amino acids - tryptophan and tyrosine are transformed to phenol and lignin (natural biopolymer present in cell walls of plants) is

decomposed to metoxyphenols such as guaiacols and dimethoxyphenols included syringols. High concentration of phenol in surface water may be related to their resuspension from sediments as this compound is intensively incorporated into humus substances [14]. Chlorinated compounds may be formed *de novo* from simple organic and inorganic compounds and also from aromatic compounds that are present in the environment.

Table 2

The means concentrations [ $\mu\text{g}/\text{dm}^3$ ] and their standard deviation obtained for phenol, chlorophenols, chlorocatechols and guaiacols determined in the drinking water of Warsaw and surface water from Vistula River six times during 2008

Determined compounds	January		March		May	
	Vistula River	Warsaw	Vistula River	Warsaw	Vistula River	Warsaw
phenol	1.67±0.306	1.53±0.208	-	1.86±0.350	2.10±0.656	0.05±0.015
2-chlorophenol	-	-	-	-	-	-
2,4-dichlorophenol	-	0.08±0.015	-	-	-	-
2,4,5-trichlorophenol	0.25±0.050	-	2.72±0.501	-	1.76±0.251	-
2,4,6-trichlorophenol	-	1.01±0.101	-	1.14±0.053	0.06±0.010	0.04±0.000
tetrachlorophenol	1.33±0.306	0.05±0.010	0.68±0.104	-	-	-
pentachlorophenol	641.3±61.655	3.27±0.252	6.87±0.666	-	0.71±0.056	-
4-methylphenol	-	-	-	0.09±0.020	-	-
4-chlorocatechol	-	-	0.65±0.150	-	-	-
3,4,5-trichlorocatechol	0.20±0.021	0.08±0.006	-	-	-	-
tetrachlorocatechol	-	-	0.20±0.035	-	-	-
guaiacol	-	-	0.63±0.205	-	-	-
4,6-dichloroguaiacol	-	-	2.83±0.289	-	-	-
trichlorosyringol	-	-	-	-	-	-

Determined compounds	July		September		November	
	Vistula River	Warsaw	Vistula River	Warsaw	Vistula River	Warsaw
phenol	2.70±0,100	0.45±0.050	2.34 ±0,060	0.43±0.058	1.70±0,141	0.06±0.007
2-chlorophenol	-	0.03±0.000	-	-	-	-
2,4-dichlorophenol	-	-	0.03± 0,000	0.03±0000	0.02 ±0,000	0.03±0.000
2,4,5-trichlorophenol	-	0.04±0.006	0.27± 0,058	0.03±0.000	0.75±0,071	-
2,4,6-trichlorophenol	-	-	0.08± 0,000	-	-	-
tetrachlorophenol	-	-	0.50± 0,100	-	-	-
pentachlorophenol	-	-	-	-	-	-
4-methylphenol	-	-	-	0.03±0.000	0.04±0,014	0.02±0.000
4-chlorocatechol	-	0.04±0.000	-	-	-	-
3,4,5-trichlorocatechol	-	-	-	-	-	-
tetrachlorocatechol	-	-	-	0.03±0.000	-	-
guaiacol	-	-	0.37± 0,058	-	-	-
4,6-dichloroguaiacol	-	-	-	0.14±0.010	-	-
trichlorosyringol	-	-	0.03± 0,006	-	-	-
3,4,5-trichloroveratrole	-	-	-	-	-	-

In water, chlorinated metoxyphenols may be dechlorinated or O-demethylated to form chlorophenols or phenol molecules [15].

The chlorination processes have very important meaning in elimination of organic matter, xenobiotics and some other pathogens from drinking water. However their use may cause risk for the formation of new, strongly toxic, mutagenic and carcinogenic compounds.

Phenolic derivatives were observed in water from Vistula River. The most often noted compound was phenol (in each samples except March). Similar in the Pilica River phenol was observed in each month. Beside of the fact that in surface water from Vistula river concentrations of phenol were higher than in water from Pilica River. After disinfection opposing correlation had occurred - higher concentrations of phenol were in tap water from Tomaszów Mazowiecki.

Analysis of the literature data confirm that a lot of low-molecular compounds can develop in treated water through degradation of dissolved natural organic matter.

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## OCENA ZAWARTOŚCI CHLOROFENOLI I ICH POCHODNYCH W WODACH WODOCIĄGOWYCH I RZECZNYCH

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**Abstrakt:** Wody powierzchniowe zawierają wiele związków organicznych zarówno pochodzenia naturalnego, jak i antropogenicznego. Związki aromatyczne ze względu na ich toksyczność i obecność w środowisku stanowią poważne zagrożenie nie tylko dla żyjących organizmów, ale również biocenozy przyrodniczych. Jedną z najważniejszych grup ektotoksyn są chlorofenole. Stosuje się je jako prekursorzy i składniki wielu substancji chemicznych, w tym polimerów, barwników oraz leków. Chlorofenole są silnie toksyczne, wykazują działanie genotoksyczne, mutagenne i rakotwórcze. Ponadto podstawienie tych związków dodatkowymi atomami chloru

powoduje wzrost ich toksyczności i wydłuża czas akumulacji w organizmach żywych. Metodą, która pozwala pozbyć się patogenów, nadmiernej ilości materii organicznej oraz ksenobiotyków z wody, jest jej chlorowanie. Proces ten wykorzystuje oksydanty chlorowe, wśród których chlor gazowy oraz ditlenek chloru mają bardzo ważne znaczenie. Skutkiem ubocznym stosowania chloru jest tworzenie nowych związków chloroorganicznych, takich jak halometany, chlorofenole, chlorowane węglowodory aromatyczne oraz wiele innych związków organicznych. Ich stężenia w wodzie pitnej zależą w głównej mierze od stężeń ich organicznych prekursorów oraz od dawki chloru zastosowanej w procesie uzdatniania wody. Celem pracy było określenie zawartości fenolu, chlorofenoli, chlorokatecholi oraz chlorowanych metoksyfenoli w wodzie pitnej z Warszawy i Tomaszowa Mazowieckiego oraz w wodzie pobranej z rzek Wisły i Pilicy, wykorzystując do pomiaru metodę chromatografii gazowej.

**Słowa kluczowe:** fenol, chlorofenole, chlorowanie, oksydanty chlorowe