

# WATER SUPPLY, SEWERAGE, BUILDING CONSTRUCTION OF WATER RESOURCES PROTECTION

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## NATURAL AND DESALTED WATER PURIFICATION FROM ORGANIC SUBSTANCES

**Problem statement.** The results of quality analysis of water from different sources before and after demineralization in ion exchange units with the use of different technological schemes are presented. The aim of the analysis is to determine the nature and quantity of remaining impurities.

**Results and conclusions.** It is established that the impurities are humic and fulvic acids. It is these acids that cannot be entirely removed during water conditioning with the help of sorption and ion exchange methods. To remove them, it is necessary to use porous sorbents selective to these substances.

**Keywords:** natural waters, water purification, humic acids, fulvic acids, desalination, sorption.

### Introduction

The continuous monitoring of the biosphere led to the conclusion about quality degradation of natural source water. Almost all industries cannot use natural water without the pretreatment.

Industries of many types need high-resistance desalinated water which is obtained by conditioning of natural source water by the methods of ion exchange, electrodialysis, reverse osmosis, etc. [1]. It is known that the major difficulties are caused by the necessity of organic substance removal.

The most frequent source of water-supply for industrial objects is a city water pipe. According to the drinking water quality standard [2], the total amount of organic substances in it is limited by the color of water and permanganate oxidizability indices. The colour of water indirectly indicates the presence of high-molecular organic acids of the humus nature, i. e. humic acid (HA) and fulvic acids (FA).

It should be noted that drinking water purification from humic acids has never been the high-priority task in this country. If the colour of water is within the moderate level, the stage of coloured impurities removal is not provided for at all in many city water purification plants. Consumers often get chlorine- or ozone-disinfected water without any correction of the amount of organic impurities. The major factor for using coagulation is water turbidity caused by colloids and coarsely dispersed substances. Due to peculiar standards of technological water quality, consumers have to build their own water treatment constructions with a different set of physical and chemical methods of affecting the impurities.

### **1. Organic impurities in natural water**

In order to work out efficient methods of organic substance removal from natural waters, the information on the chemical content of impurities in both the initial and desalted water. Without this, it is impossible to develop scientifically proven methods of water-conditioning.

Obtaining the data on the character of impurities in the water before and after desalting presupposes finding out those organic components which are not detected during the water-treatment process in none of its stages. There are hardly any data of this kind available in literature where the main quality index is permanganate oxidizability (PO) or the total organic carbon content (TOC). Some scattered data are presented in the work [1].

We have tested the water in 46 sources for the organic substance content in different seasons. The test results of some of them are given in Table 1. The methods by A. D. Semyonov, 1964, [5] and O. V. Bryzgalo, 1966 [4].

The water was tested not only for humic and fulvic acids but also for some other classes of organic substances which are typical of natural waters not contaminated by drains: electrolytes (e. g. organic acids) and non-electrolytes (sugars, esters, etc.) as well were found out. Thus, methods of removal of impurities of this type should be selected in each specific case with the consideration of the character of the substances present in the water.

Table 1

## Organic substances content in natural waters

City, source	PO <sub>4</sub> , (±0.2) mgO <sub>2</sub> /l	HA, (±10) microgram/dm <sup>3</sup>	FA, (±0.1) mg/dm <sup>3</sup>	Amino acids, (±1) microgram N/dm <sup>3</sup>		Sugars, (±20) microgram/dm <sup>3</sup>	Esters, microgram-equiv/dm <sup>3</sup>	Carbonic acids, microgram-equivalent/dm <sup>3</sup>
				free	in hydrolyzates			
Zaporozhye	9.3	450	13.5	4	80	-	140	15
Moscow	9.0	440	13.4	5-6	120	-	110	17
Tallinn	4.8	260	5.7	10	55	-	102	21
Voronezh, Usmanka River	4-13	200-500	9.6-16.4	25-40	170-300	330-680	80	30
St. Petersburg, Neva River	6-10	340-540	9.8-10.0	6-1	40-120	260-780	100	20
Kiev*	1.0	<10	0.5	5-6	70	380	80	16
Ryazan*	0.9	<10	1.1	-	-	-	-	15
Ryazan, Oka River	7.0	210	6.6	50	230	760	-	19
Krasnoyarsk*	2.2	60	2.3	27	110	260	-	20
Vyatka River	5-16	800	26.0	18-35	130	1250	20-40	20-30
Pitkyaranta	9-13	-	12.0	19	140	1600	30	10
Zelenograd*	0.9	-	4.8	25	100	680	20	-
Novgorod	15.0	800	4.0	4	140	2000	-	80-100

**Notes on the table:** \*is artesian water; the other sources are rivers and lakes.

The analysis of the data obtained allows to draw the following conclusions:

- humic and fulvic acids prevail in natural waters, the latter 10—30 times outnumbering the former;
- seasonal oscillations in the quantitative content of organic impurities of water;
- water oxidability does not always correlate to the content of HA and FA: at identical or similar permanganate oxidizability values the humic acid content in the waters may

be 3—4 times different. Thus, it is not HA or FA but easily oxidized compounds of other classes that determine this index;

- impurities of organic nature are in greater number in water of open water bodies than in water of artesian sources;
- fulvic acids are present even in very clean artesian sources when oxidizability is low and humic acids are not present;
- there are a lot of amino acids in water which are found out after acid hydrolysis of dry remains of water, which is a result of humic and fulvic acids (HA and FA) destruction.

Dry remains of a number of natural waters were exposed to acid hydrolysis (in the solution of 2 mole/dm<sup>3</sup> HCl) in sealed ampoules for 6 hours. During the process there were multiple increases in the number of amino acids and sugars (Table 2). Undoubtedly, this is caused by the presence of humic and fulvic acids in water which contain amino acids and sugars acting as side radicals [6].

Table 2

Reducing sugars and amino acids content before  
and after the hydrolysis of dry remains of natural waters

City, source	Month	Reducing sugars (±10), microgram/dm <sup>3</sup>		Amino acids (±5), microgram N/dm <sup>3</sup>	
		Before the hydrolysis	After the hydrolysis	Before the hydrolysis	After the hydrolysis
Voronezh, Usmanka River	September	650	3180	25	180
Voronezh, artesian water	January	660	1820	20	70
Voronezh, artesian water	April	650	2030	30	70
St. Petersburg, Neva River	September	780	1210	10	40
Ryazan, Oka River	March	760	1470	50	235

Hence, the tests performed on the water of a large number of sources in various geographical zones showed (see Tables 1 and 2) that the most frequent impurities in natural waters are humic acids which are washed away from the soil and are also products of decomposition of water plants and hydrocoles.

## 2. Organic substances in desalted water

The analysis of water desalted by the ion exchange method (according to various technological schemes) showed that depending on the content of impurities in the initial water) it contains 0.5—2.0 mg/dm<sup>3</sup> of analytically revealed organic substances, i. e. 10—15 times as much as impurities of mineral nature remaining in high-resistance water.

At the very beginning of the ‘electronic’ era desalted water was generated from a distillate. During the process not only non-organic but also organic substances were removed. The production of high-resistance deionat with the change in the water state of aggregation was performed on low-power setups. According to our research, however, there are still a lot of impurities of organic nature even in water cleansed by distillation (Table 3).

Table 3

Organic substances in water before and after the distillation

City, source	PO, ( $\pm 0.2$ ) mg O <sub>2</sub> /dm <sup>3</sup>	HA, ( $\pm 10$ ) microgram/dm <sup>3</sup>	FA, ( $\pm 0.05$ ) mg/dm <sup>3</sup>	Amino acids, ( $\pm 1$ ) microgram N/dm <sup>3</sup>	Carbonic acids, ( $\pm 0.5$ ) microgram-equivalent/dm <sup>3</sup>	Sugars, ( $\pm 0.02$ ) mg/dm <sup>3</sup>
Moscow, water pipe	9.0	40	13.45	6	17.0	-
Moscow, distillate	0.3	10	0.10	1	4.0	-
St. Petersburg, water pipe	7.4	540	13.25	6	22.5	0.27
St. Petersburg, distillate	0.4	40	0, 20	1	7.0	0.06
Voronezh, water pipe	1.2	10	1.10	20	9.5	0.65
Voronezh, distillate	0.4	10	0.10	8	3.0	0.08

At large-scale consumption of deionized water natural water desalination is mostly frequently performed by the ion exchange method. It was experimentally [4] found that it is impossible to provide a high ( $\rho = 18$  M $\Omega$ ·cm) limit electrical resistance of the water treated at the content of 2—6 mg TOC/dm<sup>3</sup> in the initial water. In order to determine the quantitative correlation between desalted water quality indices and humic acids, the effect of HA on limit resistance and deionat pH was examined.

### 3. Estimation of the dependence of water quality parameters on HA concentration in desalted water

Humic acids were extracted for the assessment from river water and cleansed from mineral impurities by the ion exchange method. The absence of the latter is proved by the same value of the specific resistance  $\rho$  of acids solutions before and after their filtration through a demineralizing column with a mixed layer of ionites. In order to identify the preparation obtained, its element content was defined, the amount of functional groups was then found and lastly the values of their ionization constants and the mass of a mole-equivalent were calculated by the method of potentiometry. By the methods of electrophoresis on paper, ascending paper and highly efficient fluid chromatography three fractions of the preparation with different fluidities and colours (ranging from yellow to black) were detected in them. These data and the obtained electric and infrared spectrums of absorption of the released HA enabled to identify them as humic acids described in literature [6].

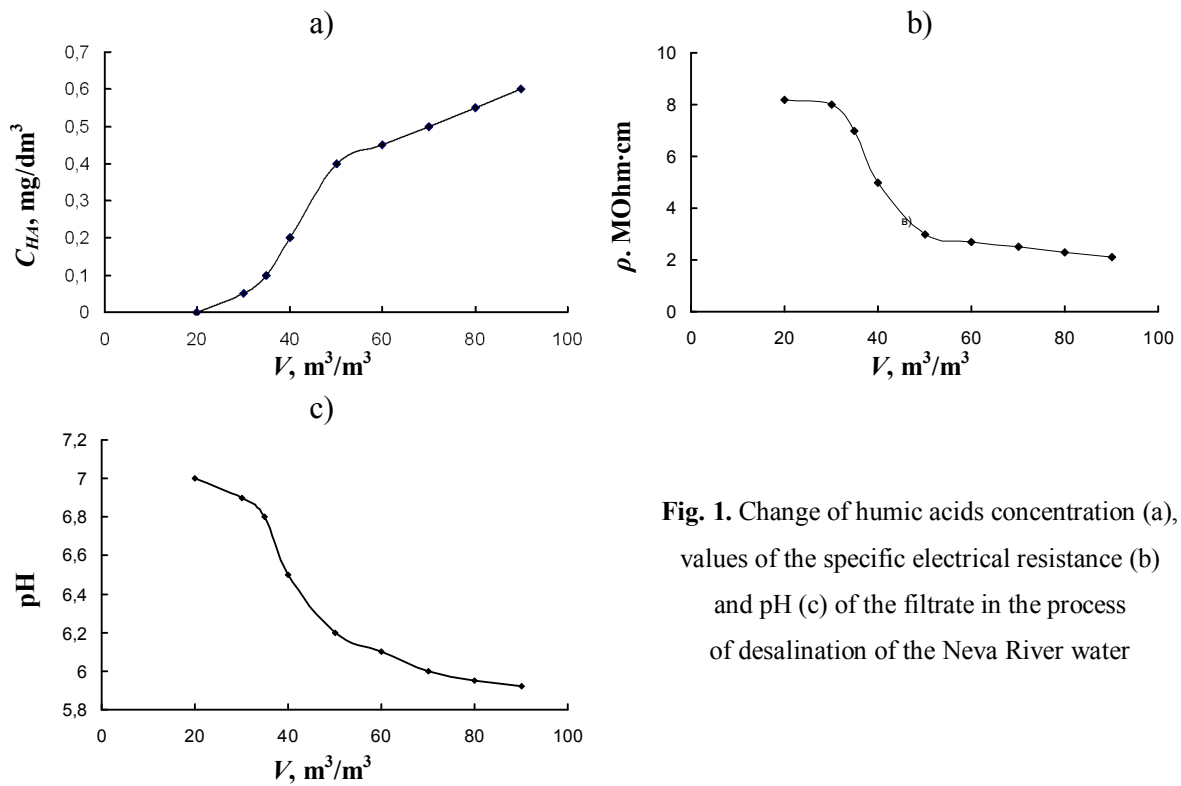
All these operations were conducted to reveal the dependence of optical density  $D$  of HA solutions on their concentration  $c$ , i. e. to draw a calibration plot in the coordinates “ $D$ - $c$ ” Value  $D$  was measured on a spectrophotometer CФ-46 at the wavelength 250 nm and the concentration of HA in the water was calculated by the calibration dependence.

The experiment aiming at revealing the effect of HA concentration on desalted water quality parameters was carried out in the following way. Water from Neva River was desalted on an industrial setup with the power of 10 m<sup>3</sup>/h by the two-step scheme which included coagulation by aluminum sulphate and sequential filtration of river water through cation and anion filters, (the water from the first stage of desalination). This water was further passed through a mixed bed filter (mixture of cation and anion in the volume proportion 1:1.4), this is the second stage of desalination. Specific electrical resistance of the water on the release from the setup was continuously measured in flow-through cells with platinum electrodes and water samples were simultaneously selected for measuring pH and determining the content of humic acids.

Comparing the curves in Fig. 1, we can conclude that the appearance of humic acids (Fig. 1a) leads to a decrease in the specific resistance of the water (Fig. 1b) and in its pH (Fig. 1c).

The research conducted on the quality of artesian, river and lake water desalted on seven industrial setups with the productivity from 1 to 25 m<sup>3</sup>/h showed that while desalting natural

waters by various technological schemes, we fail to remove the organic substances from it, which can be concluded from Table 4.



**Fig. 1.** Change of humic acids concentration (a), values of the specific electrical resistance (b) and pH (c) of the filtrate in the process of desalination of the Neva River water

Table 4

Content of organic substances in river water desalted by various technological schemes

City, source	Water treatment scheme*	Initial water			Treated water		
		PO, ( $\pm 0.2$ ) mg O <sub>2</sub> /dm <sup>3</sup>	HA, ( $\pm 10$ ) microgram/dm <sup>3</sup>	FA, ( $\pm 10$ ) microgram/dm <sup>3</sup>	PO, ( $\pm 0.2$ ) mg O <sub>2</sub> /dm <sup>3</sup>	HA, ( $\pm 10$ ) microgram/dm <sup>3</sup>	FA, ( $\pm 10$ ) microgram/dm <sup>3</sup>
Tallinn, Ulemiste lake	C-C <sub>1</sub> -D-S-A <sub>1</sub> -MBF	4.8	260	570	0.3	<10	400
St. Petersburg, Neva River	C-C <sub>1</sub> -S-A <sub>1</sub> -MBF	8.5	480	1220	0.4	100	650
Voronezh, artesian water	C <sub>1</sub> -D-A <sub>1</sub> -C <sub>2</sub> -A <sub>2</sub>	2.2	50	240	0.2	30	160
Voronezh, artesian water	C <sub>1</sub> -D-A <sub>1</sub> -MBF	2.0	50	240	0.2	<10	80
Novgorod, Volkhov River	C <sub>1</sub> -D-S-A <sub>1</sub> -MBF	11.5	730	450	0.5	70	160

**Notes on the table:** C is the coagulation with aluminum sulphate; C<sub>1</sub> and C<sub>2</sub> are cations of 1<sup>st</sup> and 2<sup>nd</sup> stages of desalination; D is the decarbonifier; S is the sorbent; A<sub>1</sub> and A<sub>2</sub> are anionites of the 1<sup>st</sup> and 2<sup>nd</sup> stages; MBF is the mixed bed filter.

It is concluded from the data of Table 1 that even during the ion exchange desalination of river water containing humic acids, even at a low permanganate oxidizability (further oxidizability) and at the presence of a special sorbent for organic substances the FA residue is 80—160 microgram/dm<sup>3</sup>, nevertheless HA are not always determined in water. Aside from humic formations, sugars, esters and carbonic acids were revealed in desalted water.

Low oxidizability value is attributable to the fact that not all impurities but only 25-40 % of them, as found by the authors [8], are liable to oxidation by potassium permanganate. It is clear that even using a special sorbent (a low-base porous anionite in this case) we fail to get a deionate with the FA content less than 600 microgram/dm<sup>3</sup> out of river water. According to Fig. 1, value  $\rho$  of such water can not exceed 10 megaohm·cm (with the norm of 18 megaohm·cm).

#### **4. Infrared spectroscopy of dry remains of distilled and desalted water**

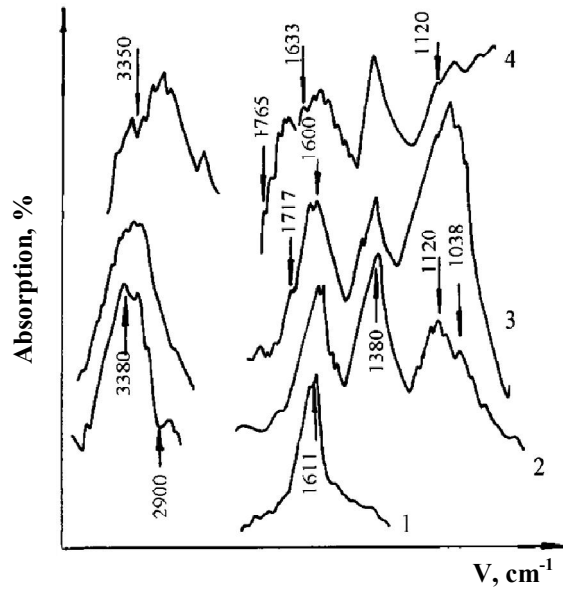
Infrared spectroscopy of dry remains of deionate evaporated under the vacuum in the volume of 3 dm<sup>3</sup> testifies to the variety of impurities of organic nature found in water demineralized in various ways. It appears necessary to compare the quality of the distillate and the filtrate desalinated by ionites.

In Fig. 2 there are infrared spectroscopies of dry remains of tri distillate, bi-distillate, distillate and fulvic acids from the Neva River.

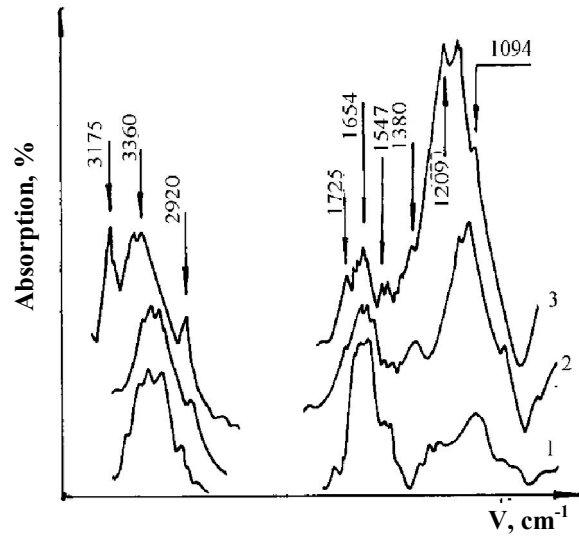
The comparison of infrared spectroscopies of absorption of dry remains of distilled water and water fulvic acids found a lot of common: the presence of methyl methylene groups (2900, 1470, 1380 cm<sup>-1</sup>), C = O (1765, 1700, 1577, 1420, 940 cm<sup>-1</sup>) and NH (3350, 3250, 1630 cm<sup>-1</sup>), formations of aromatic nature (1614, 1500, 1474 cm<sup>-1</sup>) and others [9]. It turned out that even the triple distillation does not provide for the complete water purification from organic impurities. The absorption band in the area 2900, 1720, 1600, 1530, 1495, 1360, 1380 cm<sup>-1</sup> can be regarded as a result of the presence of substances with methyl methylene, carboxyl and amino groups. The bands in the interval 1000—1300 cm<sup>-1</sup> are typical of C-C and C-O bonds. The absorption in the wavelength of 3400 and 3200 cm<sup>-1</sup> is caused by oscillations in functional groups with O-H and N-H bonds.

In Fig. 3 there are infrared spectroscopies of dry remains of water from various superficial sources which was desalinated by the ion exchange method, in Table 5 the amount of some organic substances in the same water is given (expendable samples).





**Fig. 2.** Infrared spectroscopy of dry remains of tri distillate (1), bi-distillate (2), distillate (3) and fulvic acids from the Neva River (4)



**Fig. 3.** Infrared spectroscopy of dry remains of desalinated water from Ulemiste lake (1), Volkhov River (2) and artesian water from Voronezh (3)

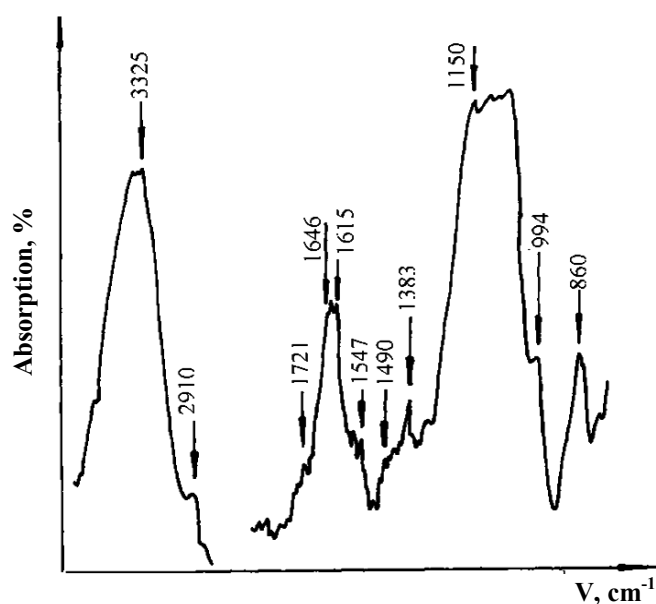
Table 5

Content of organic substances in raw water before (numerator) and after desalination by the ion exchange (denominator)

Source	PO, mg ( $\pm 0.2$ ) O <sub>2</sub> /dm <sup>3</sup>	HA, ( $\pm 10$ ) microgram/dm <sup>3</sup>	FA, ( $\pm 10$ ) microgram/dm <sup>3</sup>	Amino acids, ( $\pm 1$ ) microgram N/dm <sup>3</sup>		Reducing sugars, ( $\pm 20$ ) microgram/dm <sup>3</sup>	
				Before the hydrolysis	After the hydrolysis	Before the hydrolysis	After the hydrolysis
Novgorod. Volkhov River	11.0/0.4	730/40	4350/160	4/2	138/10	2100/320	3230/750
Tallinn, Ulemiste lake	4.8/0.3	260/0	5680/250	6/2	55/20	-	-

The character of infrared spectroscopy of absorption of dry remains of desalinated water of open water bodies is obviously determined by the presence of humic and fulvic acids containing carboxyl groups and nitrogen in amino acid side radicals chemically found in desalinated water.

Infrared spectroscopy (Fig. 4) of dry remains of desalinated artesian water containing a significantly lower number of organic impurities (Podmoskovie) are fairly similar. The desalination scheme is cationite → anionite → mixed bed filter. The productivity of the setup is 25 m<sup>3</sup>/h. The chemical analysis of the filtrate samples revealed 45—50 microgram/dm<sup>3</sup> of HA, 200—250 microgram/dm<sup>3</sup> of FA, 7—10 microgram of nitrogen of amino acids/dm<sup>3</sup> (in the initial water the content of these components was 100, 750 and 20 microgram/dm<sup>3</sup>, 175 microgram/dm<sup>3</sup> of reducing sugars).

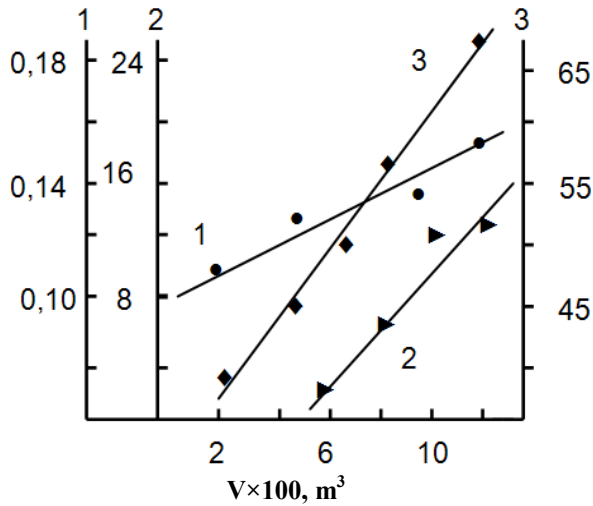


**Fig. 4.** Infrared spectroscopy of dry remains of artesian water desalinated without preliminary purification from organic substances

In the process of the two-stage desalination of artesian water of Voronezh on the setup with the power 20 m<sup>3</sup>/h operating according to the scheme C<sub>1</sub> → A<sub>1</sub> → C<sub>2</sub> → A<sub>2</sub> (where C<sub>1</sub>, C<sub>2</sub> are cationite KY-2-8; A<sub>1</sub>, A<sub>2</sub> are anionite ЭДЭ-10П and AB-17-8), the content of the impurities throughout the whole filtration cycle was kept under control (the data are relevant for the setup operation period before the filling of the Voronezh water reservoir). The chemical analysis of artesian water desalinated in two stages (without using a mix bed filter) showed that HA cannot be at all determined in the first 600 m<sup>3</sup> of the water, while FA were present in the deionate from the very beginning of the filtration cycle (Fig. 5).

Animo acids were not found in the filtrate, after the acid hydrolysis of the dry remains, however, a large number of them was found (in sealed ampoules at 120 °C within 24 hours) in the solution of 2 mole/dm<sup>3</sup> of HCl, which is testified by a significant nitrogen concentration. The results of the determination of HA, FA and amino acids throughout the filtration cycle are presented in Fig. 5.

This supports the view that in deeply desalinated water even at a fairly high specific resistance ( $\sim 10$  megaohm-cm) organic substances are mainly represented by humic acids with a low degree of ionization.



**Fig. 5.** Change in the organic substances content in desalinated water through the filtration cycle:

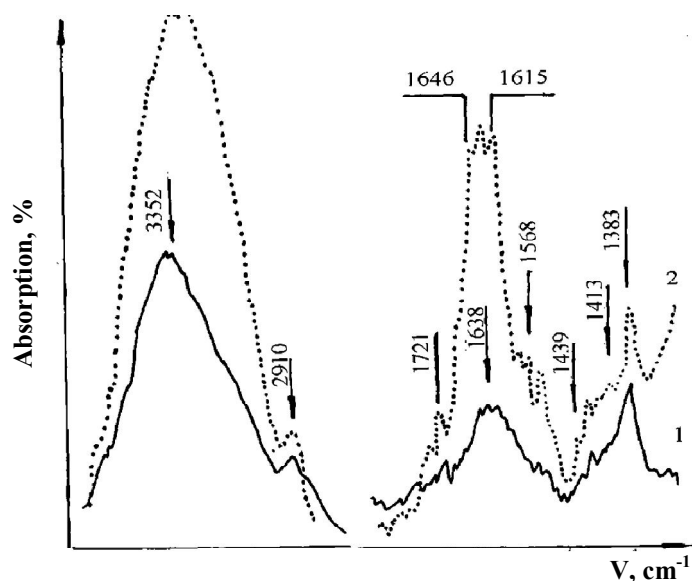
- 1 is fulvic acids, mg/dm<sup>3</sup>;
- 2 is humic acids, microgram/dm<sup>3</sup>;
- 3 is amino acids nitrogen, microgram/dm<sup>3</sup>

Infrared spectroscopy of the dry remains of the desalinated water obtained out of the same initial water only according to the scheme  $A_1 \rightarrow C_1 \rightarrow$  mixed bed filter (the setup productivity is 25 m<sup>3</sup>/h) demonstrate the chemical analysis results, the dynamics of the increasing content of organic substances in desalinated water, which is concluded from the growing height for peaks of main absorption band in the spectrum as the volume of the water passed through the mix bed filter at 3350—3370, 1721, 1610—1638, 1562, 1413, 1383, 944 cm<sup>-1</sup> typical of fulvic acids infrared spectroscopy (Fig. 6).

Fairly similar results of the dry remains of desalinated water analysis were obtained in the work [10]. The identification of functional groups and comparison of infrared spectroscopy conducted in it showed that the main absorption bands in desalinated water lie in five areas: 3400—3200, 2900, 1700—1600, 1400 and 1000—1300 cm<sup>-1</sup>.

The presence of carbonic and other organic acids with long hydrocarbon radicals was found, which is testified by a significant increase in the insensitivity of the bond C-H. An increase of oxygen compounds content, nevertheless the content of organic acids prevails.

With the help of the methods of gas chromatography and spectrometry of chromate masses in desalinated water ([10], — the source is the Dnepr River), individual components of more than 50 classes of organic substances were identified.

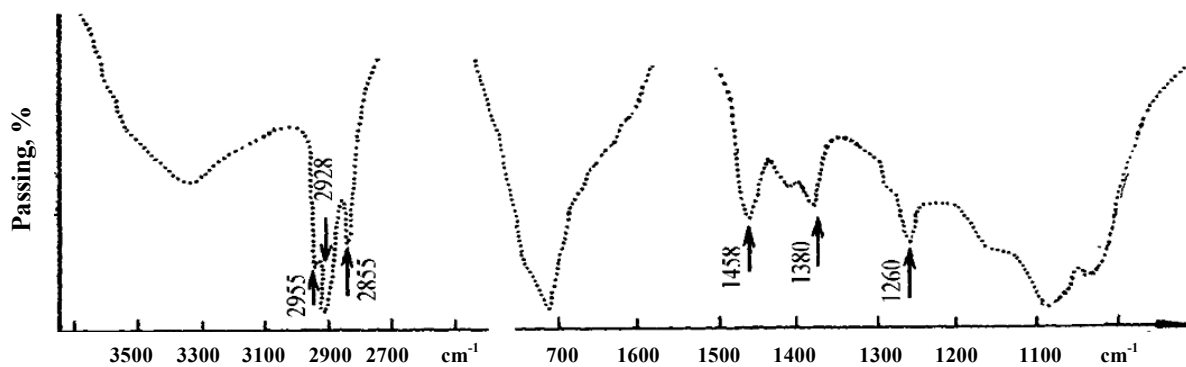


**Fig. 6.** Infrared spectroscopy of the dry remains of artesian water through 9 (1) and 40 (2) hours after the start of the mixed bed filter operation

There is a wide intensive absorption band in the area of  $3400\text{--}3200\text{ cm}^{-1}$  on infrared spectroscopy of the filtrate and distillate which may be related to the manifestation of valency oscillations of N-H bond in acids amides or of bond O-H in carboxyl or alcoholic groups.

The wide absorption band of  $1750\text{--}1450\text{ cm}^{-1}$  with the maximum at  $1660\text{--}1630\text{ cm}^{-1}$  characterizes bond C = O of carboxyl or carbonyl groups, aromatics as well as amides (Fig. 7):  $1650\text{ cm}^{-1}$  (amide I),  $1540\text{ cm}^{-1}$  (amide II).

The narrow absorption band at  $1385\text{--}1375\text{ cm}^{-1}$  manifests itself in the spectrums of all the dry remains. It characterizes deformational oscillations of bond C-H in  $\text{CH}_3$ -groups, which is proved by the absorption band with the maximum at  $1440\text{ cm}^{-1}$ . The presence of methylene groups is determined by the absorption in the area of  $2920\text{--}2910$  and  $810\text{--}790\text{ cm}^{-1}$  [9].



**Fig. 7.** Infrared spectroscopy of the dry remains of desalinated water [10]

The absorption band at  $1180\text{--}1060\text{ cm}^{-1}$  on some absorption spectrums may be variously interpreted, since polysaccharides, alcohols, ethers and polysilicic acids are absorbed in this interval of frequencies.

The presence of the identical components in desalinated waters from different sources is obviously determined the identity of the absorption spectrums of the dry remains of these samples. It was found out that they are greatly similar to infrared spectroscopy of fulvic acids, i. e. the typical absorption bands in infrared spectroscopy of the dry remains of deionate are mainly caused by the fulvic acids.

### **Conclusions**

1. The analysis of the water from a large number of artesian sources and the water of open water bodies found that they all contain organic substances both under the influence of anthropogenic factors and without any.
2. It was found out that regardless of the water source type (an artesian well, a river, a lake, a water reservoir), the major components of the organic composite of water impurities are humic and fulvic acids, high-molecular organic acids with condensed aromatic nuclear which give natural waters a colour varying from yellow to brown.
3. The analysis of desalinated water showed that these very acids are not completely removed in the process of water conditioning by the sorption and ion exchange methods.
4. The quantitative correlation of pH, specific electrical resistance and the humic acids content in desalinated water is presented, i. e. their skip leads to a decline in desalinated water quality according to the parameters given which are normalized for desalinated water used in a precision production and heat and power engineering.
5. With the help of chemical methods and infrared spectroscopy, it was found out that in water demineralized by the ion exchange according to various technological schemes even with the use of a porous sorbent for organic substances there are nitrogen-containing substances remaining which destroy as a result of the oxygen hydrolysis with the splitting of the amino acids.
6. With high requirements for conditioned water, particularly for electronic industry and heat and power engineering enterprises it is necessary to provide for porous sorbents selective to humic, fulvic and amino acids.

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